Process Optimisation of Squeeze Cast Magnesium-Zinc-Rare Earth Alloys and Short Fibre Composites

by

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Dedication

To my father, Mr Yong Bon Tong
and mother, Madam Tham Sai Moi,
for their care, encouragement and love

to my fiancee, Edelweis Lam Nga Ping,
for her continuous encouragement and patience
throughout the final stages of my Ph.D.

to my sisters and brother-in-laws,
Estella Yong Meei Jen and Tan Kok Yong,
Yong Mei Ling and Sim Yong Soon
for their encouragement and support

to my grandmothers, uncles, aunts and cousins,
for their continual advice and love

and finally

to the teachers who taught me in the past,
who have contributed in one way or other to my development
and who made this Ph.D. possible

Abstract

The work reported in this thesis demonstrates the potential of the squeeze casting process for the production of castings using magnesium alloys and its composites. In particular, the studies involving composites are focused on fabrication through squeeze infiltration. These show the ability of the squeeze casting process to produce castings of high metallurgical integrity. The work offers a clear understanding of a number of key parameters for the squeeze casting process which are prerequisites for the production of high strength castings of magnesium alloys and composites. A better understanding of the behaviour of the squeeze cast material tested at both ambient and elevated temperature has been achieved. A methodology, which allows the identification of optimum squeeze casting conditions, has been developed. This has been successfully used in the identification of casting conditions which produce the best tensile properties at both test temperatures.

Two casting programmes, namely: primary and secondary programmes, were designed to evaluate the controlling parameters for squeeze cast magnesium alloys and composites. The investigation was conducted with two magnesium alloys: ternary RZ5DF (Mg-4.2%Zn-RE) alloy and commercial RZ5 (Mg-Zn-RE-Zr) alloy.

Different preform systems were investigated during the primary casting programme and the results showed that 14% volume fraction alumina fibres with 5% silica binder provided the most satisfactory results in terms of ease of fabrication, improvement in strength and cost. Applied pressures of 0.1 to 120 MPa were studied with and without the addition of fibre reinforcement. Pressures of 60 MPa and 80 MPa were found to yield optimum tensile properties in the RZ5DF alloy and its composite respectively. It was also found that a preform temperature of 600°C or above was necessary to achieve minimum resistance to magnesium infiltration at the preform surface. Other process settings, such as applied pressure duration, were also investigated.

The influence of pouring and die temperature on the tensile properties was studied during the secondary casting programme. It was found that a higher pouring and intermediate die temperature provided the highest tensile properties. The mechanical properties of castings were tested at both ambient and elevated temperatures. It was found that fibre reinforcement improved the mechanical properties of the materials at ambient temperature but the most significant improvement was observed at 250°C. The effect of grain refinement (zirconium) addition on the squeeze cast magnesium alloys and composites was also investigated. The results indicated that the tensile properties in the zirconium-free RZ5DF alloy were comparable to those of the RZ5 alloy grain refined with zirconium. The influence of zirconium addition on the tensile properties of RZ5DF and RZ5 MMC was similarly reported. Heat treatment improved the properties of the alloys by a small margin and adversely affected the properties of the composite. The overall results showed that there is an opportunity to achieve a significant saving in material and process cost when producing Mg-Zn-RE alloys and composites by the squeeze casting process.

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CONTENTS

Chapter 1

Introduction

Magnesium alloys are recommended for a wide range of applications, principally because of their lightness, easy machinability, excellent castability, good weldability, superior damping characteristics and availability. Magnesium in its pure state has been of limited use as an engineering material mainly because of its low wear resistance, low tensile properties and loss of strength at high temperatures. Over the years, there has been a substantial amount of development in alloying technology to produce high strength magnesium alloys.

Many modern technologies require materials with unusual combinations of properties that cannot be met by conventional metal alloys. This is particularly true for materials that are needed for future aerospace and automotive applications. Automotive engineers are increasingly searching for structural materials that have low densities and are strong, stiff, abrasion and impact resistant. Pressures from environmental law (i.e. CAFE legislation) across the world have compelled car manufacturers to improve fuel economy. One obvious and important method of meeting Corporate Average Fuel Economy (CAFE) goals is to reduce vehicle weight, and magnesium alloys are considered good candidates for this because of their low density.

However the most complex of magnesium alloys have been unable to meet the demands of the rather formidable combination of characteristics required by the automotive parts. Light materials like magnesium and aluminium are relatively weak in strength, stiffness and abrasion resistance when compared to the required mechanical properties that the applications demand.

Such property combinations may be achieved through the novel technology of a material system composed of a mixture of dissimilar chemical compositions. Such a combination will generally exhibit a significant proportion of properties from both constituent phases such that a better combination of properties is realised. This class of material is referred to as Metal Matrix Composites (MMCs), and research has generally been focused in this area to study the effect of strengthening on magnesium. Strengthening is achieved through the addition of thin, stiff and high strength fibres into the magnesium alloy (usually referred to as the matrix).

MMC components can be manufactured by several methods, which include powder metallurgy and diffusion bonding. However, the metal casting route is especially attractive in terms of its ability to produce complex near net shapes. However gas and shrinkage porosity are common defects for most conventional casting processes. The tendency for porosity formation will be pronounced when fibres

are introduced, as fibres tend to restrict the flow of the molten metal and cause even greater gas entrapment within the casting. It is pointless to fibre reinforce a casting if defects are present, since the addition of fibres will not compensate for poor metallurgical integrity. In order to fulfil the potential of fibre reinforcement and produce pore free castings the squeeze casting process may be proposed. The unique feature of squeeze casting is that the molten metal is pressurised throughout solidification. This prevents the formation of gas and shrinkage porosity, and produces a metallurgically sound casting. Selection of this process is also based on its suitability for mass production, ease of fabrication and its consistency in producing high quality composite parts.

With the continual interest in searching for high strength light materials, the author proposes to evaluate the potential for squeeze casting magnesium alloys and short fibre composites.

The thesis is arranged into twelve chapters, the overall structure and contents are illustrated in figure 1.1. Firstly the purpose of the project is introduced (this chapter) and subsequently the aims and areas of research are discussed (chapter 2). This is followed by a literature review on the magnesium (chapter 3), metal matrix composites (chapter 4) and squeeze casting process (chapter 5). Subsequently critical assessments on research direction are made (chapter 6), and methods and equipment selected for the experiments are listed (chapter 7). The results and observations gathered from the primary and secondary casting programmes are presented (chapters 8 and 9). Next the discussion and summary of the findings of the research are outlined (chapters 10 and 11). Finally, the recommendations for further work are listed (chapter 12).

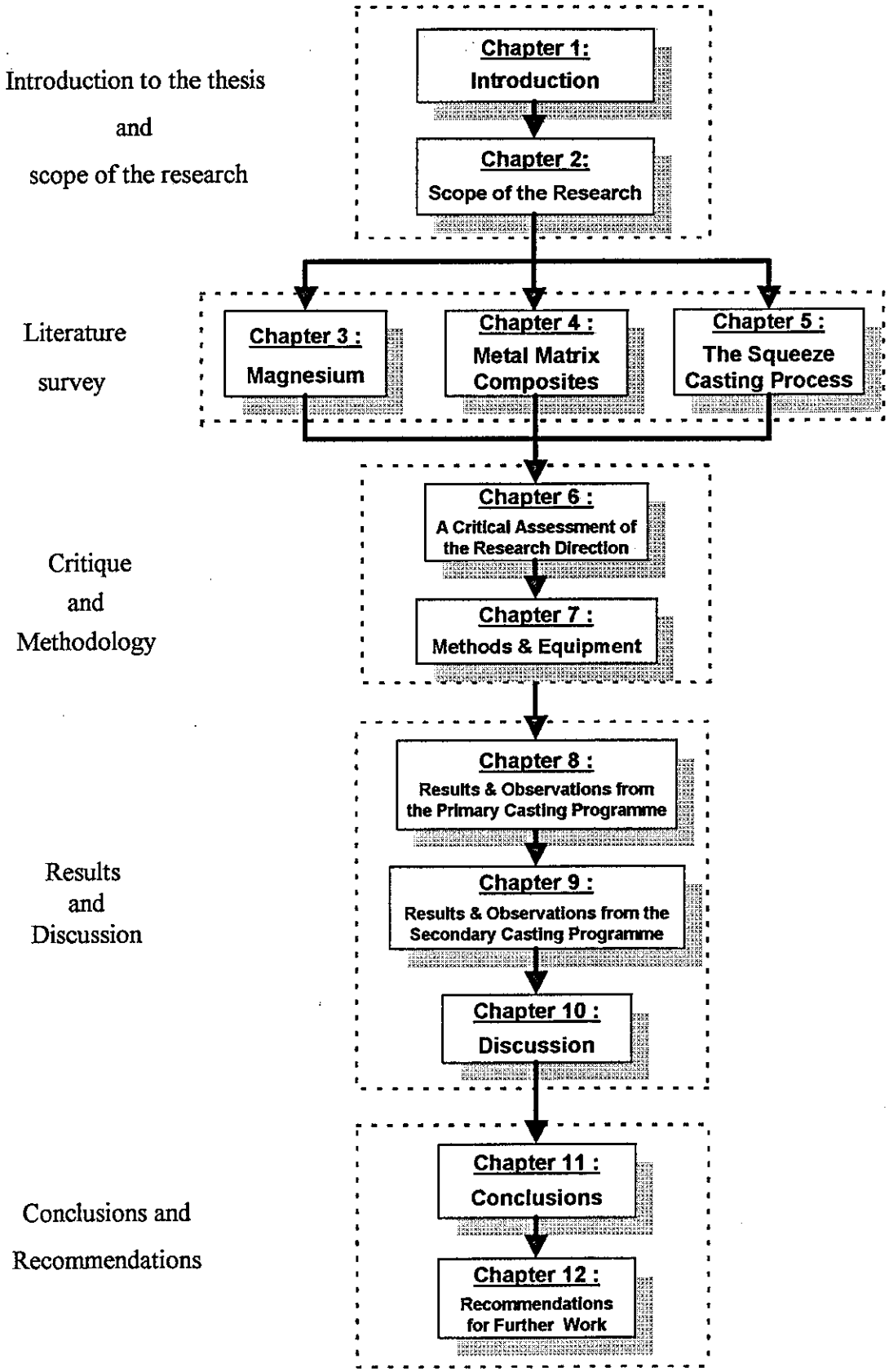


Figure 1-1 The structure of the thesis

Chapter 2

Scope of the Research

2.1 Introduction

This chapter presents the aims and objectives of the research. The growth of magnesium utilisation in lightweight engineering applications will first be discussed, highlighting the potential areas for greater application. The argument for further development of high strength magnesium alloy and composite castings will be presented and the specific problem area which the research will address is highlighted. The scope of the research is outlined in the final part of this chapter.

2.2 The Growth of Magnesium Applications

Advances in the aerospace, automotive, and general engineering applications require the development of new materials with a low density and improved mechanical properties. In recent years, the automotive industry, in particular, has been under increasing pressure to improve the fuel efficiency of cars through the reduction of weight. Weight reduction has, in part, been achieved by the progressive substitution of iron castings by those manufactured in aluminium alloys [1][2][3][4] and, most recently, magnesium based alloys [5][6][7][8]. Furthermore, there has been some application of Metal Matrix Composites (MMCs).

Magnesium, which has a density of only 1.8g/cm^3, is one of the lightest structural materials known to mankind. The materials, such as titanium and aluminium, which are traditionally used in the aerospace industry have densities of 4.5g/cm^3 and 2.7g/cm^3 respectively; they are 2.5 and 1.5 times heavier than magnesium [9]. In addition magnesium alloys also offer many other good engineering characteristics, these include easy machinability, excellent castablility, good weldability and superior damping characteristics. Further details of these characteristics will be presented in section 3.3. In spite of its advantages, however, the widespread application of magnesium, as an engineering material, has often been limited due to its low mechanical properties [10].

With the recent development of Metal Matrix Composites (MMCs), magnesium alloys can better meet the various demands of many applications. The addition of reinforcements in the magnesium alloy, discussed later in Chapter 4, results in superior mechanical properties and good thermal stability. The range of composite materials, highlighted in section 4.3.1, is diverse and each has its own particular characteristics and advantages. From the vast selection of composite types, the discontinuous and randomly oriented fibre-reinforced composites offer the best "value to strength ratio".

Several processes can be used to produce discontinuous MMCs. Of all fabrication methods, the squeeze casting method is the most highly developed and advantageous for producing commercial MMC products [11][12][13][14]. Some examples of well established products are aluminium engine pistons and connecting rods [1][3].

2.3 The Potential Areas for Research

The properties of as-cast components are influenced through the combination of three elements, namely: material selection, casting design and process parameters. This is illustrated in figure 2-1. To optimise the as cast properties, improvements must be made to each of these parameters. The selection of material refers not only to the choice of alloys, but also to the selection of fibres or preform.

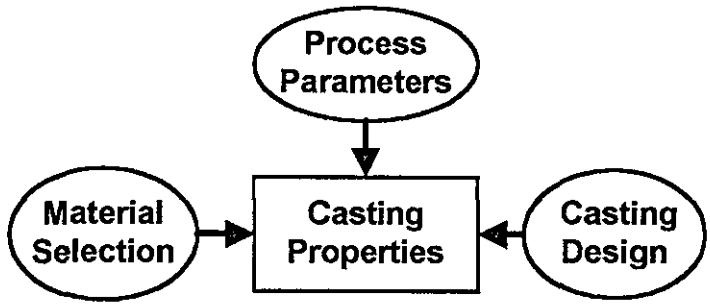


Figure 2-1 The main parameters which contribute to the final properties of the casting.

The optimisation of properties through material alloying and the addition of fibres has been actively studied by researchers in both Germany and Japan. Researchers like Kainer [15][16][17], Singer [10][18], Kamodo [19][20][21], Kamio [22][23[24] and their co-workers have explored new alloys and process routes to optimise the properties of magnesium.

In spite of the advantage of low density that magnesium has over aluminium and zinc, a knowledge of squeeze casting parameters for magnesium alloys and composites has not been established, in comparison to their aluminium and zinc counterparts. Fukunaga [25][26][27], Zantout [28], Chadwick and his co-workers [29][30] have conducted extensive studies on the effects of process parameters on squeeze cast aluminium alloys and composites. These included the effects of applied pressure, ram speed, fibre and pouring temperature. Begg [31] and Yacoub [32] studied the effects of process parameters on squeeze cast zinc base alloys and their composites. They evaluated the various squeeze casting process parameters such as applied pressure, duration of applied pressure, and influence of die and pouring temperature.

The work of Fukunaga, Chadwick, Zantout, Begg and Yacoub has contributed significantly to the establishment of squeeze casting process parameters and their influence on casting properties. In spite of their contribution, certain process parameters still remain to be examined in greater detail to provide a more complete understanding.

Further research is required to understand the influence of applied pressure on tensile strength of the alloys and composites. The influence of applied pressure on the hardness properties has also to be studied. Another important aspect is to establish the effects of the temperature gradient (between the pouring and die temperature), on the tensile properties and microstructure of the casting.

At the outset of the author's research most of the studies conducted on process parameters for squeeze casting had been concentrated on aluminium alloys, zinc base alloys and their composites. There were only a handful of results from studies of process parameters for casting magnesium alloy and its composites; these have been generated by researchers like Chadwick, Ha, Kainer and Kamado. Chadwick [33] and Ha [34], investigated the effects of various process parameters, such as applied pressure, pouring temperature and fibre additions, on the mechanical properties of the casting. Kainer and his co-workers conducted several studies on the influence of fibre volume fraction and the types of fibres [35][36]. Kamado and his co-workers examined the effects that fibre volume fraction, pouring and preform temperatures had on the magnesium matrix composites [19][37].

There is, at present, a significant need for more investigations on the influence of process parameters on the as cast magnesium properties. This research is, therefore, focused on the establishment of in-depth knowledge of squeeze casting process parameters, in particular for magnesium alloys and composites. This research will build on the foundations of magnesium alloying and processing established by Kainer, Kamado, Chadwick, Ha and Singer. The research will also extend the studies carried out by Fukunaga and Begg on the process parameters for squeeze casting.

The author considers that the study of process parameters is important because it has the potential to enable maximum material properties to be attained without incurring additional cost. This is in comparison, for example, to alloying methods such as the addition of expensive materials like silver and yttrium to magnesium. The results of this research, presented in chapters 8 and 9, will indicate why it is essential to understand the effects that these process parameters have on the material properties.

2.4 A Statement of the Research Problem

The focus of contemporary research on squeeze casting has been based on the development of a

suitable magnesium alloy. The focus of squeeze infiltration[1] has been on the pursuit of a suitable magnesium alloy for fibre reinforcement and/or a suitable fibre system for selected commercial magnesium alloys. There is, however, insufficient work on the study of the influence of casting process parameters. In order to carry out this research on process parameters external influences, such as material and casting design variables, have to be constrained. There is a further need to determine a suitable combination of alloy, preform system and casting design. This correspondingly requires investigations into the external factors to underpin a set of fixed variables, in order to evaluate the casting process parameters more accurately.

2.5 Aims

This research aims to contribute significantly to the advancement of magnesium and squeeze casting technology by establishing both high strength magnesium alloy and composite casting. This is to be achieved through obtaining the best combination of material, advanced technology and process optimisation to attain the best mechanical properties.

2.6 Objectives

The research is aimed at providing a better understanding of the effects of process parameters on the properties of the fabricated magnesium alloys and composites. In order to meet the stated aims, the objectives of this research are:

(i) To develop an inexpensive and effective magnesium alloy system that is suitable for the infiltration of fibre reinforcement using the squeeze casting process.

(ii) To determine a suitable preform system for the selected magnesium alloy.

(iii) To study the effects of squeeze casting parameters (pressure, temperature, etc.) on the structure and properties of magnesium alloys and magnesium MMCs.

(iv) To study the influence of grain refinement addition (zirconium) on squeeze cast Mg-Zn-RE alloy and its composite.

(v) To optimise squeeze casting conditions with respect to the room and elevated temperature properties of the alloy and composite.

(vi) To examine the potential of heat treatment to enhance the properties of the cast magnesium alloys and the reinforced magnesium MMCs.

[1] **Squeeze infiltration** is similar to squeeze casting, the only difference is the inclusion of a fibre preform. In this case the pressure is used to infiltrate the molten metal into the preheated preform. Further elaboration of this process can be found in section 5.3.2.

2.7 Scope of the Research

In order to meet these objectives, the following research will be conducted.

2.7.1 Magnesium and its Alloys

A review of magnesium and its alloys will be conducted to establish a better understanding of the material's characteristics and the way magnesium is processed by casting. Its current and future applications will be examined and the potential areas of research interest will be highlighted. The effects of alloying elements on the metallurgical properties of magnesium will be studied. This is to establish a thorough understanding of the principles of magnesium alloying to enable a suitable alloy composition to be designed and thereby take full advantage of the advances in process technology, fibre reinforcement and the squeeze casting process.

2.7.2 Metal Matrix Composites (MMCs)

A review of metal matrix composites technology will establish a better understanding of fibre reinforcement morphology. Studies will, in particular, be conducted to establish a suitable fibre system to be used with magnesium and its alloys, where consideration of the possible reaction between the fibre and the matrix is of interest. It is also necessary to review the fabrication techniques for metal matrix composites as this will enable the selection of the most suitable fabrication method for the research. The current and future applications will also be identified and potential areas of research interest will be highlighted.

2.7.3 The Squeeze Casting Process

A review of the squeeze casting process will be conducted to establish a better understanding of the process parameters. The pros and cons of the process will be studied in great detail as an attempt will be made to implement the process for components which will take full benefit of the process and its inherent characteristics. This study will also have to include areas of applications and potential research interest.

2.7.4 A Critical Assessment of the Contemporary Research on Magnesium MMCs

A critical evaluation of the contemporary research will be carried out to establish the research direction through consolidation of the literature review on magnesium, metal matrix composites and the squeeze casting process. This will provide information on the representative solidification characteristics of the process and enable studies of the true process capabilities to be conducted.

Finally, the specific areas of research will be clearly identified, based on both the information collected from the literature review and through a better understanding of the materials and process.

2.7.5 Research Methods and Equipment

A detailed methodology to include materials, experimental approach, casting parameters, metallography and testing methods will be provided following a review of research methods and equipment. A die-set will be designed to produce appropriate test specimens with consistent grain structure. In order to enable a complete study of the effect of solidification on tensile properties, a suitable die cavity design must be developed to allow the extraction of tensile specimens along the longitudinal and transversal direction. Details of the preliminary and secondary programmes to evaluate process variables and casting parameters for the experimental work will be presented.

2.7.6 Critical Evaluation of Results from the Primary Casting Programme

In order to examine the influence of grain refinement addition in squeeze cast magnesium alloy and its composite, the process variables must first be established. Hence, preliminary castings must be produced to establish the optimum process parameters. These parameters will be used, subsequently, to examine the influence of grain refinement addition, die and pouring temperature on the cast properties during the secondary programme of experiments.

Different preform systems will be examined first in order to determine which will provide the best properties when infiltrated with the RZ5DF (Mg-4.2%Zn-RE) alloy. Following selection of the preform, research will be carried out to determine the influence of process parameters by evaluating the tensile properties, hardness and microstructure of the castings. Analysis of the alloy distribution will be carried out to examine the material solidification characteristics. Finally, the fracture surfaces of the tensile specimens will be studied to provide an understanding of the failure mechanism in the alloys and composites.

2.7.7 Critical Evaluation of Results from the Secondary Casting Programme

Based on the optimum process parameters established from the preliminary work, further evaluation will be carried out to establish the effects of pouring and die temperature on the alloys and composites. In order to determine the optimum production process parameters, the tensile properties and microstructure of the specimens fabricated with the various process parameters will be examined. The influence of grain refinement (zirconium) addition on the mechanical properties will be studied at both ambient and elevated temperature. In order to determine the optimum heat treatment condition for the casting, peak hardness examination will be carried out for both alloys and composites.

Chapter 3

Magnesium and its Alloys

3.1 Introduction

This chapter starts with a short overview of the application of magnesium alloys in the context of their advantages and disadvantages before providing a review of alloy development. It goes on to describe melting and processing procedures before identifying developments such as metal matrix composites.

3.2 The Applications of Magnesium Alloys

In the past decade cast magnesium alloys have enjoyed a renaissance in many manufacturing industries, especially the automotive [38][39] and electronics industries [40][41][42] where weight reduction is critical. The aerospace industry is also responding to the growing acceptance of magnesium [43][44], resulting from the recent advances in magnesium technology.

The uses of magnesium die castings derive essentially from their weight-saving effect, though in many instances, the saving in machining costs alone can be a sufficient justification. The die casting of magnesium alloys is forecast to increase by 400% from 1990-2000 according to Magnesium Service Ltd [41] as compared to 154% for aluminium and 37% for plastics. Cole [40] also predicted that the use of die cast magnesium components will increase over the next decade, based on the projected narrowing of the price-per-pound gap between magnesium and aluminium.

A principal requirement in the selection of materials for aerospace applications is weight. Inevitably, magnesium has been of significant importance to the aerospace industries. The BAe 146 commuter aircraft, for example, has more than 200 magnesium alloy components ranging from cockpit canopies, wheels, gearboxes and electronics casings [45]. Eurocopter, in France, has selected magnesium for the Tiger intermediate and tail gear box casing which has reduced the overall weight by approximately 3kg (30%) [46].

The substantial increase in the use of magnesium alloy castings in automotive applications is attributed to increasingly stringent environmental laws, for example CAFE legislation which has compelled the reduction of vehicle weight to improve fuel economy. It is estimated that a 10% reduction in vehicle weight yields approximately a 5.5% improvement in fuel economy [47]. Allison and Cole calculated that a 91kg weight reduction on a vehicle will increase fuel efficiency by approximately 1mile/gal or 0.43 km/l [48]. The use of magnesium in the new Volkswagen (VW) Passat's gearbox housing results

in a 4.5kg weight reduction [49]. The VW Polo door, fabricated by pressure-cast magnesium with carbon fibre reinforced outer panel, is reported to reduce weight by as much as 40% [49]. The substitution of a magnesium alloy for the Fiat instrument panel carrier has yielded better static and dynamic performance than the previous steel sheet solution with a 41% weight reduction at no additional cost [7].

Magnesium materials are used in defence applications because of their light weight. Typical applications are portable radar, missile launchers, spacecraft and high mobility multipurpose wheeled vehicles for the US army, all of which use magnesium castings in their components [50].

Magnesium castings are used in many commercial applications, especially where their lightness and rigidity are required. With superior light weight characteristics, magnesium has become the major structural material in mobile communications, i.e mobile phones and other electronics related casings, in preference to aluminium [41][49]. One major advantage of magnesium is its EMI (electro-magnetic interference) shielding capability.

3.3 Criteria for the Selection of Magnesium

Magnesium is superior to other materials in a number of properties. A knowledge of these properties, and the full implication of each for the service of a part, is required for proper selection of the material. Thus the favourable and unfavourable properties of magnesium are listed as follows:

3.3.1 The Advantages of Magnesium

Some of the major advantages of magnesium are:

(i) ***Superior high stiffness to weight ratio*** [40][51].

Most magnesium alloys have densities ranging from 1.8 g/cm^3 to 1.85 g/cm^3. Such low density means that on a volume basis magnesium alloys are two thirds the weight of aluminium and one quarter the weight of steel [51]. This means for a specific part, a lower density can be translated into lower weight, lower cost, improved ruggedness, or even all three.

(ii) ***High Electro-Magnetic Interference (EMI) shielding capability*** - This has particular significance when using it in electronic and electrical casings for communication devices [41][49][52].

(iii) ***Very good thermal conductivity*** - Useful for parts such as engineering casings that require rapid heat dissipation [40][53].

(iv) ***Excellent machinability*** - It requires only 55% of the power required to machine aluminium, or 20% that of carbon-steel [53].

(v) ***Higher rate of production*** - It can be cast up to 1½ times faster than aluminium [41].

(vi) ***Good Castability*** - This has particular significance for casting complex shapes [47][54].

(vii) ***Low affinity for iron and steel*** - This has particular significance in die casting, where the low adherence to steel dies results in easy ejection of magnesium castings from the die cavity, thus prolonging tool life [40].

(viii) ***Unlimited supply*** - It is the eighth most abundant element and the sixth most abundant metal on earth [51].

(ix) ***Superior dampening characteristics*** [40][41].

(x) ***Good weldability characteristics*** [44].

3.3.2 The Disadvantages of Magnesium

Some of the major disadvantages of magnesium are: (i) moderate strength at elevated temperature, (ii) poor corrosion characteristics, (iii) high notch sensitivity, (iv) poor stress properties, (v) low hardness value and low ductility [10][44]. Nevertheless the poor characteristics of magnesium can be overcome through alloying techniques and through the introduction of thin, stiff and high strength fibres into the magnesium alloy.

3.4 Basic Metallurgy of Magnesium and its Castability

Pure magnesium, as with aluminium and other metals is too weak to be used commercially without alloying, having an ultimate tensile strength of only 92 MPa [55] as cast. The effects of alloying elements on the metallurgical properties and the castability of magnesium will be discussed in greater detail in the following sections. This discussion will be based on the alloy constitution, as cast tensile properties, fluidity, resistance to shrinkage microporosity, susceptibility to heat treatment, and the relationship between microstructural features and mechanical properties.

3.4.1 Magnesium-Aluminium Alloys

Aluminium is added to refine the grain of magnesium, thereby improving the mechanical properties. It has been reported by Fox [56] and Emley [57] that an aluminium content of up to 6 per cent increases as cast properties, after which the ultimate tensile strength (UTS) falls gradually and the elongation rapidly [56]. With full heat treatment, Fox found that maximum strength is achieved with 10 per cent aluminium addition through the formation of β-phase particles ($Mg_{17}Al_{12}$) in the cast structure.

Castability increases with increasing aluminium content and this is due to the improvement of fluidity. However, aluminium also increases the tendency for shrinkage micro-porosity owing to the increase in freezing range.

3.4.2 Magnesium-Zinc Alloys

Zinc is added to magnesium to impart strength [58]. However, the alloys have the tendency for microporosity and display brittleness characteristics [55]. Figure 3-1 shows how as cast properties increase with zinc content up to approximately 6 per cent, after which both the UTS and the elongation fall. Two factors, namely grain size and marked tendency to microporosity cause low tensile properties. The main reason for this decrease in strength is that the alloy is a long freezing range (LFR) alloy. As a consequence, it inherits the poor characteristics of LFR materials, such as shrinkage porosity when fabricated with conventional casting processes (i.e. sand casting).

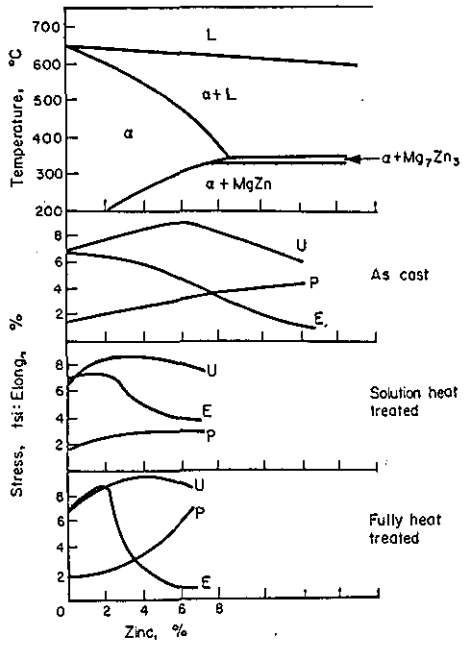


Figure 3-1 Relation of properties to constitution in Mg-Zn alloys: effects of composition and heat treatment on the tensile properties of sand cast binary Mg-Zn alloy [56]

It has been reported by Emley [57] that the increase in strength due to zinc addition results from the formation of a Mg-Zn phase as a degenerate eutectic in the grain boundaries and as "footballs" within the grains, which arise from the sectioned dendritic arms [57]. The formation of "footballs" is mainly due to the tendency of zinc to diffuse into the grains.

Zinc is the strengthening element used to enhance tensile properties. However, Mg-Zn binary alloys are not well suited to the production of castings due to shrinkage porosity although castability increases with increasing zinc content due to an improvement in fluidity. On its microstructural features, Fox [56] reported that the grain size of Mg-Zn alloy was over twice that of Mg-Al alloy. Another limitation of the binary Mg-Zn alloy is that it is not responsive to grain refinement by superheating. Although Mg-Zn alloy is not a material of significance in binary form, the addition of rare-earth and/or zirconium improves overall properties.

3.4.3 Magnesium-Manganese Alloys

Manganese is added to improve the mechanical properties, pressure-tightness and weldability of castings. It is also added to improve the high temperature stability of magnesium components through the creation of micro manganese particles within the structure which restrict grain growth. As a result of this characteristic, Mg-Mn is used in the nuclear energy field to produce fine-grained magnesium fuel cans that are resistant to grain growth at operating temperature. The manganese also helps to minimise the loss of tensile properties on hot forming or annealing [57]. Even though it can offer such advantages, manganese is not use for Mg-Zr or the Mg-RE family of alloys because it has a high affiliation for zirconium and rare earths elements and forms insoluble compounds (i.e. Mn-Zr and Mn-RE) [47][59]. Manganese impairs the fluidity and renders the alloys liable to hot cracking when cast [58].

3.4.4 Magnesium-Zirconium Alloys

Zirconium provides grain refinement and improves the mechanical properties [59]. Zirconium is only soluble in liquid magnesium to the extent of 0.6% to 0.7% but its presence exerts a strong grain refining effect [55][60]. The liquidus temperature of zirconium is at 1852°C, whilst the liquidus temperature of magnesium alloys is usually below 850°C. Therefore, zirconium provides solid nuclei even above the melting temperature of magnesium alloy. The grain refinement is due to precipitation of small zirconium particles which aid the formation of grains [47][57] and as a consequence it provides an alloy amenable to precipitation heat treatment. Another advantage of zirconium is that it reduces the amount of Mg-Zn compound in the grain boundaries so that more zinc can be usefully employed in the alloy [57]. Zirconium is compatible with zinc, silver, and rare earth elements but it is incompatible with the widely used aluminium addition [61].

3.4.5 Magnesium-Rare Earth Metals Alloys

Rare Earth Metals (REM) contribute better creep resistance up to temperatures of 250°C and improve weldability, but give low tensile properties and do not contribute to ductility [58][62]. Rare Earth

additions to Mg-Zn alloy reduce the microporosity and consequently enables high tensile properties to be achieved after full heat treatment. The presence of the relatively low melting point eutectic in the grain boundary network tends to suppress microporosity and consequently improve the casting qualities [9].

Creep resistance, through the addition of REM, is due to a favourably oriented sub-microscopic precipitate forming in the body of the grains at elevated temperature and these reduce grain boundary sliding [57]. REM elements can improve fluidity, resistance to micro-porosity and hot-tearing but they are susceptible to oxidation during melting and casting.

3.4.6 Magnesium-Silicon Alloys

Silicon is known to improve the wear properties of the magnesium alloy. Such improvement is mainly due to the formation of the Mg_2Si brittle compound [57]. However, excessive silicon has been reported to reduce ductility [59].

3.4.7 Magnesium-Silver Alloys

Silver is added to improve the mechanical properties, hardness and weldability [62]. It forms a heat treatable eutectic system with considerable solid solubility. Response to full heat treatment is greatly increased in the presence of REM [57]. There has been little information relating to silver addition to magnesium, this is because the silver addition is only beneficial in the co-presence of REM and zirconium.

3.4.8 Magnesium-Thorium Alloys

Thorium is added to reduce microporosity and hot tearing and improve fluidity and creep resistance up to 350°C, but it is susceptible to oxidation during melting and casting [59]. Thorium addition improves resistance to hot tearing and creep resistance at elevated temperature owing to a favourably orientated precipitate which hinders grain boundary migration [57]. Microscopically, the alloys show discontinuous networks of thorium compound in the form of a degenerate eutectic. These compound networks are distributed between the magnesium dendrites.

3.4.9 Magnesium-Yttrium Alloys

Yttrium is added to confer superior tensile properties, improve corrosion and creep resistance up to 300°C. Yttrium has a maximum solid solubility in magnesium of 12.5% which is significantly greater than that of thorium or REM. The addition of 2% or 4% yttrium was studied by Kaye and Street [58],

who found that the yttrium-containing alloys showed superior creep resistance. Yttrium is expensive and the use of less pure grades is cheaper and, therefore, commercially desirable.

3.4.10 Magnesium-Lithium Alloys

Lithium addition confers superior ductility when added in amounts of at least 10%, the lattice then becoming cubic. Some ternary and quaternary alloys show remarkably high tensile properties which are unfortunately unstable and that is the reason why lithium is seldom used as an alloying element for magnesium.

3.5 General Characteristics of Commercial Casting Alloys

Most commercial magnesium casting alloys can be classified into two main groups (i) lower cost alloys primarily intended for use at moderate temperatures, and (ii) more expensive alloys primarily intended for use at elevated temperatures. These groups may be further subdivided according to the alloying elements which are used, as highlighted in the following sections.

3.5.1 Lower Cost Magnesium Alloys

Any alloy developed for commercial automotive applications or for most consumer products must be cost competitive. Hence the cost criterion immediately limits options to alloy systems containing Al or Zn as the major alloying elements and these form two distinct groups of alloys for general application.

3.5.1.1 Magnesium-Aluminium Family of Alloys

This group is used at moderate temperatures, with aluminium as the primary and zinc, manganese or silicon as the secondary alloying elements. These were the first and main alloys of magnesium to be introduced as structural materials. They constitute about 90% of all structural applications of magnesium and are the least expensive. However, they are generally unsuitable for use above 150°C and considerable losses in strength are evident even at this temperature [57]. The most commonly used magnesium die casting alloy is AZ91 (Mg-Al-Zn series alloys), others are: AM (Mg-Al-Mn series alloys); and AS (Mg-Al-Si series alloys).

3.5.1.2 Magnesium-Zinc Family of Alloys

This group is used for moderate to higher temperatures, where zinc is used as the primary, and very low concentration of rare earth, copper, calcium or zirconium as the secondary alloying elements. The extremely effective precipitation hardening reactions of Mg-Zn binary and the grain refining effect of zirconium combine to yield high strengths with good ductility [57][59]. However these grades are more

costly than the alloys of the AZ series. Typical commercial alloys are ZE41A, ZC61A and ZK51A which are used at temperatures of up to 160°C. This group offers a number of important advantages over AZ alloys: (i) the properties are developed by a low temperature ageing (T5) treatment, (ii) properties obtained in castings are more uniform and less influenced by section thickness, (iii) they are free from stress corrosion cracking and (iv) they have better stability at elevated temperatures [53].

3.5.2 Higher Cost Magnesium Alloys

Where cost is not a major factor, for instance alloy development driven by the military, aerospace or speciality industries, more exotic alloying elements may be added to attain the desired properties. This may open the alloying option to include rare earth, thorium, silver or yttrium as primary alloying elements.

3.5.2.1 Magnesium-Rare Earth, Thorium, Yttrium and Silver Family of Alloys

These alloys are the most expensive and are used when service temperatures exceed 150°C [59]. They have great strength stability at elevated temperatures, mainly attribute to stable grain boundary precipitates. Mg-REM alloys are used at the lower end of the temperature range, while Mg-Th/Ag for higher temperature applications [53]. Typical commercial alloys of this series are ZRE1, EQ21A, QE22A, QH21A, HK31A and HZ32A.

A recent introduction to this series are the Magnesium-Yttrium alloys, i.e. WE54 and WE43. These alloys have room temperature properties similar to QE22 but retain high strength up to 300°C [43][63]. Another virtue of Magnesium-Yttrium alloys is that their corrosion rate is similar to that of AZ91E. The main detraction is that, with the high cost of yttrium, the alloy is expensive for normal use. It has also been found that at a moderate temperature (200°C), for extended lengths of time (example 2000 hrs), there is a pronounced loss of ductility [44].

More specific details regarding these alloys can be found in Busk [53], Miller and Ryntz [59].

3.6 Melting Methods and Process Parameters

In general, the casting process comprises a melting and pouring stage. There are two main methods for melting magnesium alloys, the flux process and the fluxless or gas-shielded process. As a consequence, the type of pouring method and filtering system will be determined by the melting method selected. Additional factors which determine the quality of the cast component are the use of filtering methods and the use of grain refinement in the melt. However, the presence of grain refinement requires further

measures to be taken to prevent the dilution of the refining elements. These methods and process parameters are discussed in the following sections.

3.6.1 Melting Methods for Magnesium Alloys

Magnesium alloys oxidise readily at elevated temperatures and the major requirement, therefore, of magnesium alloy melting is the prevention of excessive oxidation. This is achieved by the use of either a flux cover or a gas-shielded (fluxless) cover.

In general, the use of flux is two-fold; firstly, it protects the metal from oxidation during the initial melting operation, and secondly, it refines the structure by the removal of certain impurities prior to pouring [55]. Gas-shielding employs a cushion of protective gas in place of flux, which protects the molten metal during casting and holding from exposure to air [64]. Air may be completely displaced by helium or argon in this instance. The author's previous work on ZRE1 alloy found that the shielded method gave a much better quality casting than the flux method [65].

3.6.2 Pouring Methods for Magnesium Alloys

As there are two different melting methods, separate pouring methods are used in practice. However, only the fluxless method is included in this survey.

For the fluxless method, final cleaning of the melt surface is carried out at the pouring station using a skimming tool, prior to pouring. The absence of flux makes cleaning of the crucible walls and lips unnecessary. During the pouring operation, oxidation of the metal stream is prevented by the use of a protective gas atmosphere (i.e. Argon). The required gas flow rate depends on the pouring speed, typically 20-40 litres/minute [60] is recommended for commercial melting. However, it was reported by Ha [34] that 5 litres/minute was adequate for laboratory work.

3.6.3 Filtering of the Melt

Without flux, settling of impurities, particularly oxide, is incomplete. As a result, larger metal residues are present in the melt. Therefore a filtering system is required when using the fluxless method to avoid carry-over of oxide inclusions into castings.

3.6.4 The Use of Grain Refinement

In the past, grain refinement for AZ type alloys was achieved by adding tablets of hexachlorethane or hexachlorbenzine [57]. However such chemicals are not used nowadays, as their by-products are reported to be environmentally unfriendly, instead environmentally acceptable titanium-boron tablets

are used to grain refine AZ type alloys [66]. Grain refinement may be achieved by the addition of zirconium to magnesium. Control of the melt process is important for magnesium alloys containing zirconium, because it reacts readily with certain elements such as aluminium, silicon, manganese, iron and hydrogen, and hence it is frequently necessary to add excessive zirconium into the melt [60]. For this reason, it is necessary to maintain a heel at the bottom of the crucible so that insoluble zirconium compounds can settle.

3.6.5 Prevention of Zirconium Dilution in Mg-Zn Alloys

For magnesium containing zirconium alloys, previously discussed in section 3.6.4, the alloying elements tend to be lost during each remelt operation. Hence during alloying, careful composition control is required to prevent dilution and correction can be achieved by adding the pure metal, or hardener alloys with a fairly high content of alloying elements. To achieve maximum saturation, adequate puddling is required [60]. Finally the melt must not be held too long to prevent the loss of zirconium.

3.7 Chemical Properties of Magnesium

The chemical reactivity of magnesium raises particular problems in its application as an alloy for die casting applications. Magnesium in its molten form has a high affinity for oxygen.

$$2Mg + O_2 \rightarrow 2\,MgO$$

This reaction is highly exothermic, and the inflammmability of magnesium was for a long time considered to hinder its use, despite the fact that it is very stable in the solid form [40]. The conditions required for ignition to occur vary as follows: with small pieces (chips or filings) in turbulent air, ignition can occur at a temperature of 310°C. With large pieces and in still air, ignition can only occur about 100°C above the melting temperature of 650°C [67]. This fact makes it clear why it is possible to weld magnesium.

3.8 Post Process Treatment of Magnesium Castings

With more demanding engineering applications, post process treatment has become essential for magnesium castings to realise their potential. This includes both heat treatment to improve properties and corrosion treatment to increase or maintain the life span of the castings in corrosive environments.

3.8.1 Heat Treatment of Magnesium

Heat treatment is conducted to enhance the range of physical and mechanical properties or characteristics obtained in the as-cast condition. Various heat treatment processes can be applied to

castings. However, only stress relief and solution and precipitation treatment will be discussed in greater depth as they influence the properties and cost of components.

The heat treatments commonly employed in commercial practice are: solution heat treatment (T4), stabilising without solution heat treatment followed by artificial ageing treatment (T5), solution treatment followed by artificial ageing treatment (T6) and solution treatment followed by stabilising treatment (T7). In the case of magnesium-zinc base alloy, only the T5 treatment is used. This is due to several reasons:

(i) the solution treatment is not used for Mg-Zn base alloy, because the high solution temperature (520°C) for homogenising the material will cause the low melting point eutectic present in the solid solution matrix to melt, as the eutectic temperature for Mg-Zn alloy is 344°C. Instead a stabilising treatment at 330°C is used to dissolve any concentrated solute remaining in the structure prior to ageing.

(ii) the elevated temperature mechanical properties of the alloys are of particular interest and the T5 treatment is generally known to act as a stabilising treatment to reduce the tendency of castings to grow during service at elevated temperatures [68]. Growth is the increase in volume resulting from second phase precipitation from a solid solution. Thus, the purpose of a stabilising treatment is to complete precipitation, so that only a negligible amount of growth will take place during any subsequent heating in service.

(iii) the Laves phase, $MgZn_2$ (not the equilibrium precipitate, MgZn) is very stable in solid solution [57]. Hence solution heat treatment will not have significant impact on the phase constituents.

3.8.2 Corrosion Treatment

Corrosion problems on magnesium alloys have been greatly improved by the developments highlighted below:

(i) more corrosion resistant alloys, i.e. WE43 [69][70] and high purity alloy AZ91D [63][71].

(ii) the use of special protective treatment in addition to chromate protection and varnish sealing [46][53], i.e. DOW 17, HAE, MAGOXID and more recently the TAGNITE process developed in the US.

(iii) improved designs, i.e. some commercial examples are illustrated by Arlhac and Chaize [46] and Busk [53].

The improved corrosion resistance of alloys like high purity AZ91D is achieved through the reduction of heavy metal elements such as copper, iron and nickel to extremely low levels. Hanawalt et al. [72]

conducted studies on the quantitative relationship between corrosion of alloys in salt water and the presence of phases in the alloy cathodic to magnesium. They reported that there is a definite 'tolerance limit' for iron in magnesium; above this limit the corrosion rate in salt water increases dramatically, as shown in figure 3-2. It had also been reported that the presence of zinc in amounts greater than 3% increases the alloy's tolerance for iron [59]. The corrosion influence of other elements is illustrated in figure 3-3.

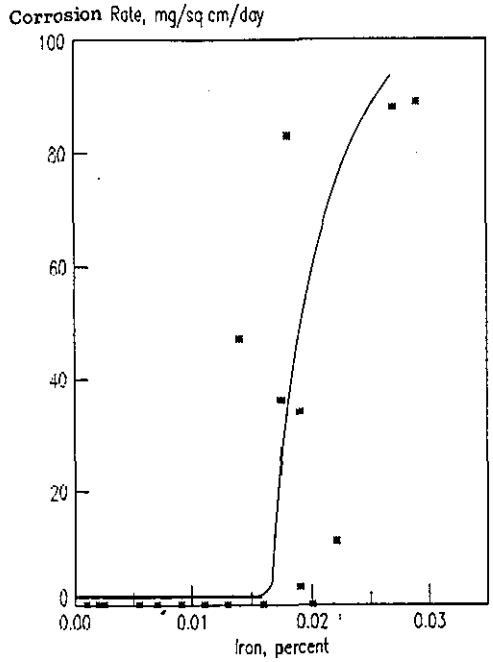


Figure 3-2 Effect of iron on corrosion of pure magnesium (alternate immersion in 3% NaCl) [72].

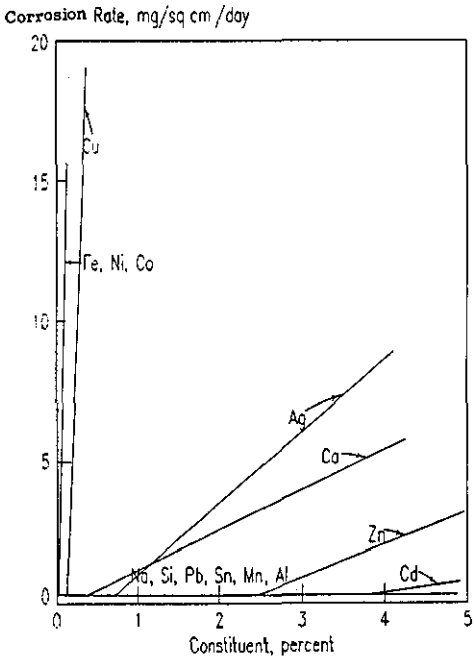


Figure 3-3 Corrosion of magnesium binary alloys (alternate immersion in 3% NaCl) [72].

Some of the common methods available to protect the surfaces of magnesium were summarised by Polmear [9]. Such methods are fluoride anodising, chemical treatments, electrolytic anodising, sealing with epoxy resins, standard paint finishes, vitreous enamelling and electroplating. Due to the hazardous environment that aerospace components are subjected to, a series of corrosion treatment processes are usually necessary for magnesium parts. In some cases a complete treatment scheme that involves chemical cleaning by fluoride anodising, pre-treatment by chromating or anodising, sealing with epoxy resin, followed by chromate primer and top coats is required.

3.9 The Recent Developments of Magnesium Alloys

One of the major concerns about the use of magnesium is its susceptibility to corrosion. In response to this problem a new alloy was developed with corrosion characteristics in salt atmospheres similar to aluminium. This alloy (AZ91E) is a high purity version of the old alloy AZ91C containing 9% Al and 0.75% zinc [67]. The removal of heavy metal elements such as copper, iron and nickel to extremely low levels reduces corrosion in salt atmospheres by a factor of 100 times [44][73]. Several new magnesium alloys are in the development phase. It is known that Magnesium Elektron Ltd is working on low cost alloys with improved elevated temperature properties for the automotive industry [44].

Kamio and his co-workers studied squeeze cast Mg-Ca-Zr alloys and evaluated the effects of calcium and zirconium additions on the microstructures and mechanical properties [22][23]. They reported that there is marked grain refinement with the combined addition of calcium and zirconium and an additional benefit of ignition deterrence during melting and casting.

Recently there has also been the development of more exotic materials such as magnesium-lithium alloys. Such developments are driven by the extraordinary light weight that these alloys possess. For instance a paper by Aida et al. [74] emphasised the possibility of developing such low density alloys for light-weight applications. However, such alloys are still in their developmental stage as poor ambient temperature creep and high corrosion, caused by lithium diffusion at room temperature, limit the commercial potential of this alloy group [75]. Currently, there is intensive development of such alloys at the University of Hannover in Germany and Nagaoka University of Technology in Japan.

One recent significant advancement is the development of the metal matrix composite, where a low density material like magnesium can be significantly strengthened by the addition of thin, stiff and high-strength fibres into the metal matrix. The combination of different materials with distinct properties provides the opportunity to offset the limitations of magnesium alloy, i.e. high notch sensitivity, poor stress properties and low hardness value, as mentioned in section 3.3.2. Examples of such improvement were reported by various researchers:

Ha [34] and Chadwick [33] investigated the effect that various reinforcement elements had on the mechanical properties of magnesium alloy AZ91. They reported that mechanical properties such as hardness, fatigue strength and creep resistance at elevated temperature were greatly improved for the reinforced alloy. Typically the fatigue strength of AZ91, reinforced with 16% vol. Saffil fibre, at 180°C was increased by 100%.

Kagawa et al. conducted an experiment on squeeze cast carbon fibre-reinforced magnesium-matrix composite [76]. They made a comparison between the mechanical properties of pure magnesium (99.5 wt %) matrix and the matrix reinforced with carbon fibres. Their results show that a tensile strength of 1400 MPa was obtained with carbon fibre-reinforced magnesium-matrix composite, as compared to 92 MPa for the unreinforced casting.

Further examples of mechanical property improvements are presented in section 4.9.

3.10 Summary

In general, the literature survey shows that the use of magnesium has increased significantly [42][53][73][77] and that the demand for magnesium is expected to rise in the future [40][41][51]. The future use of magnesium alloys will depend mainly on developments in magnesium alloy technology such as: the development of new extraction methods to address the problems of the present costly and energy intensive electrolysis and metallothermic reduction methods; development of high strength magnesium alloys for both ambient and elevated temperature applications for the aerospace industry; and the development of cost-effective alloys for automotive components.

Based on the literature review, the limitations of poor mechanical properties may be overcome by novel technologies such as metal matrix composites. Fibre reinforcement has shown, in many instances, its potential and promise to improve the overall mechanical properties of the material. As a result, it is likely that the future development of magnesium technologies will focus on this area and the basic principle of metal matrix composites is, therefore, covered in the next chapter (chapter 4).

Chapter 4

Metal Matrix Composites (MMCs)

4.1 Introduction

Metal-matrix composite materials have been so intensely researched over the last century that many new high strength-to-light weight materials have been produced and patented. This chapter presents a brief review of the applications and future of MMCs. The constitution of MMCs and characterisation of fibre composites are outlined. The mechanics of reinforcement and the interactions between the matrix and reinforcement are discussed. The various methods of fabricating MMCs and brief aspects of the fibre system are outlined. Finally, recent developments in MMC technology are highlighted

4.2 Typical Applications and the Future of Metal Matrix Composites

Currently, many aerospace and automotive companies are developing fibre-reinforced materials. Components reinforced with continuous fibres are generally restricted to aerospace applications [78], in spite of their excellent properties and low densities when produced with a light matrix, i.e. aluminium or magnesium alloy. From the view of performance and fuel efficiency, further weight saving can be achieved by using magnesium as the matrix. This is where performance rather than cost is important and where savings in weight and fuel consumption justify the high production cost. In non-aerospace applications, cost and performance are important requiring an optimum combination of these factors. Discontinuous fibres and particle-reinforced MMCs are, therefore, preferred for non-aerospace applications, i.e. automotive components.

An important application of MMCs is the incorporation of short fibres of alumina (Al_2O_3) or alumino-silicates in diesel piston crowns [1]. The replacement of the nickel cast iron by aluminium matrix composite results in a lighter, more abrasion resistant and cheaper product. Several patents have been submitted for this product [79][80][81][82]. Another application involves the use of carbon fibre and alumina particles in an aluminium matrix for use as cylinder liners [2]. This achieves further weight reduction and improves the engine operating efficiency through improved thermal conductivity and reduced friction by improving block stiffness and dimensional stability.

One limitation to the wide spread commercial use of MMC is the cost penalty; the use of MMCs is inevitably more expensive than monolithic alloys [1][11]. The multiplying factor for costs ranges from 7 times for the A356+20% SiC foundry alloy to 24 to 400 times for squeeze infiltrated preform

composites, alumina preforms being the cheapest. In some instances, the cost factor for continuous fibre reinforced composites can be 10,000 times that of the alloys [11]. However, the initial cost penalty may be offset by reduced maintenance cost, longer service life and weight reduction. For example, a significant weight reduction in an aircraft engine shaft can have many cumulative engineering effects [83]. Use of the lighter metal composite can increase load-bearing capabilities, reduce wear, reduce lubrication needs, also increase output speed, all of which result in cost savings. As the process and materials develop with time, the utilisation will eventually benefit from the economies of scale.

Many opportunities for the application of MMCs have been identified, however, a number of barriers must be overcome to ensure widespread introduction of these materials in high production volume automobiles. Allison and Cole [48] classified these barriers as either technology or infrastructural. Final part costs for most MMC components are higher because of the higher costs associated with raw materials, shape fabrication and machining. Technological developments that reduce these costs, such as development of inexpensive raw materials, near-net-shape forming technology, rapid and inexpensive machining processes, and acceptable recycling methods will lead quickly to the acceptance of these materials.

Infrastructural issues refer to the acceptance of these new materials based on economic and human factors. The economic risks for substituting new materials can be high and this makes automotive engineers and planners reluctant to take risk in the uncertain product function, quality, reliability and cost factors in large scale production. The full commitment of top management is required to encourage the implementation of novel materials. As more corporate giants expand the development of MMC technological capabilities, prices should continue to decline. With the promise of cost reductions in the near future, and aided by large metal resources, increasing automation, and rapidly advancing technology, the future commercial development of MMCs seems positive.

4.3 The Constitution of Metal Matrix Composites

Metal matrix composite materials are generally composed of two phases, namely the reinforcement and matrix phases. Reinforcement is introduced to increase the strength, stiffness and wear properties of the metal. The other phase, usually known as the matrix, is present to surround the reinforcing element. Details of these two phases are discussed in the following sections.

4.3.1 Reinforcement Elements in Metal Matrix Composites

The range of materials used as reinforcements is diverse. Composites can be categorised into three

main divisions, namely, particle-reinforced, fibre-reinforced and structural composites. The classification of the composite materials family is shown in figure 4-1.

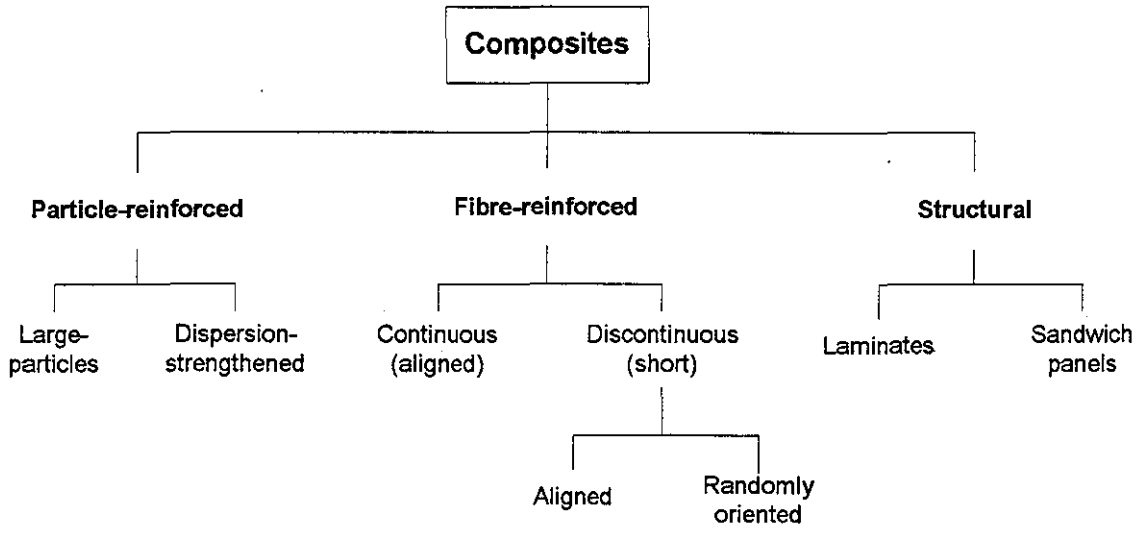


Figure 4-1 Range of Composite forms [84]

On the basis of size and properties, the reinforcement elements can generally be classified into four different groups, namely: whiskers (fibre-reinforced), fibres (fibre-reinforced), particulates (particle-reinforced) and metallic wires (structural). Each of these reinforcement elements is discussed in the following sections.

4.3.1.1 Whiskers

Whiskers are very thin single crystals that have extremely large length-to-diameter ratios. They are considered to be one of the strongest known materials due to their high degree of crystalline perfection. In spite of their high strength, whiskers are not utilised extensively as a reinforcement medium because they are extremely expensive [85]. In addition, it is difficult and often impractical to incorporate whiskers into a matrix. Examples of whisker materials are graphite, SiC, silicon nitride, and Al_2O_3 [84][86].

4.3.1.2 Fibres

A fibre is characterised geometrically not only by its high length-to-diameter ratio but also by its near crystal-sized diameter. Despite the presence of minor crystalline imperfections, a fibre is still considered to be a very strong element. As a consequence of their lower cost, fibres have been widely used commercially to achieve a high level of reinforcement. There are generally two forms of fibre materials namely:

(i) Continuous fibre reinforcement is known for its excellent anisotropic mechanical properties. In terms of component cost, it is the most expensive, mainly due to the extra care required during fabrication [87].

(ii) Discontinuous fibre reinforcements have lower properties compared to continuous fibre reinforcement. Nevertheless, they offer the most significant improvement in isotropic properties when the fibres are placed "randomly" in the matrix. In terms of cost, they lie in the middle-priced region of the composite range [78].

Typical fibres are Al_2O_3, boron, SiC and carbon [84][86].

4.3.1.3 Particulates

Particulate reinforced metals provide the least expensive of reinforced parts and lie at the opposite end of the cost/performance spectrum [88]. Particulates can be seen as a three-dimensional reinforcement that can lead to isotropic material properties, since the material is symmetrical across three orthogonal planes. Strength of the particulate composites normally depends on the diameter of the particles, inter-particle spacing and the volume fraction of the reinforcement. In spite of its cost advantage over other reinforcement elements, strengthening of the matrix does not occur even with a very high particle content. This is because particles do not provide the same aspect ratio as fibres, as the length of the particles is too short to transfer sufficient load by shear stresses from the matrix to the particle. The most common ceramic particulates are SiC and Al_2O_3 [1][84].

4.3.1.4 Metallic Wires

Wires have been used generally as radial steel reinforcement in automotive tyres and in filament-wound rocket casings [84]. Metallic wires are fine wires that have relatively large diameters, typical materials include steel, molybdenum, and tungsten. However this form of reinforcement, in metal matrix composites, does not provide the ideal composite strength as the reinforcement diameter is too large to enable significant strengthening of the material. Ibe [86] and Rauch [89] have demonstrated that composite strength generally decreases with an increase in fibre/wire diameter.

4.3.2 Functions of Matrix Phase

The matrix phase of fibre composites serves several functions [90]. First, it binds the fibres together and acts as the medium through which an externally applied stress is transmitted and distributed to the fibres. The second function of the matrix is to protect the individual fibres from surface damage as a result of mechanical abrasion or chemical reactions with the environment. Finally, the matrix separates

the fibres and, by virtue of its relative softness and plasticity, prevents the propagation of brittle cracks from fibre to fibre, which could result in catastrophic failure. In other words, the matrix phase serves as a barrier to crack propagation.

4.4 The Characterisation of Fibre Composites

Several attributes are used to characterise composite types. Each of the categories is also a primary variable in fibre composite materials. The factors that determine the classification are elaborated in the following sections.

4.4.1 Fibre Length

Fibres can be continuous, discontinuous, or both, within a given composite material. However, the fibre length or the technically preferred fibre aspect ratio (length to diameter ratio, *l*/d) can have a pronounced effect on the properties and on the failure modes of the composite material. In most cases a critical fibre length, *l*c is necessary for effective strengthening and stiffening of the material [91].

If the fibre length is less than the critical length (i.e. $l < l_c$), there will be a greater tendency for fibre pull out from the matrix during fracture, resulting in poor mechanical properties [91]. Conversely, when the fibre length (l) is greater than l_c, the fibres will fracture rather than pull out and this contributes higher strength to the composite. This is generally due to the greater contribution of fibre strength to the matrix as the fibre length increases, since short fibres in a composite cannot be directly loaded at their ends and stress must therefore be transferred into them by shear forces at the fibre-matrix interfaces [92].

In addition the fibre length will also affect the ability of the fibres to be aligned and packed. Usually short fibres are more difficult to align and to pack densely than are continuous fibres. In most cases the maximum volume fraction for discontinuous fibres is usually no more than 50 - 60% [93]. The difference between continuous fibre and discontinuous fibre is the fibre aspect ratio. Fibres for which $l >> l_c$ (normally $l > 15\ l_c$) are termed continuous, whereas discontinuous or short fibres have lengths shorter than this [84].

4.4.2 Fibre Volume Fraction

Fibre volume fraction may be described as the fibre concentration within the composite materials. For any fibre reinforcement to occur, the fibre volume fraction present in the composite must be above a certain minimum value, V_{min} [90][94]. If the volume fraction of fibres is above the minimum value V_{min}, the Rule of Mixtures (ROM) may well apply to the system. A review of ROM is provided in section 4.5.1. However, the fibres do not lead to strengthening until a critical volume V_{crit} is reached,

which is slightly larger than V_{min}. Fibre fraction beyond this critical volume will improve composite strength. A typical V_{min} value of around 7% has been reported by Rauch et al [89], while Kamado et al. [37] reported V_{crit} = 13% for reinforced Mg-4.2%Zn-RE alloy with alumina short fibres. Detailed information on the effects of volume fraction can be found in Anderson [91] and Kelly et al. [90][94].

4.4.3 Fibre Diameter

The diameter of the fibre usually ranges from less than 1 μm to over 250 μm [93]. Griffith [95] studied the effect of glass fibre diameter on the composite and showed that smaller diameter fibres are much stronger, especially at a diameter of less than 100 μm. The lower possibility of crystalline imperfection for a smaller diameter fibre results in a stronger fibre element.

4.4.4 Fibre Orientation

The orientation of the fibres within the matrix is the prime variable in achieving the desired directional properties on the composite part. Maximum loading of the fibres and composite strength occurs when all the fibres are aligned in parallel to the tensile load [91]. However the composite is extremely weak when the tensile stress is applied normal to the axis of the fibre. This indicates that the transverse strength is very low for aligned fibres, in this instance their properties are known to be anisotropic. Continuous fibre is known to fall into this category, i.e. having extremely high strength when load is applied axially to the fibre, but having poor shear properties along the aligned fibre axis [96]. As a result, the undirectional properties of continuous reinforced composites tend to limit their applications. In contrast, the discontinuous reinforced composites (randomly orientated) have slightly lower mechanical properties than the former, but exhibit greater isotropy. This is probably the main reason for the increased interest in using discontinuous MMCs for practical applications in recent years [1][97].

4.4.5 Fibre Shape and Surface Finish

The fibre shape can, to some degree, affect the composite properties. For instance, the fibre cross-sectional shape and taper affect the magnitude of the stresses in and around the fibre-matrix interface. In addition, the surface finish of the fibre will inevitably have some influence on the wettability. Mileiko [98] conducted a study on the influence of surface roughness on wetting and found that wetting decreases as surface roughness increases.

4.4.6 Fibre Density and Properties

The density of most ceramic fibres is slightly higher than that for magnesium alloys and there will be a weight penalty if large amounts of fibre are introduced. Of the fibre materials, only carbon has a

density below that of magnesium. Table 4-1 presents the properties and density of some commonly used fibres for the reinforcement of magnesium alloys.

Property	Magnesium (e.g. RZ5)	Carbon (e.g. RK10)	Saffil™ (e.g. Al_2O_3)	Boron
Density (g/cm^3)	1.84	1.8	3.3	2.4
Tensile Strength (MPa)	200	2200	2000	4000
Youngs Modulus (GPa)	44	250	300	390
Fibre Diameter (μm)	-	8 (mean)	3 (mean)	140
Fibre Length (μm)	-	120 (mean)	500 (mean)	continuous
References	[101]	[102]	[103]	[86] [92]

Table 4-1 Properties of Magnesium alloy and Fibres

4.5 The Mechanics and Interfaces of Reinforcement

In order to fabricate strong composites, both the chemistry and mechanics of composite materials must be considered. As a result, the chemical and mechanical interactions are of prime importance, especially with regard to the reaction occurring at the fibre-matrix interface. Interfacial reactions between the fibres and matrix can damage the fibres. However, some degree of reaction is desirable in order to provide adequate interfacial bonds. The following sections discuss issues relating to the mechanics of reinforcement and the chemical interaction between fibres and the matrix.

4.5.1 The Mechanics of Reinforcement

Based on the Rule of Mixtures, a linear relation exists for both the modulus and the tensile strength of the composite relative to the fibre volume fracture, V_f [90]:

$$E_{c\,(cont)} = E_f . V_f + (1 - V_f) E_m \quad \text{----------} \quad (1)$$

$$\sigma_{c\,(cont)} = \sigma_f . V_f + (1 - V_f) \sigma_m \quad \text{----------} \quad (2)$$

where E is the modulus, σ is the strength, V is the volume fraction, and the subscripts c, f and m refer to the composite, fibre and matrix respectively. However the theoretical composite strength from equation (2) is never achieved in practice and, in most cases, the experimental results tend to fall below the theoretical values. Such observations were reported by Kagawa [76] and Ibe [86]. They generally found that alignment problems with the fibres, orientation deviation, and insufficient bonding causes

the actual fibre strength to be lower than the theoretical fibre strength. Therefore, proper control of the process conditions is of a prime importance to achieving full composite strength. It should be noted that the equations (1) and (2) only apply for continuous aligned fibre composites with load being applied longitudinally, i.e. in the direction along the aligned fibres. More complex equations exist for short fibres. The strength of random oriented discontinuous short fibre composites is even harder to predict because more variables are involved. For example, the precise fibre orientation in each batch of random oriented short fibre composite is often uncertain.

Theoretical analysis for random oriented short fibre composite systems is very limited and much of the available work has arisen through the analysis of the effect of fibre misalignment on composite strength. As a result, the Rule of Mixtures for discontinuous fibre composite must be modified. If it is assumed that the load on a given fibre is due only to the shear stress at the fibre-matrix interface, then a critical fibre length *lc* can be defined by [90]:

$$\frac{l_c}{d} = \frac{\sigma_f}{2\tau} \qquad \text{----------} \qquad (3)$$

where d is the diameter of the fibre and τ is the shear stress at the fibre-matrix interface which can be taken as equal to one half of the matrix yield strength σ_{Em} [90][104], i.e.

$$\tau = \tau_{Em} = \sigma_{Em}/2 \qquad \text{----------} \qquad (4)$$

If the fibre is shorter than this critical length, the composite will fail by plastic flow of the matrix; if it is longer, fracture of the fibres will cause the failure. The strength of discontinuous aligned fibre composites can subsequently be rewritten according to the fibre length classification [105]. Hence the modified rules of mixtures for randomly oriented fibre are [94]:

$$l \geq l_c \qquad \sigma_{c\,(disc.)} = C\,\sigma_f v_f \left(1 - \frac{l_c}{2l}\right) + \sigma_m (1 - V_f) \qquad \text{----------} \qquad (5)$$

$$l < l_c \qquad \sigma_{c\,(disc.)} = C\,v_f \frac{\tau l}{d} + \sigma_m (1 - V_f) \qquad \text{----------} \qquad (6)$$

C is the fibre orientation factor for fibres randomly oriented in three dimensions. Various C values were suggested. Lim et al. [106] employed C = 0.25 in their calculations, while Friend [107], and Cox [105] suggest values of C = 0.2 and C = 0.17 respectively.

4.5.2 The Interactions between the Metal Matrix and Reinforcing Material

The interactions between metal and fibres are a major concern in relation to the composite strength. The reactivity between the two constituents of a MMC (metal matrix and ceramic reinforcement) depends on a number of reactions in which both are involved, namely: (i) the standard free energy changes ($\Delta G°$), (ii) interfacial reaction, (iii) surface energy and (iv) kinetics of infiltration (presented in 5.5.1). Hence in this section these interactions are reviewed.

4.5.2.1 The Thermodynamics of Oxidation

With reference to standard free energy changes relating to the formation of metal oxides, any element (in pure or in alloy form) lower than the Al/Al_2O_3 free energy change should 'wet' Al_2O_3 by reduction of Al_2O_3 to aluminium [108]. Examples of suitable elements include zirconium, magnesium, cerium, lanthanum, lithium, calcium and barium. Thermodynamics is useful in predicting what reaction is likely to occur under standard state conditions. However, in a casting melt the reaction(s) are far from equilibrium and most cast materials are not 100% pure.

4.5.2.2 Interface Bonding and Chemical Reaction in MMCs

It is essential that the adhesive bonding force between fibre and matrix be high to minimise fibre pull-out. It has been reported by many researchers that bonding strength is an important consideration in the choice of the fibre-matrix combination [35][84][109]. Where the UTS of the composite depends to a large extent on the magnitude of this bond, adequate bonding is essential to maximise the stress transmission from the weak matrix to the strong fibres. It can be seen that magnesium is a highly reactive material and has high affiliation with oxygen; it is liable to many interfacial bonding types including mixed bonds. Metcalfe [110] proposes six basic bonds between matrix and reinforcement phase in metal matrix composites, namely: (i) mechanical bond, (ii) dissolution and wetting bond, (iii) reaction bond, (iv) exchange reaction bond, (v) oxide bond. A combination of any of the five bond types is also possible and this will lead to (vi) mixed bonds.

When a reaction bond is created, a new chemical compound is formed at the interface of the matrix and reinforced phases. Oxide reinforcements (such as Al_2O_3) in magnesium form oxide bonds which enhance the bond between the magnesium matrix and the oxide fibres. Fibre-matrix interface reaction studies conducted by Kamodo and his co-workers [19][20][37] on Mg-Zn alloy with Al_2O_3 fibres, showed evidence of MgO as a reaction product at the interface between the matrix and reinforcements. They reported that these magnesium oxide layers with a thickness of 0.1μm to 0.3μm provide good interface bonding. Petitcorps [111] attributed these reactions to magnesium diffusing into the Al_2O_3

skin (by diffusion) which chemically reacts with the fibre to form thermodynamically favourable phases like the spinel Mg_2SiO_4 or Al_2O_4Mg, oxides (MgO), or intermetallics (Mg_2Si).

In order to achieve good bonding there must be adequate wettability between the reinforcements and the metal. This is generally affected by the surface energy within each element. The principle and effect of wetting is discussed in the next section.

4.5.2.3 The Principles of Wetting

The wettability of a solid by a liquid is indicated by the contact angle θ which is related to the three surface tensions γ_{sg}, γ_{sl} and γ_{lg}. Where γ_{sg} is the surface energy of solid, γ_{sl} is the solid-liquid interfacial energy and γ_{lg} is the surface energy of liquid. The basic relationship between contact angle and surface energy in equilibrium can be expressed by the Young-Dupré equation [112]:

$$\gamma_{lg} \cos\theta = \gamma_{sg} - \gamma_{sl} \qquad \text{----------} \qquad (7)$$

Based on the Young-Dupré equation, the angle of contact θ must be less than 90°, to obtain wetting of the fibre (solid) by the molten metal (liquid), this can be achieved by raising the surface energy of solid γ_{sg} or lowering the solid-liquid interfacial energy γ_{sl}. In practice, this can be achieved by coating the substrate with a higher energy element [87]. Another alternative to improve the wetting effect is to lower the metal surface tension γ_{lg}, and this is achieved by selecting a metal or using alloying additions to lower the surface tension of the alloy (e.g. bismuth, lead, and titanium in aluminium) [86]. Methods to further improve wetting such as heat treatment of fibres/particles and ultrasonic irradiation of the melt were mentioned by Rohatgi et al. [113].

4.6 Methods for the Fabrication of MMCs

The fabrication techniques for metal-matrix composites depend very much upon the choice of fibre and matrix. There are, in general, three main fabrication methods and these are [114][115]:

(i) ***Fibre surface coatings and composite preforming technique, which includes:*** chemical coating, chemical vapour deposition, physical vapour deposition [87], spraying deposition [88] and electrochemical plating.

(ii) ***Solid-phase fabrication methods, which includes:*** diffusion bonding, hot rolling, extrusion and drawing, hot isostatic pressing, powder metallurgy [36][88], high speed hot pressing and explosive welding

(iii) ***Liquid-phase fabrication methods, which includes:*** infiltration under atmospheric pressure, infiltration under inert gas pressure [18][116], vacuum infiltration [117], combination of methods [118], squeeze casting [14][27][88][119][120], vortex method [115], rheocasting and compocasting [121][122][123].

However, from this range of MMC fabrication methods, the two most established methods are powder metallurgy and squeeze casting [36][109][114][124]. In most cases, powder metallurgy is usually not recommended for fibre reinforcement, this is mainly due to the fact that the high applied pressure used in the process will cause fracture of the fibres. Extensive fibre damage will generally lead to poor fibre properties, and in some cases, the fibres may fracture and become shorter than the critical fibre length which defeats the purpose of fibre reinforcement as there is no difference from a particulate composite.

Other methods, such as vacuum infiltration under insert gas pressure [125] and the vortex method [28][31][32] have been studied. However these methods are only in their research phase, mainly because the squeeze casting process exhibits far greater benefits in fabricating reinforced composites. Further elaboration of the squeeze casting process is provided in chapter 5. More specific details relating to the range of fabricating methods listed above for MMCs, can be found in Chou et al. [114], Fridlyander [126] and Zhu [122].

4.7 Fibre Preform for MMCs

One of the most commonly used methods of casting MMCs involves the injection of liquid metal into the interstices of an assembly of short fibres, usually in a pre-arranged fibre orientation block, known as a 'preform'. This method of incorporating fibres into the matrix has been widely used in industry over recent years, due to the significant advantages offered, namely:

(i) it significantly reduces the contact time between the fibres and the molten magnesium, which eventually provides better control of the reaction at the fibre-matrix interface. This is particularly significant for magnesium, as this material is known to be very reactive in its molten stage.

(ii) it provides the possibility of creating localised reinforcement, which allows fibre reinforcements to be used only in critical areas. This also offers the ability to obtain the maximum performance with the least cost.

(iii) it is able to provide an even distribution of fibres within the cast volume, generally because the preform is produced before casting. In this way, it provides better control of the fibre distribution within the preform volume and at the same time pre-determines the fibre distribution and enables its volume fraction to be measured. It was reported by Toaz et al. [99] that, even for fibre volume fractions as low as 5%, the preform is durable enough to be easily handled without fear of damage.

4.7.1 Fibre Preform Manufacture and Infiltration

The fabrication of short fibre preforms involves several stages of processing [87]. These include mixing the short fibres homogeneously in water accompanied by a small addition of binder. The fibre-suspended liquid is next decanted into an open-topped die having fine drainage holes around the base. The liquid is subsequently removed by suction while the residue is compressed by a ram. In this case the applied pressure will influence the fibre volume fraction (usually in the range of 5% to 30% for random oriented short fibres).

4.7.2 The Selection of Suitable Fibre Types

One of the main criteria which influences the selection of fibre types is the compatibility with the matrix. It is especially significant for a reactive matrix like magnesium. Rohatgi [127] outlined the range of suitable fibres for each type of matrix. The fibres that were recommended for magnesium are carbon, boron and alumina (Al_2O_3). In addition to Rohatgi's report, there has been a significant amount of research work on magnesium composites which has focused on those fibre elements. A review of this work is provided in section 4.9.

4.7.3 The Selection of a Binder System

In order for the preform to retain its integrity and shape, it is often necessary for a binder to be used. This is particularly true when the fibre preform has less than 10% volume fraction of fibre, as it is difficult to infiltrate the molten metal without excessive deformation of the preform [27].

Silica and alumina-based binders are the most widely used mixture in most preforms, mainly due to their high temperature properties [87]. The binding agent is normally introduced via the suspension liquid so that it deposits or precipitates on the fibres, often forming preferentially at fibre contact points, where it serves to lock the array into a strong network. Typically, a binder might be present in the preform at levels of about 5% to 10% by weight [128].

There has been interest in the chemical effects induced by the presence of the binder. It was reported that Mg_2Si and MgO phases were formed by a chemical reaction between the magnesium alloy materials and the silica (SiO_2) coating of reinforcements [129]. Such instances of silica binder being rapidly attacked by magnesium in the melt during squeeze infiltration were reported by MEL - Norsk Hydro - ICI [130], Cappelman et al. [131] and Li et al. [132]. The attack on the silica generally affects both interfacial properties and matrix age-hardening characteristics [133].

4.8 Heat Treatment of MMCs

Most casting alloys are heat treatable and this process is worthwhile when the mechanical properties can be appreciably improved by a suitable heat treatment process. In contrast to the extensive knowledge on the influence of heat treatment on the tensile properties and fracture behaviour of aluminium matrix composites, little is known concerning the effect on magnesium matrix composites.

A collaborative project between MEL - Norsk Hydro - ICI [130] studied the effects of heat treatment on various magnesium binary alloys with 20% vol. fraction Saffil™ fibre. It was reported that the standard heat treatment (T4) for commercial alloy produces a high degree of reaction at the fibre-matrix interface and, as a result of this reaction, a significant reduction in the overall mechanical properties of the MMC was experienced.

A similar experience was reported by Kainer [16] who studied the influence of heat treatment on the properties of short-fibre-reinforced magnesium composites using AZ91 (Mg-Al family alloy) or MSR (Mg-Zr family alloy) matrices. The results generally showed that heat treatment of AZ91 matrices, in general, reduces the mechanical properties of the composites. The reason is that the bond at the fibre-matrix interface is impaired which subsequently leads to a marked decrease in the tensile strength perpendicular to the fibre orientation. On the other hand, it was found that composites with the MSR matrix showed an increase in mechanical properties when heat treated. The reason given was that the alloying elements containing silver, rare earths and zirconium prevented an attack of the fibre at high homogenising temperatures.

Petitcorps and his co-workers [111] studied both the effect that alloying elements and T6 treatment had on the adhesion between the fibres and Al-Si-Mg matrix. They reported that heat treatment increased the interface reaction and caused interfacial shear strength to increase. Nevertheless, they warned that over-aging will cause magnesium to attack the fibres and subsequently cause fibre degradation.

4.9 The Recent Developments of MMCs

There has been continuous research on hybrid composites which are obtained by using two or more different fibres in a single matrix [84]. Such hybrids are known to have a better all-around

combination of properties than composites containing a single fibre type. Examples of such hybrids have been implemented by Honda Co. Ltd [2][4]. They used an alumina-carbon hybrid composite for their engine block liner and reported that it provided improved strength and wear resistance.

In another development, Lim [109] investigated a hybrid process which combined the squeeze and investment casting processes to infiltrate liquid metal into a ceramic shell mould. He reported that this combination exploited the best features of each process to provide alloy flexibility, complex casting capabilities (through the flexibility of a shell mould) and better casting integrity (through the applied pressure of squeeze casting).

The majority of the development work on metal matrix composites has been concentrated on aluminium based alloys. Other alloys, such as magnesium base alloys, have also been studied but on a smaller scale. Research work has been carried out in Germany, UK, US and Japan, but the work has concentrated on AZ91. Other alloys such as AS41, AM20 and Mg-Zn alloy have been studied, but on a smaller scale. Some of the research findings are summarised below.

Kainer et. al. [35][36] investigated the mechanical behaviour of magnesium alloy MMCs. The material used for the studies was the magnesium alloy AZ91, which was reinforced with alumina (Al_2O_3) short fibres and silicon carbide particles using the powder metallurgy and squeeze casting processes. Their results showed that squeeze cast alumina fibre test specimens exhibited higher tensile strength and 0.2% proof stress at elevated temperature (200°C) than the alternative powder metallurgy specimens. In addition, the results showed that the alumina fibre test specimens demonstrated improvements of 50% at ambient temperature and 100% at elevated temperature for both the U.T.S. and 0.2% proof stress compared to the unreinforced test specimens.

In another study, Kainer and his co-workers [134] conducted an experiment on squeeze infiltrated carbon fibre preforms with AS41 and AZ91+Ca alloy. They reported that the creep rate of fibre reinforced AS41 is only one-tenth compared to that of the unreinforced alloy. In addition, there was only minor improvement for the tensile properties of reinforced AZ91+Ca alloy with 20% vol. carbon fibres, displaying 225 MPa to 260 MPa at ambient temperature and 120 MPa at 250°C.

Singer et al. [18][135] conducted a series of investigations relating to the effects of continuous carbon fibre reinforcement on the mechanical properties of magnesium alloys, AZ91 and AM20. The composites were manufactured by a gas pressure melt infiltration process and the volume fraction of fibre reinforcement was approximately 63%. They reported that a tensile strength of 1049 MPa and modulus of 249 GPa were obtained, compared to 132 MPa and 45 GPa for the unreinforced casting.

In another study, Singer et al. [116] investigated the isotropic properties of the 0/90° woven fabric C/Mg-composite. They reported that strength and stiffness for 0/90° AM20/Carbon fibres (T300J) increased linearly with the fibre volume fraction. Tensile strength in the direction of fibre alignment was three times higher than that in unreinforced AM20. However, the strength at ±45° to the fibre orientation was only slightly higher than the matrix.

Kamado et al. [136] investigated the influence of alumina short fibres on AZ91D magnesium alloy fabricated using the squeeze infiltration method. They reported an ambient temperature UTS of less than 200 MPa, while T6 treatment improved the strength to 260 MPa. In another study, Kamado et al. [21] investigated the effects that various alloying elements had on Mg-Zn alloy. They found that a neodymium addition to Mg-4%Zn with 14% vol. Saffil fibres improved its UTS to 230 MPa whereas the as cast properties of the Mg-Zn alloy and MMC were a mere 150 MPa and 200 MPa respectively. They also studied the influence that Saffil fibre volume fraction had on ZE41 commercial alloy [37], and their results indicate that V_{crit} for this composite system is 13%. Their results also show that UTS increased with increasing fibre volume fraction, where 200 MPa was obtained for 14% vol. fraction and 275 MPa for 24% vol. fraction.

Others studies conducted by Kamado and his co-workers [19][20][137] evaluated the properties of Mg-Zn-Ca composites reinforced with aluminium borate whisker and hybrid alumina short fibres + aluminium borate whisker. They reported that 28% vol. fraction aluminium borate whisker reinforced composites had higher tensile properties at ambient temperature (as-cast UTS of 350MPa), but that this decreased significantly with an increase in temperature. They subsequently found that this problem could be overcome by mixing aluminium borate whisker with alumina short fibres.

4.10 Summary

In this chapter the principles, characteristics and uses of metal matrix composites have been reviewed, together with different types of reinforcements. Based on the literature review, the discontinuous and randomly oriented fibre-reinforced composite-type was chosen for the research work. This form of reinforcement offers the best "value to strength ratio" and it is one of the easiest to incorporate into a matrix by most fabrication processes.

From the literature review in section 4.6, on the extensive range of fabrication processes for producing a discontinuous short fibre composite, the squeeze casting process was selected mainly because it exhibits far greater benefits in fabricating reinforced composites. The following chapter (chapter 5) will elaborate on this process.

On the review on fibres selection in section 4.7.2, it was found that carbon, boron and alumina (Al_2O_3) fibres are the most compatible with magnesium. Hence the primary casting programme will study the compatibility and ease of fabrication of these fibre elements with magnesium.

In general, the literature survey showed that the use of metal matrix composites has increased significantly over the past few years and the future demand of reinforced components is expected to rise [1]. As a result, the future use of MMC will depend largely on the development of composite technology such as the development of new fabricating methods, to address the present problems of inconsistency in composite production, and development of suitable fibre elements that are compatible with the highly desired low density material, magnesium. Hence the future expansion of the fibre reinforced industry will largely depend on the development of those processes mentioned earlier.

Chapter 5

The Squeeze Casting Process

5.1 Introduction

This chapter presents a brief highlight of the applications and future potential of squeeze casting. A description of the squeeze casting and squeeze infiltration processes will be presented. The features and the important parameters of these processes are outlined. Finally a review of some of the recent research work is provided.

5.2 Typical Applications and the Future Potential of Squeeze Casting

Squeeze casting is a hybrid of conventional casting and forging techniques and combines the strength and confidence levels of forging with the economics and shape capabilities of casting. It shows a potential for cost reduction in the fabrication of large batches. The process has been used for a wide range of metals, ranging from the lowest melting point alloys of lead and zinc, to the very high melting point metals such as iron and nickel, but most effort has been concentrated upon light metal alloys (aluminium) [138]. Due to its characteristic of pressure assistance, this process is also used for the production of MMCs.

Two of the best known products which have benefited from this process are automotive hubs [139] and pistons [79][80][81][82][140][141] cast from aluminium-based alloys. Other applications are [1][139]: (i) defence components (wheels, shaped armour, missile nose cones), (ii) automotive cylinder liners for high performance engines, (iii) automotive brake calipers, (iv) automotive suspension components, (v) rail vehicle structural members (vi) constructional support members (bridge brackets), and (vii) piston connecting rods.

In terms of casting size and weight range, the smallest reported component was 125g, and the largest to date from GKN Technology Ltd weighed 35 kg. On average, components weighing 18 kg are made on a regular basis by GKN [142].

Over the years, the squeeze casting process has been proved to be an ideal way to produce near-net-shape, high quality components for the automotive industry in both conventionally cast and wrought alloy compositions. The superiority in properties of squeeze castings over those produced by the conventional casting process and die forging process is apparent. However, there are factors yet to be

considered and problems to be solved in applying this process to some specified alloys or product applications.

The increasing use of the squeeze casting process for fabricating MMCs will compel the development of more sophisticated control systems. This in turn, will invariably lead to better product quality and a more economical production rate for a future squeeze casting process. Therefore continued research to establish information regarding the effects of process parameters on component properties is required, in order to expand the use of the process.

5.3 The Process Description for Squeeze Casting and Squeeze Infiltration

In general, there are two variants of the squeeze casting process. The two variants are the "direct" and "indirect" squeeze casting methods. These differ in the way in which the molten metal is metered and in the way movement of the molten metal is controlled, as a consequence the attainable quality of the castings produced from the direct and indirect casting methods also differs. In 'indirect' squeeze casting, metal is injected into the (closed) die cavity by a small diameter piston which also exerts sustained pressure during solidification. The technique is therefore a hybrid process between low pressure die casting and high pressure die casting and as such does not possess the attributes of the direct process.

Further details of the indirect squeeze casting may be found in the paper by Suzuki [143]. In this thesis, however, only the principles relating to direct squeeze casting will be discussed. The principal advantage is that the direct action of pressure on the solidifying metal is sufficient to prevent the occurrence of either gas or shrinkage porosity. Squeeze casting is best classified as a hybrid process of gravity die casting, pressure die casting and the forging process, this being illustrated in figure 5-1.

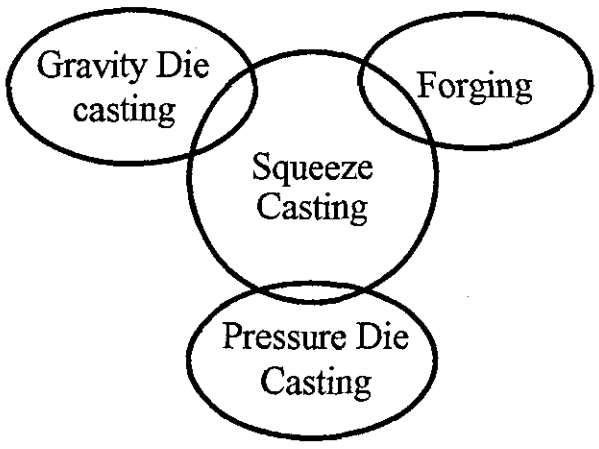


Figure 5-1 Relationship of Squeeze Casting to other metal forming processes

5.3.1 The Squeeze Casting Process (for Alloys)

There are generally four stages involved in the squeeze casting process [142], the steps are as follows:

(i) An accurately metered amount of molten metal is poured into a preheated die cavity located on the bed of a hydraulic press.

(ii) The press is activated to bring down a top die or punch to close off the bottom die cavity and pressurise the liquid metal. This is done as soon as possible after pouring to minimise the amount of solidification occurring without pressure.

(iii) The pressure is held on the metal until solidification is complete. This forces the metal into intimate contact with the die faces, thereby increasing the heat flow rate significantly. Most importantly, the pressure encourages any micro/macro-shrinkage cavities to be filled up.

(iv) Finally, the punch is withdrawn and the component is ejected and removed from the die cavity, which is made ready for the next operation.

A schematic of the squeeze casting process used in this research is shown in figure 5-2.

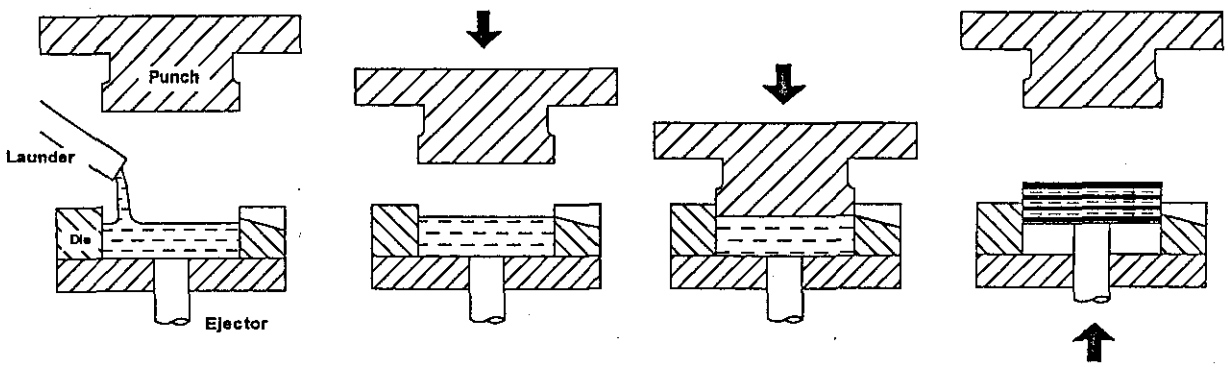


Figure 5-2 Schematic of the Squeeze casting process

5.3.2 The Squeeze Infiltration Process (for MMCs)

The squeeze infiltration process is similar to the squeeze casting process, the only difference is the involvement of the fibre preform. Again the process for squeeze infiltration of metal matrix composites can be generally divided into four stages, the steps are as follows:

(i) positioning of a preheated preform into a heated steel die

(ii) quiescent pouring of molten alloy into the die

(iii) application of pressure in order to infiltrate the preform and to solidify the casting

(iv) extraction of the casting

A schematic of the squeeze infiltration process used in this research is shown in figure 5-3.

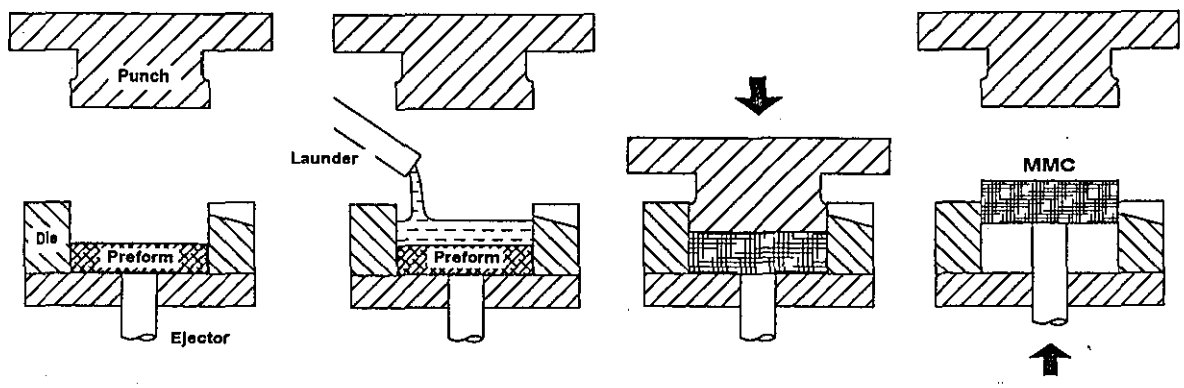


Figure 5-3 Schematic of the Squeeze Infiltration process

5.4 Features of Squeeze Casting Process

Squeeze casting is based upon the principle of pressurised solidification wherein finished components can be produced in a single process from liquid metal to solid components utilising re-useable dies. The constant applied pressure produces microstructures that display fine grains and an absence of gas and shrinkage porosity. The following sections detail how these features are achieved and their influence on heat treatment is presented in section 5.4.3.

5.4.1 Absence of Gas and Shrinkage Porosity in Squeeze Cast Components

During solidification, once the initial pressure of the dissolved gas in the melt is greater than the atmospheric pressure, metallostatic pressure and capillary pressure added together initiate gas bubble nucleation. Upon solidification, some of these gas bubbles become trapped in the metal and form gas porosity. The pressurised solidification in squeeze casting prevents the dissolved gases from nucleating thus preventing the formation of gas porosity in the casting.

The absence of shrinkage porosity in squeeze cast components is attributed to the continual application of pressure throughout solidification which feeds, by force, the molten metal and/or semi-liquid metal into the voids thus producing a fully dense material.

5.4.2 Fine Grain Size in Squeeze Cast Components

As the solidification times are much shorter than for many casting processes [144], fine grained structures with small dendrite cell sizes are a feature of squeeze cast materials [28][29][30][139] [145][146]. The excellent grain refinement is achieved by a combination of the thermodynamic effect (modelled by the Clausis-Clapeyron equation) and high heat transfer coefficient.

5.4.2.1 Thermodynamic Effect in Squeeze Casting

The application of high hydrostatic pressure during solidification will affect the melting point of a material. The expected degree of change in freezing temperature of a material with increasing pressure is indicated by the Clausius - Clapeyron equation [144]:

$$\frac{dT}{dP} = \frac{T_f (V_L - V_s)}{L_f} \qquad \text{----------} \qquad (8)$$

where, T_f is the equilibrium freezing temperature
V_L is the specific volume of the liquid
V_s is the specific volume of the solid
L_f is the latent heat of freezing

According to the above equation (8), the application of pressure to a pure metal which contracts on freezing (most metals) will raise its melting point. When the reverse is true, then an increase in pressure causes a corresponding decrease in melting point (e.g. silicon and bismuth) [29][31].

The effect of pressure on the equilibrium diagram of an alloy was studied by Chadwick [147]. He illustrated the effect by making a comparison of two thermographs obtained from Al-5wt%Si alloy cast under atmospheric pressure and under 115 MPa applied pressure, shown in figure 5-4. Subsequently, a new equilibrium diagram was constructed for freezing under a pressure of 115MPa. Chadwick showed that applied pressure during the solidification stage will have the effect of increasing the liquidus, solidus and eutectic temperatures on the equilibrium diagram, shown in figure 5-5. In relationship to magnesium, he reported that an applied pressure of 100 MPa produces a 6.5°C increase in freezing temperature [33].

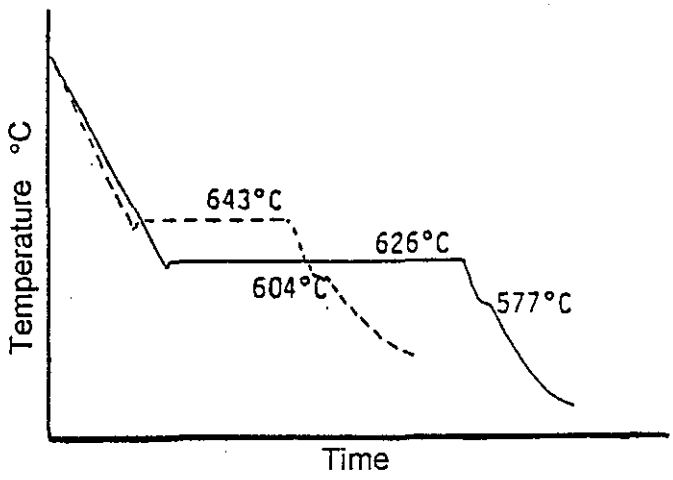


Figure 5-4 Thermographs of Al-5%Si alloy cast under atmospheric pressure (solid lines) and 115 MPa applied pressure (dashed lines) [147].

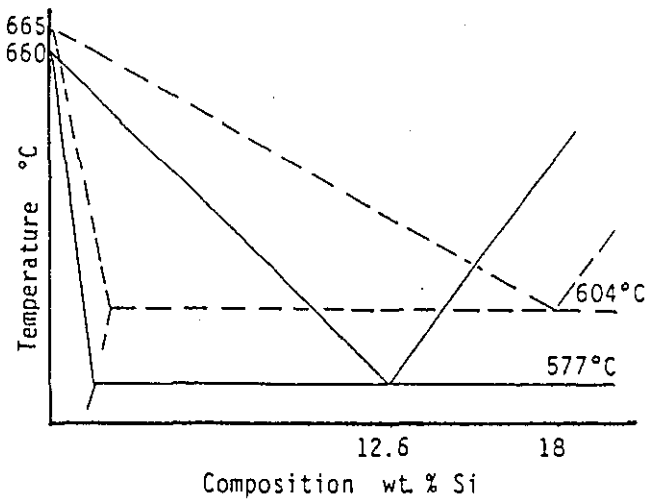


Figure 5-5 Effect of Pressure on Equilibrium Diagrams, atmospheric pressure (solid lines) and 115 MPa (dashed lines) [147].

5.4.2.2 High Heat Transfer Coefficient in Squeeze Casting

The pressurised solidification in squeeze casting ensures a particularly faithful reproduction of the surface of the die set. This reflects the intimate contact between the die and solidifying metal at all stages of solidification. Studies conducted by Nishida and Matsubara [148] on the heat transfer at the metal mould-casting interface found that squeeze castings solidify ten times faster than gravity die castings.

5.4.2.3 Combined Effect of Clausis-Clapeyron and High Heat Transfer Coefficient

The increase in liquidus temperature (due to the Clausis-Clapeyron equation) and the absence of an air gap leads to an increased rate of heat extraction, i.e. shorter solidification time and, subsequently, results in an overall refinement of the structure. An example of the effect of pressure on the microstructure of an aluminium alloy [29] is shown in figure 5-6. Compared to the gravity die cast structure the applied pressure has produced a very fine grained and defect-free structure.

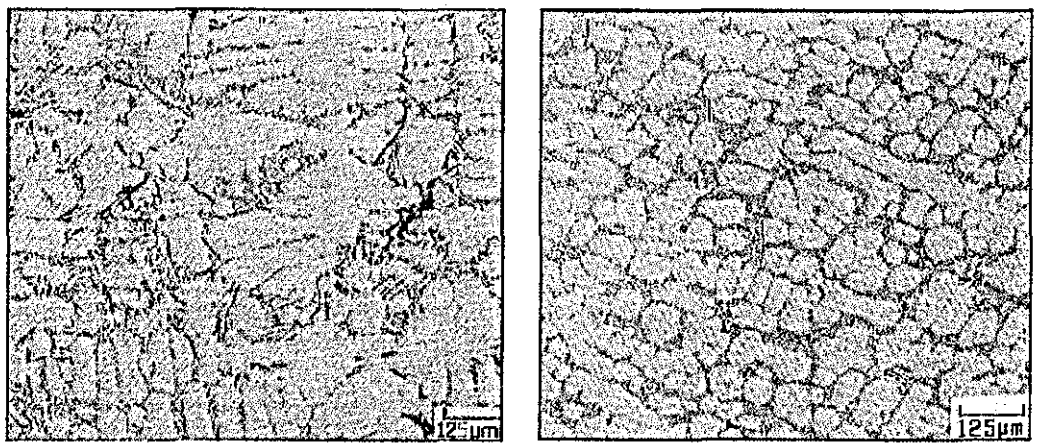


Figure 5-6 Al-7%Si cast (left) under atmospheric pressure, (right) at 150 MPa [29]

5.4.3 The Influence of Casting Integrity on Components following Heat Treatment

The absence of micro-porosity in the casting enables optimum mechanical properties to be attained after heat treatment. Improvement is attributed to the fine grains and smaller DAS in squeeze castings, which is known to improve the solutionising of constituents. This is because the constituent in coarse grains occurs as larger particles which require more time to solutionise. Also it requires greater diffusion distances to attain complete homogeneity within the solid solution phase.

5.4.4 The Advantages of Squeeze Casting

Some of the major advantages of the squeeze casting technique are:-

(i) Microporosity (shrinkage and porosity) is eliminated, due to the effect of solidification under load [29][138].

(ii) The pressurised solidification improves casting accuracy, dimensional consistency and product quality attributes such as surface finish and mechanical properties [149], hence providing the opportunity to reduce inspection costs [138].

(iii) Isotropic properties.

(iv) A near net shape process which results in excellent material yield due to the absence of a runner and feeder system [29][30][138][139].

(v) The applied pressure ensures complete mould filling so metal fluidity is not a prime requirement [30], for examples Long Freezing Range (LFR) and wrought alloys.

(vi) Microstructual control can be achieved by controlling process parameters such as pouring temperature and die temperature [29].

(vii) Faster solidification times than most other casting methods yields a very fine microstructure, resulting in very good tensile properties, significantly exceeding conventional castings and at least comparable to forging [139][144].

5.4.5 The Disadvantages of Squeeze Casting

Despite all it advantages, the process itself has some drawbacks over other casting techniques, they are: high capital investment (requires sophisticated press), high tooling costs for prototypes, difficult to achieve detailed features [138]. Nevertheless, cost factors generally diminish with larger production quantities and improved casting integrity provides a further cost saving through less scrap and rework.

5.5 Principles of Squeeze Infiltration Process for Fabricating MMCs

Given the advantages of squeeze casting, it is not surprising that this process is favoured for MMC fabrication. Many papers have indicated that squeeze casting is the most developed and advantageous process for commercial mass production of MMCs [105][150]. One of the most successful applications is the fibre reinforced engine piston [1], where the process is used to produce the infiltration of molten metal into a fibre preform. Such a fabrication method has the following fundamental advantages [105][150]:

(i) It is relatively simple and economical as compared to other MMC fabrication techniques. Furthermore, it has all the basic advantages which the casting techniques offer over other methods of production.

(ii) The pressure assisted infiltration ensures, that the castings are free from common defects such as porosity and shrinkage cavities.

(iii) The rate of production of samples is much faster than the diffusion bonding or hot pressing process, once the equipment is properly set up.

(iv) Pressure assisted infiltration compensates for the poor wettability between the reinforcements and the liquid metal. Consequently, the rapid solidification of the process prevents further reaction between the reinforcement and the melt, i.e. oxide reinforcements in magnesium alloys.

(v) Ability to reinforce selected regions of parts, e.g. piston and engine block [1].

5.5.1 Kinetics of Infiltrating Reinforcement Preforms

In preform infiltration processes, friction forces due to metal viscosity significantly increase the pressure which must be applied above the capillary pressure opposing flow of the metal into the fibre preform. This pressure increase can be calculated from the Blake-Kozeny equation [151], due to the narrow gaps between fibres within the porous preform, it can reach very high values (above 10 MPa) if high infiltration velocities are desired [152][153][154][155]. It is usually desirable to keep temperatures as low as possible to minimise chemical reactions between the fibres and the matrix. It is possible to infiltrate fibrous preforms held at a temperature below the liquidus of the alloy, which leads to the formation of some solid metal between the fibres during infiltration [153][154]. The formation of solid metal in the midst of the fibre preform, however, increases the pressure necessary to overcome friction forces, since the effective channels to be infiltrated are narrowed. This effect further complicates modelling of the process, since heat flow and solidification must be treated simultaneously.

5.6 Process Parameters for the Squeeze Casting Process

A number of important parameters have been identified in the squeeze casting [29][30] and squeeze infiltration processes [26][153][156]. These are: (i) melt temperature, (ii) melt quality (i.e. absence of oxide films or particles) and quantity, (iii) die temperature, (iv) applied pressure, (v) duration of applied pressure, (vi) infiltration velocity, and (vii) preform temperature. These parameters are discussed in the following sections.

5.6.1 Melt Temperature

Control of pouring temperature is an effective way of obtaining a refined structure in conventional casting processes and will clearly be an important factor in squeeze casting. In general, thin section and non-uniform profiles require a higher casting temperature to promote die filling.

5.6.2 Melt Quality and Quantity

Melt quality is always an important factor in casting processes, as it determines the mechanical properties. Therefore it is important to improve quality by using such means as fluxing, skimming, filtering or bottom pouring. In terms of production, precise monitoring of melt poured into the cavity is important in the production of net-shape products by squeeze casting.

5.6.3 Die Temperature

Low temperature can lead to thermal fatigue failure in the die and cold laps on the surfaces of the casting. Very high temperatures can cause hot spots in the die and shrinkage pores in the casting. Therefore careful selection of die temperature is essential to maintain a longer tool life. In general the die temperature for commercial non-ferrous alloys is maintained between 200°C and 300°C [29][138].

5.6.4 Applied Pressure

The pressure applied must be sufficient to offset shrinkage and gas porosity by forcing molten or semi-molten metal though a network of solid skeletons. The dissolved gases are either contained in the solid solution or dispersed through the micro gap between the ejector and the punch.

Fukunaga and Kuriyama [153], in their experimental studies on the fabrication of aluminium matrix composites by squeeze casting, reported that the applied pressure must exceed 50 MPa to produce composites free from porosity. A similar report was produced by Chadwick [11], who pointed out that 50 MPa is the minimum pressure required to ensure pore free composites. However, Ha [34] reported that a minimum 100 MPa is required to provide pore free castings for magnesium AZ91 composites containing 16% volume alumina fibres. He also indicated that an applied pressure of 50 MPa was sufficient for the AZ91 alloy.

A number of equations exist for prediction of the required infiltration pressure to achieve pore free composites [25][152][157][158][159]. These equations depend on a number of reactions between metal and fibres, highlighted in section 4.5.2. Furthermore the volume fraction of the fibres (capillarity resistance) must be taken into account. However differences in alloy characteristics and component geometry make it difficult to predict actual pressure levels.

5.6.5 Duration of Applied Pressure

Pressure duration depends on material use, casting dimensions and heat transfer conditions. One second per millimetre of casting section thickness is normally quoted as being sufficient to ensure solidification of the casting under pressure [160]. Longer levels of pressure duration may affect solid state transactions.

5.6.6 Infiltration Velocity

It has been reported by Fukunaga and his co-workers [25][153] that infiltration speed does not affect infiltration length, the microstructures or tensile strength. This may be seen in the chart shown in figure 5-7 of infiltration speed against the tensile strength. Infiltration velocity changes between 1 and 5cm/s have little influence on the tensile properties.

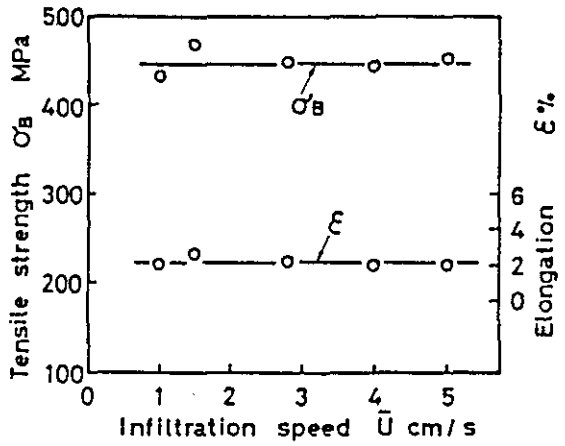


Figure 5-7 Effect of infiltration speed on mechanical properties [153].

5.6.7 Preform Temperature

Preform composition, fibre volume, fibre type, and degree of fibre orientation, previously mentioned in sections 4.4 and 4.7.2, are all essential to good metal matrix parts using the squeeze casting method. Equally important is control of the preheating of the preform in order to avoid freezing of the liquid on the surface of the preform. Such a freezing effect will result in incomplete infiltration of the final cast composite. Therefore heating of the preform is necessary to achieve a sound composite casting.

It has been reported by Toaz et al. [99] that it is usual to preheat the preform to 60% of the casting temperature. During the pressurisation cycle the liquid metal infiltrates the voids in the preform such that the reinforcing fibres become an integral part of the casting. Long et al. [161] argued that the preform temperature should be above the melting temperature of the alloys to achieve optimum mechanical properties.

However, the extensive work carried out by Fukunaga and Kuriyama [26][153] has shown that there exists a suitable temperature range for the fabrication of reliably sound composite materials with a given volume fraction of fibres. The optimum production temperature (which lies in the hatched range) is presented in the chart shown in figure 5-8.

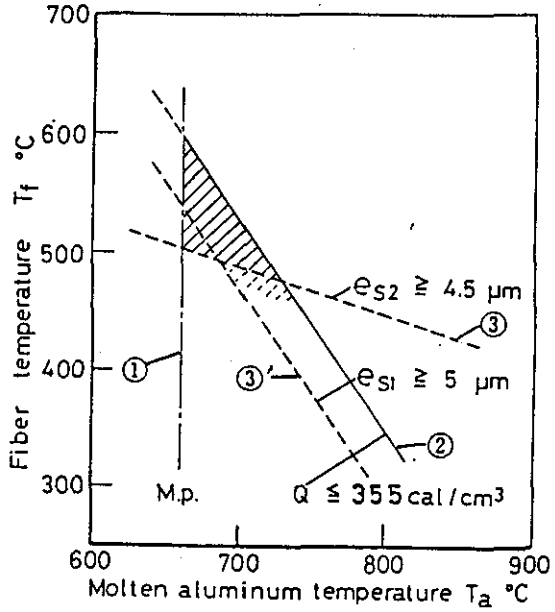


Figure 5-8 Optimum production temperature range [26][153].

Fukunaga and Kuriyama [26][153] explained the existence of an optimum production (preform and pouring) temperature range. They found that a high preform temperature tends to degrade fibres because of an active mutual reaction between the fibres and molten metal (aluminium). Low preform temperatures, on the other hand, may give rise to poor interface bonding strength.

5.7 Past Research Work on Squeeze Casting

Much research work on squeeze casting has been carried out in both industry and academic institutions, but the focus of the work has been concentrated on aluminium alloys. Other alloys have also been studied, but on a smaller scale, such as copper base [162], zinc base [31][32][145] and ferrous alloys [146]. Some of the researchers' findings are briefly reviewed below.

Chadwick and Yue reviewed work on Al-Si alloys LM24, LM25 and A357 [29]. They generally found that squeeze cast materials exhibited superior mechanical properties as compared to conventionally cast materials. They also highlighted that heat treated, squeeze cast LM24 alloy can provide better mechanical properties than the more expensive LM25 and A357, with U.T.S. improved by two-fold and 0.2% proof stress by three-fold. On the % elongation value for squeeze cast Al-4.5%Cu, they reported that it was three times higher than that for the sand cast alloy [29].

In another study, Chadwick [33] evaluated the variation in properties of AZ91 for sand cast, gravity die cast, high pressure die cast and squeeze cast material. He reported that the squeeze cast material showed the highest values of UTS, 0.2% proof stress and elongation to failure. His results also showed

that the heat treatment cycle can further increase the UTS of the squeeze cast material from 200 MPa to 260 MPa.

Williams and Fisher reviewed work on LM5, LM18 and LM25 [142]. When compared with typical chill cast properties, squeeze cast components exhibited higher mechanical properties; especially in the elongation to failure.

Zantout [28] studied the effects of squeeze casting Al-4.5%Cu and Al-3.75%Mg. He reported that all castings produced were porosity-free and fine equiaxed structures were observed. In addition there was a greater than 50% improvement in the U.T.S. and the increase in ductility was found to be more than two-fold.

Yakoub [32] worked on squeeze casting ZA alloys, and reported that porosity was eliminated due to the effect of applied pressure. Begg [31] studied the effect of variables in squeeze casting ZA alloys. It was reported that all alloys were porosity free and the important parameters were pouring temperature, die temperature and section thickness. Like Zantout and Yakoub, Begg reported that a critical pressure is required to produce porosity-free castings.

There has been a significant amount of work done on MMCs using the squeeze infiltration process. It has been reported that the current matrices used for automotive MMCs are largely aluminium alloys, which account for 90% of the overall MMC products [1]. Other materials, such as magnesium have been worked on, but on a smaller scale. Some of the research findings are briefly outlined below.

Hann from A.E. Engine Components Inc. reported that the squeeze cast Al-Si fibre reinforcement engine piston bowls survived up to 1500 hours of a loading test as compared to 200 hours with conventional gravity cast pistons [140].

Chadwick and Stubbington studied the castability of A356+20% vol. SiCp [30]. They found that squeeze casting considerably helped in the infiltration of the fibre/particulate with the molten metal and this significantly improved the strength and stiffness of aluminium alloy matrices.

In another study, Chadwick [33] and Ha [34] experimented with three different fibre types, namely Saffil™, glass/graphite mixture, and stainless steel wire on magnesium AZ91 matrix. In their comparison on the hardness of the composite, the glass/graphite composite which contained only approximately 9% vol. of graphite, had a hardness very similar to that of the 25% vol. Saffil™ reinforced alloy. However, they reported the low temperature characteristic of glass fibres had prevented it from being the most viable option for magnesium-based alloy. They observed the glass

fibres melted during liquid metal infiltration and generated large brittle crystals of Mg_2Si, which significantly reduced overall strength. In their studies on the creep and fatigue strength on 16% vol. Saffil™ reinforced alloy, compared with AZ91 tested under the same conditions, they reported that the composite material had a creep life of an order of magnitude better than the matrix phase and a fatigue strength double that of AZ91 alloy alone.

Other work on magnesium matrix composites was described in section 4.8.

5.8 Summary

In general from the literature review, the potential cost saving is influenced greatly by the production quantity requirements, the complexity and size of the component, and the work material. When only a few dozen components are required, sand casting and machining from solid blanks are preferred. When larger production quantities of the order of a few thousand are required, squeeze casting quickly becomes a promising production technique. Complex geometry and difficult-to-machine materials would favour squeeze casting. When quantities are large and the component geometry is relatively simple, forging becomes more viable and should be preferred to squeeze casting. Of course, a complex geometry such as the car wheel is completely beyond the capability of the forging technique regardless of the required quantities.

Previous research has shown that squeeze-cast mechanical properties are superior in all respects to those obtained by conventional casting. The ultimate tensile strength has been improved by squeeze casting and of particular significance is the marked improvement in the elongation to failure. The improvements were mainly due to the presence of a fine-grained structure and the absence of micro-porosity in the squeeze cast material.

With the increase in production of MMCs, especially in the automotive industry, squeeze casting has gradually established itself as one of the major processes for fabricating MMC components. Therefore based on the literature survey, it is fair to conclude that in most cases the squeeze casting process or squeeze infiltration process is competing well with forging in all mechanical respects, and that the process will have the edge over forging process if isotropic properties are important.

Chapter 6

A Critical Assessment of the Research Direction

6.1 Introduction

The main research issues which underpin this thesis will be identified and discussed in this chapter. The selection of a specific magnesium alloy, fibre type and process conditions to achieve the highest possible component properties with the most economical cost will be discussed. A critical assessment of the selection of the magnesium alloy and reinforcement materials which will constitute the MMC is given. This is followed by a study of the related process parameters for the squeeze infiltration of the magnesium MMC. It will be shown how the selection of the process parameters has been underpinned by a critical assessment of the literature. This will identify the potential areas that could be further explored in order to establish a high strength magnesium alloy.

6.2 The Selection of Matrix Materials for MMCs

The classification of magnesium alloys, discussed in section 3.5, showed that the Mg-Zn family of alloys offers the potential for further research as it is the next most economical in cost after the Mg-Al family alloy. Initially, however, the literature review on magnesium (chapter 3) and initial discussions [163] led to the belief that research on the AZ91 (magnesium-aluminium family alloy) was the most viable, as it was both an economical and widely used magnesium alloy in die casting.

The literature survey revealed a significant amount of work in the area. Work on squeeze casting AZ91 alloy and its composite has been extensively reported by researchers like Kainer et al. [16] [35][36], Lee et al. [164], Kamado et al. [136], Chadwick [33] and Ha [34]. Their research explored the full potential of the AZ91 alloy. Their results did not show dramatic improvements in the mechanical properties when this material was used as the matrix for magnesium MMCs. Investigations carried out by Kamado and his co-workers [136] on the elevated temperature properties of AZ91 MMC showed no improvement (relative to its matrix) despite the presence of reinforcement. Therefore the use of an alternative, such as the magnesium zinc family of alloys, which is of equivalent economical value to the AZ91 offers the potential for further research work.

Although it is known that a zinc addition to magnesium imparts strength to the alloy, Mg-Zn alloys are of little commercial interest in binary form. Mg-Zn alloys have relatively poor casting qualities, are susceptible to microporosity and are not weldable [47]. However, from the metallurgical study of magnesium in section 3.4, it is known that these problems are largely overcome when Rare Earths

(RE) elements are added. An undesirable consequence is that the tensile properties at ambient temperatures are reduced because of some loss of zinc from the solution through the formation of stable Mg-Zn-RE phases in grain boundaries. Nevertheless, the addition of RE elements provides significant improvement at elevated temperatures [58]. Such an increase in tensile properties at elevated temperature is important, since the automotive and other general applications have become more demanding over the years. More importantly, the trend of materials requiring higher mechanical properties, especially at elevated temperatures for future applications, will increase. Hence research on higher performance materials is required in preparation for future engineering applications.

For an elevated temperature range, commercially available alloys like RZ5 will be in demand. At present, the sand casting process (slow solidification) is mainly used for the production of the majority of Mg-Zn-RE family alloy components. The slow solidification process, however, results in large grain sizes and reduced mechanical properties. Zirconium is invariably included in this family of alloys to provide grain refinement, at the expense of cost and increased process complexity.

However, a major assertion is that the only benefit in using this material, is when the cost is competitive to that of the Mg-Al family and this is possible through squeeze casting. The unique feature of the process is that the applied pressure performs the grain refinement role, thus eliminating the requirement of zirconium as the alloying element [165]. As a result, advantages can be gained through reduced melting costs and the elimination of the problems associated with processing zirconium. As a consequence, it may be considered that research on Mg-Zn-RE family alloys holds promise and it is proposed that two matrix materials be considered, these are (i) RZ5DF alloy (Mg-4.2%Zn-1.3%RE) and (ii) RZ5 alloy (Mg-4.2%Zn-1.3%RE-0.7%Zr).

In this research, the extent of the improvement (in %), for RZ5DF and RZ5 will be determined. Full studies on the mechanical properties tensile and microstructures of these materials, as 'matrix only' and 'composites' will be conducted. Details of the two proposed material compositions are discussed in the following chapter.

6.3 The Selection of Reinforcement Materials for MMCs

Based on the literature survey on metal matrix composites (sections 4.72 and 4.9), the most compatible reinforcement materials for magnesium are [16][19][34][35][76][127]:

(i)	Alumina (Saffil™)	~	widely used in UK, Japan, Germany, etc.
(ii)	Boron	~	widely used in America
(iii)	Graphite/ carbon	~	widely used in Germany and Japan

However, from this selection of reinforcement materials, only Saffil™ and carbon fibre is available in short filament form. Although Boron filament is known to be compatible with magnesium, it is not available in the United Kingdom. As a result, the preliminary experiments have been restricted to Saffil™ and carbon filaments due to their availability. From the literature review on the recent development of MMCs (section 4.9), alumina-carbon hybrid composite appears to offer an interesting option. Hence three fibre options were selected for the in depth studies of their properties when cast with magnesium. The three fibre options were (i) alumina, (ii) carbon, and (iii) alumina-carbon fibre composites.

In addition to the choice of filament, the fibre volume and binder elements to be present in the composite must also be determined. The criteria for selecting the appropriate fibre volume fraction and binder system will be discussed in the following sections.

6.3.1 The Selection of Suitable Fibre Volume Fraction

Based on the literature survey and some of the considerations listed below, 14% and 20% volume of fibre were selected for the casting production programme.

In most cases, the presence of a higher % volume of reinforcement fibre is desirable, as this tends to increase the mechanical properties and wear resistance of the component. However consideration must also be given to the disadvantages that will be imposed on the mechanical properties. In this case, the author will only outline factors that influence the selection for higher % volume of fibres reinforcement, as the advantages of having greater volume fraction of fibres are obvious. Hence the reasons for not selecting a higher % volume of fibre reinforcement are:

(i) A higher % volume of fibre reinforcement increases material cost, reduces the ductility and causes tool wear due to the abrasive nature of the reinforcement. Even though the squeeze casting process produces near net shape components, some machining is invariably required in order to achieve the high tolerances needed for assembly.

(ii) A greater % volume of fibre will generally encourage a rise in localised stresses, thus initiating failure at low strains.

(iii) There is a significant difference in thermal expansion coefficient between the matrix and reinforcement. The thermal expansion coefficients for matrix and reinforcements are shown below.

$$\text{Mg matrix} \sim \alpha = 26 \times 10^{-6}\ K^{-1}$$
$$Al_2O_3 \text{ fibre} \sim \alpha = 6 \times 10^{-6}\ K^{-1}$$
$$\text{Carbon fibre} \sim \alpha = 3 \times 10^{-6}\ K^{-1}$$

It may be seen that a greater fibre volume will result in higher internal stresses in the MMC. However, a certain % volume fibre is necessary to obtain an improvement in mechanical properties. A balance must be achieved to allow the composite to retain desirable mechanical properties.

(iv) The introduction of a discontinuous ceramic reinforcement into a metal matrix reduces its thermal conductivity. Again a balance must be made if the thermal conductivity properties are of importance to the component.

(v) There is a limit to the improvement to wear resistance. Wang and Hutchings [166] studied the effect of fibre volume fraction on aluminium alloy (6061) matrix. They found that, in general, maximum wear resistance was achieved with an intermediate fibre volume fraction of about 20%. They argued that an increase in fibre volume fraction greater than this limit would increase the probability of fibre fracture and lead to greater material removal by micro-cracking.

(vi) There is ease of infiltration with minimum volume fraction. Fukunaga [26] reported that it is difficult to achieve infiltration of the molten metal into a preform which is less than 10% of the volume fraction of fibres without deformation. Therefore, there should be a minimum of 10% of the volume fraction of fibres.

It may, therefore, be reasoned that a good balance between required mechanical properties, ease of processing and cost may be achieved within the range of 10% to 20% volume fibre. The preliminary studies evaluated the four combinations of fibres listed below for their compatibility with magnesium:

(i) 14% vol. fraction Saffil™ fibre

(ii) 20% vol. fraction Saffil™ fibre

(iii) 20% vol. fraction Carbon fibre

(iv) 12% Saffil™ + 9% Carbon vol. fraction fibre

In relation to the selection of fibre composition and its contents listed above, little work has been conducted on their compatibility with the Mg-Zn-RE of family alloys. The only studies which have been conducted are those by Kamado and his co-workers [37] on composite systems (i) and (ii) cast with the RZ5 alloy. As their findings showed very little improvement in the as-cast properties of the reinforced Mg-Zn-RE family alloys, no further work has been reported. The majority of work for

composite systems (iii) and (iv) focused on the aluminium and magnesium-aluminium family of alloys. Experiments were conducted on these systems with the Mg-Zn-RE family of alloys because of the potential of carbon fibres to provide low cost and low density magnesium MMCs.

6.3.2 The Selection of Suitable Binder System

With reference to the literature review on binder systems in section 4.7.3, it was reported that the commercial silica binders are not suitable for magnesium. As a result, an evaluation of different binder systems was required to determine the compatibility of each with Mg-Zn-RE[2] alloys. Hence three binders were to be investigated in preliminary studies, they were (i) silica binder, (ii) alumina binder, and (iii) alumino-silicate binder.

Silica binder (i) is used in the majority of preform systems to retain integrity and shape. Therefore silica binder was used as the basis for comparison to binders (ii) and (iii). It was reported in the literature that alumina binder (ii) has less of a reaction problem with magnesium alloys in comparison to (i). Hence an evaluation of its suitability for the Mg-Zn-RE family of alloys was necessary. Studies were also carried out on alumino-silicate binders (iii), a hybrid of (i) and (ii). This combination has the potential to combine the merits of each and provide the best binder mix for the preform. Further studies to identify a compatible binder were necessary to achieve a MMC with much improved properties.

6.3.3 Summary of the Selection of Reinforcement Materials

With the determination of suitable reinforcement materials, fibre volume fraction and binder elements based on the literature survey, an evaluation of the various material combinations was conducted in the preliminary casting programme. In the investigation, a combination of five different preform systems was evaluated in order to determine their compatibility and suitability with Mg-Zn-RE[2] alloys fabricated by the squeeze casting process. The potential preforms studied were:

(i) 20% vol. fraction Saffil fibres with 5% silica binder
(ii) 20% vol. fraction Saffil fibres with 5% alumina binder
(iii) 14% vol. fraction Saffil fibres with 5% silica binder
(iv) 12% Saffil + 9% carbon fibres with 5% alumino-silicate binder
(v) 20% vol. fraction carbon fibres with 5% alumino-silicate binder

[2] **Mg-Zn-RE alloys** refers to both the RZ5DF and RZ5 alloys, both contain the basic element of magnesium, zinc and rare earths. The only different between the two is the presence of grain refinement element (zirconium) in the RZ5 alloy.

Studies on the performance of preform systems (i), (ii) and (iii) on Mg-Zn family alloys were conducted by Kamado et al. [21][37]. Based on their results, these preform systems did not provide much improvement in tensile properties. It is the author's opinion that highly beneficial results can be obtained through a major effort to optimise the squeeze casting parameters to improve as-cast properties. This is discussed in the next section. At the same time it is necessary to compare the potential of these preform systems in relation to one another. As for preform systems (iv) and (v), studies have concentrated on aluminium and magnesium-aluminium family alloys. Therefore a complete study of these preform systems with Mg-Zn-RE family alloys will be beneficial.

6.4 Determination of Process Parameters for the Fabricating of MMCs

Clearly, there has been very limited work on establishing squeeze casting parameters for magnesium alloys and composites, in comparison to aluminium and zinc counterparts. Since the correct process parameters are critical for a sound squeeze cast structure, preliminary work was required to establish the appropriate casting parameters. This was necessary before the influence of grain refinement additions and die and pouring temperatures on cast properties could be determined at the secondary experimental stage.

The main issues which underpin this thesis have been identified and discussed. The following chapters present the research to establish process optimisation in the squeeze casting of magnesium alloys and their composites.

Chapter 7

Experimental Methods and Equipment

7.1 Introduction

The aim of this chapter is to tabulate clearly the selected casting parameters and focus on the design of the experiments. The intention is to provide an overview of the experiments by first introducing all three casting programmes and the parameters used. The sets of experiments have been categorised into initial, primary and secondary programmes. These are inextricably linked, where results from preceding stages determine parameter values for succeeding tests. It should be noted, however, that the detailed description of the results, which have led to the choice of casting parameters, has been relegated to later chapters. The composition of the two magnesium alloys used, and their properties, are outlined. The procedures for the mechanical testing, hardness measurements and other evaluation methods for obtaining comparative results are also described.

7.2 The Casting Equipment

The equipment required for the experiments comprises a hydraulic press, a die set, a melting furnace and melting crucibles. These are listed and briefly described in the following sections.

7.2.1 The Squeeze Casting Press and Die

The castings were produced using a 100 tonne Norton press. The specifications for the individual operating parameters and the range of their values are summarised in table 7-1. The hydraulic actuation system of the press can either be operated manually or automatically and the basic components which the press is constructed of are shown in figure 7-1. These are briefly described below.

During the casting cycle the clamping force exerted causes the upper punch to pressurise the molten metal as it solidifies. After each casting cycle, the solidified casting is ejected from the cavity by the lower punch. The maximum pressures exerted by the upper and lower punches are 100 and 50 tonnes respectively.

A transducer is attached to the upper punch and wired to a digital readout to give the operator an indication of the pressure settings. The movement and approach speed of the punch is controlled by a speed dial and a timer is installed for controlling the duration of applied pressure.

Hydraulic Control Variable	Range	Mode of Operation
Fast approach speed	80-106 (mm/s)	set dial-automatic on/off button-manual
Pressing speed	2 - 6 (mm/s)	set dial; variable level sensors
Main ram return speed	14 - 42 (mm/s)	automatic setting / manual
Main ram pressure	10 - 100 (tons)	set dial
Main ram stroke	25 - 508 (mm)	infinitely variable
Max daylight	710 mm	-
Ejector ram pressure	5 - 50 (tons)	set dial
Holding / dwell time	0 - 5 (min)	set dial
Ejector forward speed	13 mm/s	fix
Ejector return speed	45 mm/s	fix

Table 7-1 Control specifications of the hydraulic Norton press

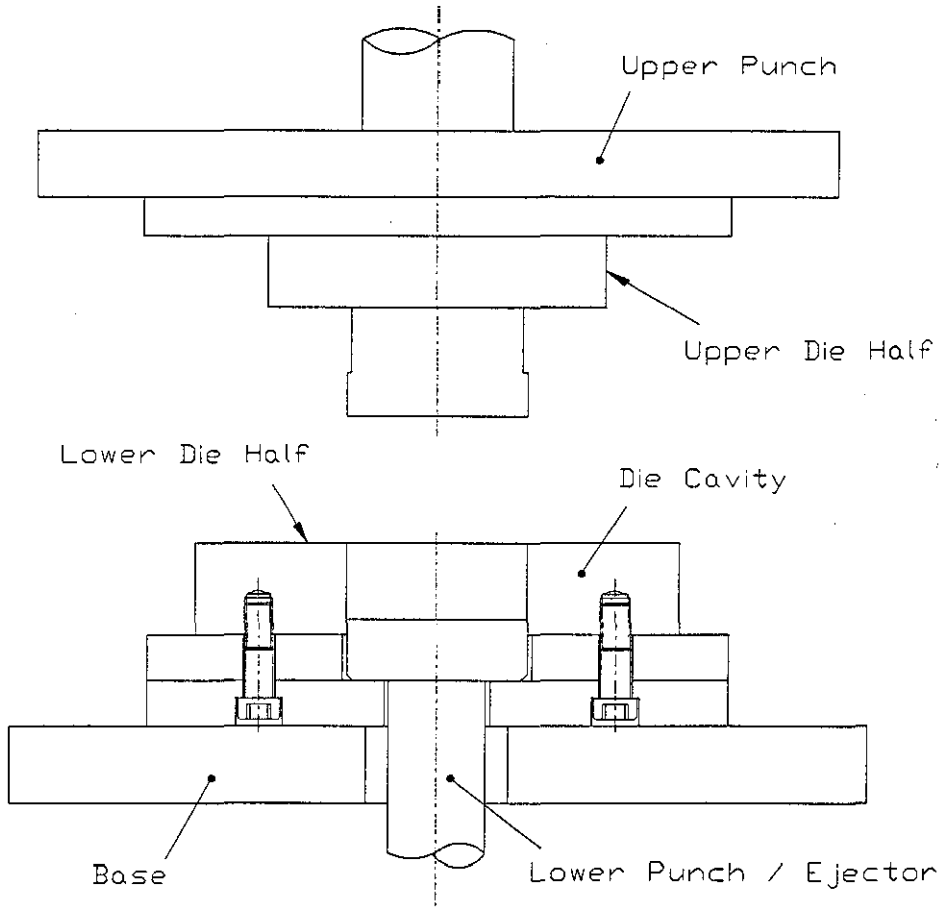


Figure 7-1 A schematic diagram showing the main components and construction of the die set

The die is made from hardened and tempered H-13 die-steel, which provides the wear resistance and strength to withstand the squeeze pressure imposed on the die. The die is made up of two halves; the top half consists of the punch (male component) which is bolted to the upper platen of the press, and the bottom half consists of the cavity (female component) which is bolted down to the lower platen of the press. The die consists of a top and back clamping plate made from mild steel. The ejection system consists of an ejector bar and a bottom ejector plate which is activated by the ejector ram of the press. A detailed assembly drawing of the die is shown in appendix A.

The size of the as cast ingot is 126mm X 75mm by 16mm. The castings produced have to be machined to the required test specimen dimensions. There are six ∅15.8mm X 76mm long heater cartridges installed in the lower die and two ∅15.8mm X 60mm cartridges in the punch.

To protect the mould from excessive wear, the die cavity is coated with boron nitride suspended in water prior to each cast. This lubricant layer serves several purposes, namely [167]: (i) it insulates the die and reduces premature chilling of the metal, (ii) facilitates casting removal and (iii) provides a barrier against direct contact between the die surface and the casting alloy, thus reducing the tendency for erosion of the die.

7.2.2 The Melting Furnace

Melting was carried out using an electrical resistance furnace. The melting temperature for magnesium alloys is between 640°C to 820°C, and this is well within the 1000°C capability of the furnace.

7.2.3 The Melting Crucibles

It is recommended that iron or steel crucibles be used for magnesium-zirconium alloys. Crucibles, for holding the melt, were fabricated by welding steel plates to the ends of a cylindrical shaft. The use of refractory crucibles (including carbon and graphite) is not recommended since they contain zirconium inhibitor elements [60]. It is known that magnesium is not susceptible to iron pick-up at temperatures less than 820°C. Therefore, the chances of iron transfer from the crucible to the melt were remote, since the highest melting temperature used in the experiments did not exceed 800°C.

7.3 Materials Composition and Properties

Two materials were used in this research, the commercially available magnesium RZ5 and RZ5DF (magnesium-zinc-rare earths) alloys. The materials were supplied by Magnesium Elektron Ltd (MEL). RZ5 is a high strength magnesium alloy which exhibits excellent castability, weldability and

pressure tight characteristics. Due to its high strength properties at ambient temperature and up to 150°C, the alloy is considered as a versatile material for a wide range of applications. As such, this material has been widely used by designers involved with aerospace, automotive, military and electronic applications [62][101][168].

The chemical composition and physical properties of RZ5 are discussed in the following section. However the physical properties of RZ5DF alloy are not included, as the information was not available from the magnesium supplier.

7.3.1 The Chemical Composition of the Two Materials Research

The chemical composition of RZ5 alloy:	Zinc	3.5% - 5.0%
	Rare Earths	0.8% - 1.7%
	Zirconium	0.4% - 1.0%
	Magnesium	Balance
The chemical composition of RZ5DF alloy:	Zinc	3.5% - 5.0%
	Rare Earths	0.8% - 1.7%
	Magnesium	Balance

7.3.2 The Physical Properties of RZ5 Alloy

The physical properties of RZ5 alloy are as follows [101]:

Tensile strength	200 MPa
% Proof stress	135 MPa
% Elongation	3 %
Specific gravity	1.84
Coefficient of thermal expansion	27.1 X 10^{-6} K^{-1}
Melting range	530 - 640°C

7.4 The Casting of Magnesium and Preparation of Test Specimens

The production of castings to generate the necessary test specimens requires proper melt and machining practice which must be observed in order to produce quality magnesium components. The following sections highlight the various melting methods for casting magnesium and the necessity of good melting and machining practice. A list of safety precautions is also presented.

7.4.1 The Selection of Melting Methods for Magnesium

As discussed in the literature review, there are two different melting methods, namely the flux and fluxless method. During the initial casting experiments and in the earlier work conducted by the

author [65], the fluxless method was found to produce a better quality. Therefore the fluxless method was used for melting the magnesium.

The fluxless method, also known as the gas covered method, involves the use of an inert gas to achieve a barrier between the molten magnesium and the atmosphere. The gas used was argon; the choice was based upon the fact that there is less chance of contamination of the melt with an inert gas. Thus the charge was protected from excessive oxidation during the melting stage under an argon (inert) gas shield. At the outset, the fluxless method may seem to be a more expensive method than the flux method. From the point of view of production and efficiency, however, it is self-evident that it is better to produce defect free castings. A scrapped casting represents greater losses than solely the die-caster's labour, it represents lost labour charges and melting losses during subsequent remelting.

7.4.2 Melt Practice when Casting Magnesium

In order to achieve consistent casting quality, good melt practice must be observed. The open nature of squeeze casting and the high affinity of magnesium for oxygen requires that significant care be exercised when working with this combination of process and material. Efforts have to be made to reduce the instances of slag and magnesium oxide inclusions during two stages, i.e. prior to casting and during the casting stage.

Casting tools, such as the skimming and puddling tools have to be cleaned (shot blasted) prior to casting to prevent any risk of oxides being carried over to the melt. All tools are preheated to remove moisture and to reduce the possibility of oxides adhering to them.

There are a number of major causes of oxide inclusions during casting, these are oxidation during melting, failure to remove oxide prior to pouring and oxidation during melt transfer. Therefore, a number of measures are used to minimise oxide formation in the magnesium during casting, these are:

(i) The fluxless method is used to create an inert environment during the melting stage. Argon gas was used for this purpose.

(ii) The impurities and oxide are skimmed off the melt surface prior to pouring.

(iii) The die cavities are flooded with argon gas to create an inert environment throughout the casting stage. To further ensure high quality melt for casting, a steel filter was used to filter off lumpy dross and oxides.

(iv) Proper melt handling is practised to minimise melt turbulence. The filter played a part in minimising the melt turbulence generated during pouring by cushioning the molten metal flowing over it.

7.4.3 Observation of Good Machining Practice

To achieve reliable specimen data, good machining practice must be observed. Such practice involves the use of:

(i) higher cutting speed and a lower feed rate to achieve better surface finish. This, in turn, minimises the severity of machining marks that act as crack propagation sites.

(ii) a sharp single point cutting tool must be used to minimise stress concentration on the machined specimen surfaces. For fibre reinforced magnesium composite, carbide cutting tools were used to overcome the abrasive nature of the reinforcement.

(iii) a smaller depth of cut to minimise stress concentration on the machined specimens.

All the measures highlighted above have the tendency to extend the time taken to produce the tensile specimens. However, such practice is essential to attain reliable results.

7.4.4 Safety Precautions in the Handling of Magnesium

As a consequence of the flammability of magnesium, several safety precautions are necessary. They include the storage of magnesium in a dry room, use of protective gear when casting magnesium, frequent removal of swarf for safe storage in steel bins with close fitting lids to prevent any chance of burning if ignited. If molten magnesium or chips burn, the fire can be extinguished by using potassium chloride powder. An important point to note is that water based liquid or proprietary extinguishers should never be used, as they are not suitable for the control of magnesium fires.

7.5 Solidification Characteristic of RZ5 and RZ5DF Alloys

The cooling curves for RZ5 and RZ5DF alloys are presented in figures 7-2 and 7-3 respectively. The former solidified under a cooling rate of 2°C/s, whilst the latter was at a cooling rate of 1.9°C/s. The liquidus (TL) and the solidus temperatures (TS) of the melt can be determined from the discontinuities (plateau temperatures) and the rate of change (gradient) in the cooling curve. In figure 7-2, TL and TS for RZ5 are at temperatures of 639°C and 512°C respectively. From figure 7-3, TL and TS for RZ5DF are at temperatures of 633°C and 474°C respectively.

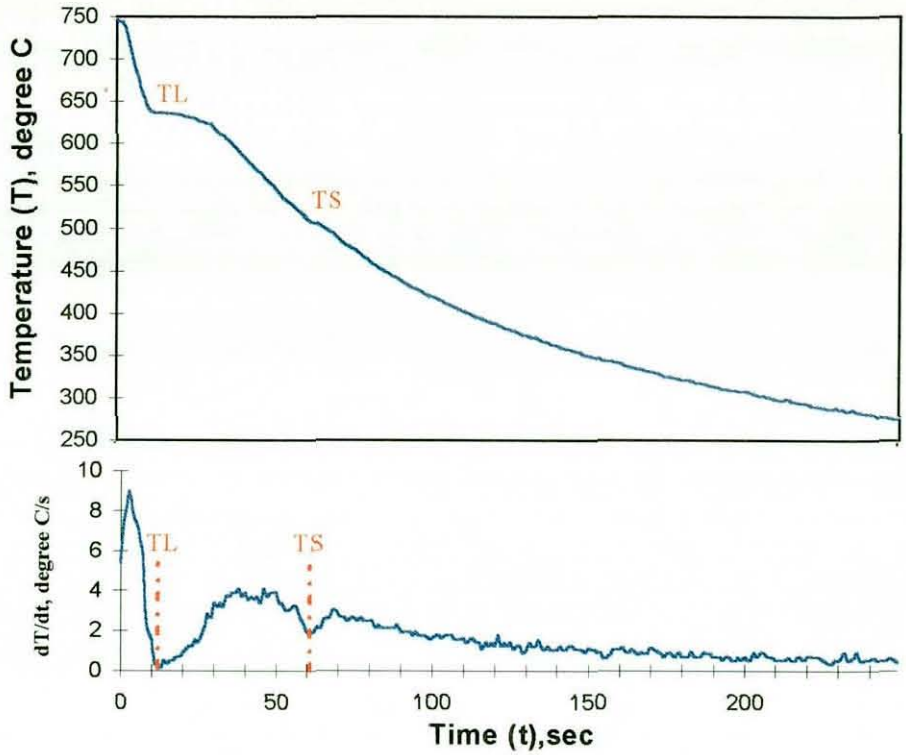


Figure 7-2 The cooling curve for RZ5 alloy solidified at a cooling rate of 2°C/s.

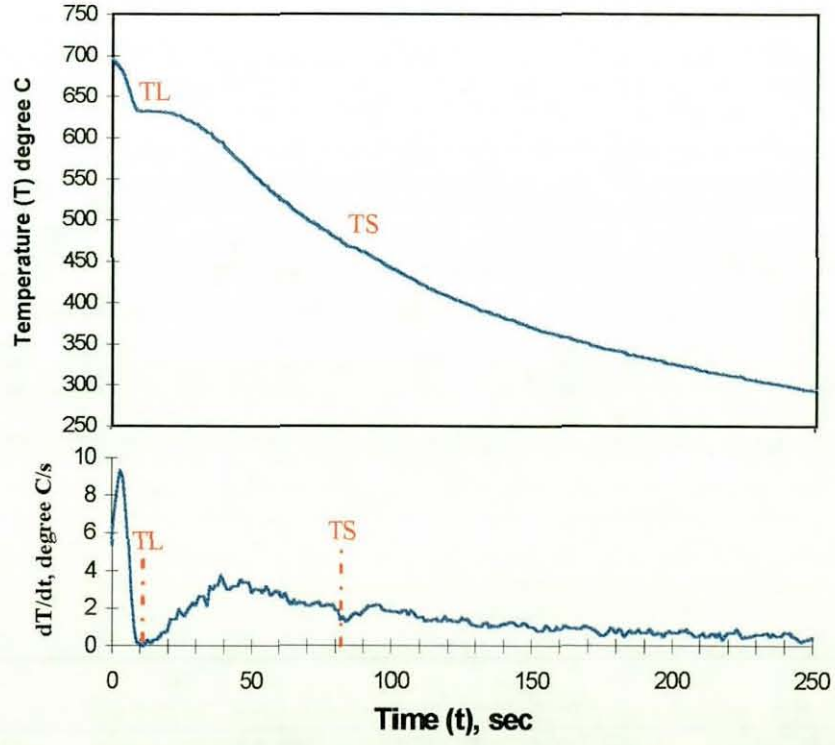


Figure 7-3 The cooling curve for RZ5DF alloy solidified at a cooling rate of 1.9°C/s.

However, the derivation of these values may only be considered as estimates of the (equilibrium) liquidus and solidus temperatures for the alloy. The correct way to obtain the true equilibrium liquidus temperature, TL°, is to extrapolate the TL vs. dT/dt plot to zero cooling curve rate, based on the equation [169]:

$$TL = TL^{\circ} + a\,(dT/dt)^{1/2} \quad \text{------------------------------} \quad (9)$$

It has also been suggested that the true solidus temperature can be determined only by analysing the melting process on heating curves [169]. However such precise measurement of equilibrium temperature is only necessary for the construction of equilibrium or phase diagrams, and for the purpose of this research, there is no necessity of doing so. Furthermore, the danger of magnesium ignition above 400°C make it impractical for such tests to be carried out on the available equipment without modifications to incorporate an inert gas flow control system. Therefore, for this research, only the cooling curve was used to establish the liquidus and solidus temperature of the two alloys studied.

7.6 The Casting Programmes

The mechanical properties of cast components are affected by a number of interacting variables such as the metal pouring temperature, the die temperature, the applied pressure on the solidifying material, the duration of the applied pressure and preform temperature. In order to study the effect of these different parameters, it was necessary to design a casting programme. The programme consisted of initial, primary and secondary programmes. An individual programme consisted of the production of the castings, production of the test specimens from the castings, mechanical testing and microstructural examination of the specimens and the evaluation of the results.

Ha [34] studied the effects of process variables on the mechanical properties of magnesium-base alloys. He reported that the pouring temperature had a significant effect on the mechanical properties. However in his work, the die temperature was maintained at a constant level. Similar studies were conducted by Begg [31], who investigated the effect of process variables on the mechanical properties of zinc-base alloys and found that the die temperature and pouring temperature significantly affected the mechanical properties.

In order to evaluate the influence of pouring and die temperature on the cast properties, the process variables must first be established. Hence the primary casting programme was focused on the

determination of these process variables for RZ5DF alloy, which were subsequently set at a constant level for the investigation at the secondary casting stage. In addition, before the commencement of the evaluation of process variables in the primary casting stage, this initial casting programme was required to establish the feasible range of casting parameters for further investigation at the primary and secondary casting stages. In the initial programme, other common casting variables such as the argon gas flow rate and the average delay before application of pressure were to be determined.

The establishment of the feasible casting range and the selection of process variables based on the literature survey are presented and discussed in the following sections.

7.6.1 The Initial Casting Programme: Establishment of the Feasible Casting Range

The findings from Ha [34] and Begg [31] indicated that the main variables affecting mechanical properties were the pouring and die temperatures. Initial castings were produced to establish this feasible casting range for the analysis of different process parameters during the primary and secondary casting programmes. Establishment of other feasible casting values are also presented in the following sections. It has been necessary to refer briefly to the relevant intermediate results obtained from the different casting programmes. However, it is important to reiterate that, where necessary, the detailed description of these results are provided in later chapters.

7.6.1.1 The Initial Casting Programme: Establishment of a Feasible Pouring Temperature Range

Pure magnesium melts at a temperature of 640°C and its alloys melt at a lower temperature. Typical magnesium alloys used for casting begin to solidify at temperatures around 530°C and are usually completely solid at approximately 400°C. There is an extended freezing range of 130°C, during which the metal is neither solid nor liquid, but is a mixture of the two states. During this period, solidification proceeds continuously and contraction or shrinkage cavities tend to form [55].

The usual casting temperature for magnesium alloys lies within the range of 720°C to 800°C [55]. In conformance with good casting techniques for any metal, the gating and running system should be designed so that the lowest practicable pouring temperature can be used. The specimen design for the experiments was a rectangular block, which did not require conventional runners and gates.

Determination of a feasible pouring range involved establishing the minimum possible pouring temperature before any solidification occurred after passing through the filter. Results from the initial casting programme indicated that a minimum melt temperature of 720°C was necessary to

avoid any significant solidification after the metal passed the filter. Therefore the feasible pouring range for the experimental studies was set at a range between 720°C to 780°C. The temperature for the primary group of experiments was set at a mid-temperature of 750°C. For the secondary casting programme, three pouring temperatures with an interval of 30°C were used, they were 720°C, 750°C, 780°C.

During the process of skimming and transfer of the crucible from the furnace to pouring, there is invariably some temperature drop from the initial reading observed. As such, the normal lift-out temperature recommended by Magnesium Electron Ltd (MEL) is 20°C above that required for pouring [60]. Therefore the lift-out temperature for the primary casting programme was 770°C and the temperatures to be used for the secondary casting programme were 740°C, 770°C and 800°C.

7.6.1.2 The Initial Casting Programme: Establishment of a Feasible Die Temperature Range

In commercial die casting applications, the temperature of the die is maintained between 200°C and 300°C [29][138]. Whilst a lower die temperature can lead to thermal fatigue failure in the die and cold laps on the surfaces of the casting, very high temperatures can cause hot spots and shrinkage pores.

Studies of a feasible die temperature range during the initial casting experiment highlighted the problem of molten RZ5DF alloy adhering to the die wall. This can occur once the die temperature exceeds 275°C. Such an effect generally reduces production efficiency and die life. Therefore the feasible die temperature range for the experimental studies was set at a range between 225°C to 275°C. The temperature for the primary group of experiments was set at a mid-temperature of 250°C. For the secondary casting programme, three die temperatures with an interval of 25°C were chosen, they were 225°C, 250°C, 275°C.

7.6.1.3 The Initial Casting Programme: Establishment of the Protective Gas Flow Rate

To prevent excessive oxidation of magnesium during casting, a protective gas cover is required. A balance must be achieved between the amount of gas required to protect the molten magnesium and the cost. Initial casting experiments indicated that an argon gas flow rate of 5 litres/minute was sufficient to protect the molten magnesium during casting. Therefore an argon flow rate of 5 litres/minute was selected for the primary and secondary casting programme.

7.6.1.4 The Initial Casting Programme: Establishment of the Delay before Application of Pressure

The best results with squeeze casting are achieved by applying pressure immediately after the melt is transferred to the cavity. In practice, however, this is not possible, as some delay will inevitably occur, i.e. the interval between pouring and pressure application. Therefore such delay or interval should be kept to a minimum during casting. The average delay before pressure application observed during the initial casting experiments was 4 seconds.

7.6.2 The Primary Casting Programme

Based on the literature survey and the findings from the initial casting programme, the feasible casting ranges were established for the primary casting programme. To examine the influence of such variables as die and pouring temperatures on the squeeze cast properties, the process variables such as different preform systems, suitable preform temperatures, applied pressures and the duration of applied pressure must first be determined. Comparisons between gravity die cast and squeeze cast magnesium alloys and composites were also made. Other studies conducted were the evaluation of different preform permeabilities, fracture face examinations, and alloy distribution at the fibre-matrix interfaces and grain boundaries. Finally studies on the consistency of casting properties along the longitudinal and transverse directions were conducted.

The RZ5DF alloy was selected as the matrix alloy in the primary studies because the initial aim of this work was to gain a good understanding of the material properties, identify potential problem areas and analyse various factors in the fabrication and use of the material.

To enable the study of the above process variables, both the pouring and die temperature were set at a constant value of 750°C and 250°C respectively. Three tensile specimens were tested to provide the average value quoted in the results tables. Additionally two other specimens were used for microstructure and hardness studies. Therefore a total of five specimens was needed to establish the effect of each individual process parameter. The following sections present details of the investigations carried out in the primary casting programmes.

7.6.2.1 Primary Casting Programme: Process Comparison

A comparison was made between the mechanical properties of specimens produced from the squeeze infiltration, squeeze casting and gravity die casting processes. The comparisons were conducted on both the RZ5DF and RZ5 alloys. Details relating to the allocation of the batches of castings are shown in table 7-2.

Process \ Alloy	Mg-2.5%Zn-RE (as-cast)	RZ5DF (as-cast)	RZ5DF (T5)	RZ5 (as-cast)	RZ5 (T5)
Squeeze Infiltration	N/A	1 - 5	N/A	6 - 10	N/A
Squeeze Casting	11 - 15	16 - 20	21 - 25	26 - 30	31 - 35
Gravity Die Casting	36 - 40	41 - 45	46 - 50	51 - 55	56 - 60

Table 7-2 Specimen numbers for comparative studies between gravity die cast and squeeze cast RZ5 and RZ5DF alloys

7.6.2.2 Primary Casting Programme: Evaluation of Different Preform Systems

The five preform systems listed in section 6.3.3 were selected to investigate their suitability when squeeze cast with RZ5DF alloy. The study aimed to establish the extent of improvement that each preform system provided to the magnesium alloy. The casting orders for the evaluation of different preform systems are listed in table 7-3.

Preform	(i)	(ii)	(iii)	(iv)	(v)
RZ5DF specimens	61 - 65	66 - 70	71 - 75	76 - 80	81 - 85

Table 7-3 The allocation of specimen identification numbers for the evaluation of different preform systems

In the squeeze infiltration process, one of the factors that contributes to the ease of infiltration is the level of preform permeability. Hence, permeability tests were designed to determine how permeable preforms were relative to one another. Details of these studies are presented in the next section.

7.6.2.2.1 Evaluation of Permeability Values in Different Preform Systems

Permeability studies were conducted on five different preform and binder systems to establish a better understanding of each system and the ease with which air could permeate through a network of fibres. Figures 7-4 and 7-5 show the permeability meter and test set-up. Figure 7-6 shows the different preform systems in the shape that was used for the test.

Figure 7-4 Permeability meter and test components. The assembly of test components was fitted on the orifice (top portion) of the meter.

Figure 7-5 Close-up view of permeability test set up. Items from left: (i) preform holder and enclosure sleeve, (ii) preform (cut to shape/sizes), (iii) air restriction seal, and (iv) rubber ring seal

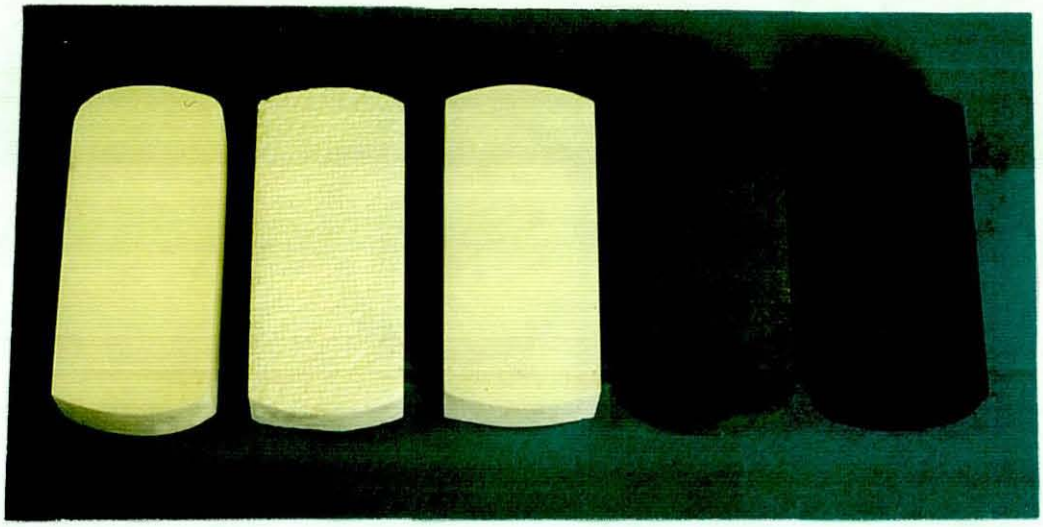


Figure 7-6 Photograph of different preform systems shaped to evaluate their permeability. The preforms shown (from left to right) are:

(i) 20% vol. fraction Saffil fibres with 5% silica binder
(ii) 20% vol. fraction Saffil fibres with 5% alumina binder
(iii) 14% vol. fraction Saffil fibres with 5% silica binder
(iv) 12% Saffil + 9% carbon fibres with 5% alumino-silicate binder
(v) 20% vol. fraction carbon fibres with 5% alumino-silicate binder

7.6.2.3 Primary Casting Programme: Establishment of the Preform Temperature

The literature review, in section 5.6.7, emphasised the importance of preheating the preforms. However, there has been very little research to determine optimum preform temperature for magnesium alloys and the only work reported focused on AZ91 alloy. The wetting capability of these alloys is different, for instance the wetting and the interfacial reaction between Al_2O_3 reinforcement and zirconium or magnesium is far better in comparison to aluminium. As a result, studies to obtain the best preform temperature for attaining optimum properties and ease of infiltration on RZ5DF alloy were required.

A comprehensive evaluation of the effects of preform temperature on composite properties and microstructures was conducted. Preform temperatures in the range of 250°C (similar to the die temperature), 400°C (intermediate temperature), 600°C (at which temperature the RZ5DF alloy is a mixture of liquid and solid), and 750°C (at which temperature the RZ5DF alloy is in the fully molten state) were studied. The allocation of casting batches for the evaluation of preform temperature is listed in table 7-4

Preform Temp. / Applied Pressure	250°C	400°C	600°C	750°C
60 MPa	-	86 - 90	91 - 95	96 - 100
80 MPa	101 - 105	106 - 110	111 - 115	116 - 120
100 MPa	-	121 - 125	126- 130	131 - 135

Table 7-4 The allocation of specimen identification numbers for investigating the correlation between preform temperature and applied pressure on RZ5DF-14% vol. Saffil fibres with silica binder composite

7.6.2.4 Primary Casting Programme: Establishment of the Applied Pressure

It was reported by Chatterjee and Das that raising the pressure level above the minimum consistent with the production of sound castings provides grain refinement and higher mechanical properties [144]. Similar interpretations have been reported by Ha [34], Begg [31], Chadwick and Yue [29].

However, work on applied pressure has mainly focused on aluminium alloys and its composites. For magnesium alloys, the only investigation on the influence of applied pressure was conducted by Ha [34] and Chadwick [33] and their work concentrated on the Mg-Al family of alloys. Mg-Al is a short freezing range (SFR) alloy. The solidification effect will inevitably be different to long freezing range alloys, such as the Mg-Zn family alloys which are the focus of this research. The difference in solidification morphology will be significant when infiltrating the melt into a porous fibre preform. For example, long freezing range alloys tend to remain molten during infiltration and thus provide better infiltration which reduces voids and subsequently improves the soundness of the composite.

An evaluation of the effects of applied pressure, between 0.1 MPa and 120 MPa, was conducted in this investigation. The maximum permissible applied pressure was limited by both the capability of the squeeze casting press and die design. Studies were conducted on the RZ5DF alloy and its composite to determine the feasible applied pressure range required to produce a sound casting. The parameters and processes for this investigation are displayed in table 7-5.

Applied Pressure / Material	0.1MPa	20MPa	40MPa	60MPa	80MPa	100MPa	120MPa
RZ5DF Alloy	136 - 140	141 - 145	146 - 150	151 - 155	156 - 160	161 - 165	166 - 170
RZ5DF alloy + 14% vol. Saffil fibres with silica binder	171 - 175	176 - 180	181 - 185	186 -190	191 - 195	196 - 200	201 - 205

Table 7-5 Specimen numbers for the evaluation of applied pressure

7.6.2.5 Primary Casting Programme: Establishment of the Applied Pressure Duration

Based on the rule of thumb for the applied pressure duration suggested by Rajagopal [160], 16 seconds of applied pressure is required for a casting of 16mm thickness. Nevertheless, an investigation was required to ensure that this duration would provide the optimum cast properties. To achieve this, applied pressure durations ranging from 16 to 35 seconds (for both the alloy and composite) were investigated. The allocation of casting batches for this investigation are listed in table 7-6.

Material \ Pressure Duration	16 sec	25 sec	35 sec
RZ5DF alloy	206 - 210	211- 215	216 - 220
RZ5DF alloy + 14% vol. Saffil fibres with silica binder	221 - 225	226 - 230	231 - 235

Table 7-6 The allocation of specimen identification numbers for the evaluation of applied pressure duration

7.6.2.6 Primary Casting Programme: Evaluation of the Consistency of Properties throughout the Casting

To evaluate the process capability of squeeze casting, a series of tests was conducted to determine the consistency of properties throughout the casting. The investigation involved the evaluation of tensile, hardness and microstructure properties. The allocation of casting batches for this investigation are listed in table 7-7.

Alloy \ Process	Squeeze Cast	Squeeze Infiltrated 14% vol. Saffil fibre with silica binder	Squeeze Infiltrated 20% vol. Saffil fibre with silica binder
RZ5DF alloy	236 - 241	242 - 247	248 - 253

Table 7-7 Specimen numbers for the evaluation of process consistency

7.6.3 The Secondary Casting Programme

The secondary casting programme evaluated the influence of pouring and die temperature and the grain refining element (zirconium) on the squeeze cast magnesium alloys and its composites. Having attained the optimum cast properties for each set of casting alloys and composite systems from the main experiments, the potential for further improvements over these cast properties by heat

treatment was investigated. The following sections present details of the investigations of the secondary casting programme.

7.6.3.1 Secondary Casting Programme: Evaluation of the Effect of Pouring and Die Temperature on Cast Properties

Based on the results obtained from the initial and primary casting programme, the following process variables were adopted for the production of squeeze casting magnesium alloys and composites in the secondary casting programme.

Applied Pressure (for alloy and MMC)	Primary casting programme to establish optimum parameters
Duration of Applied pressure	Primary casting programme to establish optimum parameters
Delay before pressure application	4 sec
Pouring Temperature	720°C, 750°C and 780°C
Die Temperature	225°C, 250°C and 275°C
Test Temperature	Ambient and Elevated temperature (250°C)
Material (Matrix)	RZ5DF and RZ5 alloys
Composite	With and without fibre reinforcement
Composite in % volume and binder	Primary casting programme to establish suitable reinforcement
Protective gas flow rate (Argon)	5 litres/minute

Based on the above process variables, two sets of casting orders were established, comprising the groupings of RZ5DF and RZ5 castings, as shown in tables 7-8 and 7-9. From these castings a total of two hundred and sixteen specimens were produced to analyse the effects of the various parameters. Again, three tensile specimens were tested to provide the average value quoted in the results tables.

Pouring Temp. / Die Temp.	RZ5DF Alloy			RZ5DF MMC		
	720°C	750°C	780°C	720°C	750°C	780°C
225°C	254 - 256 (257-259)	272 - 274 (275-277)	290 - 292 (293-295)	308 - 310 (311-313)	326 - 328 (329-331)	344-346 (347-349)
250°C	260 - 262 (263-265)	278 - 280 (281-283)	296 - 298 (299-301)	314 - 316 (317-319)	332-334 (335-337)	350-352 (353-355)
275°C	266 - 268 (269-271)	284 - 286 (287-289)	302 - 304 (305-307)	320 - 322 (323-325)	338-340 (341-343)	356-358 (359-361)

Table 7-8 Secondary Experimental Programme for RZ5DF Castings
( ) indicate elevated temperature test specimens

Pouring Temp. / Die Temp.	RZ5 Alloy			RZ5 MMC		
	720°C	750°C	780°C	720°C	750°C	780°C
225°C	362-364 (365-367)	380-382 (383-385)	398-400 (401-403)	416-418 (419-421)	434-436 (437-439)	452-454 (455-457)
250°C	368-370 (371-373)	386-388 (389-391)	404-406 (407-409)	422-424 (425-427)	440-442 (443-445)	458-460 (461-463)
275°C	374-376 (377-379)	392-394 (395-397)	410-412 (413-415)	428-430 (431-433)	446-448 (449-451)	464-466 (467-469)

Table 7-9 Secondary Experimental Programme for RZ5 Castings
( ) indicate elevated temperature test specimens

7.6.3.2 Secondary Casting Programme: Establishment of the Optimum Heat Treatment Parameters for Alloys and Composites

Having attained the optimum cast properties from the secondary experimental programme, further improvements in the properties can be achieved with heat treatment. Based on the recommendations of Magnesium Elektron Limited (MEL) [101], and the justification presented in section 3.8.1, the heat treated T5 condition was selected to achieve optimum properties for the RZ5 alloy. However, a suitable heat treatment condition for the RZ5DF alloy was needed as no data is available for the designed alloy used in this research.

As for the composites, it was proposed (based on the literature review in section 4.8) that different heat treatment conditions should be determined for magnesium matrix composites in order to attain optimum mechanical properties from the heat treatment process. Hence a series of peak age hardness and tensile curves was required to establish the optimum heat treatment condition for the designed (RZ5DF) alloy and composites. To establish peak hardness curves, an average of five hardness measurement were taken from each specimen to ensure better statistical significance. The allocation of casting batches is listed in table 7-10.

Material / Properties	RZ5DF alloy	RZ5 + 14% vol. Saffil fibre	RZ5 alloy	RZ5DF + 14% vol. Saffil fibre
Peak Hardness	470 - 483	493 - 512	N/A	528 - 547
Peak Tensile	484 - 486	513 - 521	N/A	548 - 556
Confirmation of Tensile	487 - 489 (490 - 492)	N/A	522 - 524 (525 - 527)	N/A

Table 7-10 Heat treatment evaluation programme
( ) indicate elevated temperature test specimens

7.7 Mechanical Testing Evaluation

The castings obtained using various process variables are compared by individual mechanical properties using tensile test results, namely the Ultimate Tensile Strength (UTS), % elongation and % area reduction.

7.7.1 Tensile Testing of the Specimens

The tensile tests were conducted on a 50 kN Mayes testing machine using position control, running at 0.1mm/min to obtain the modulus. The rate was subsequently increased to 1mm/min for the measurement of ultimate tensile strength. The modulus of the specimens was determined using a strain gauge attached on the gauge section parallel to the direction of tensile loading. Test specimens were machined according to the British Standard, BS18 (1987) and Magnesium Electron Ltd, RB4 specification. Figure 7-7 shows the dimensions of the test specimen machined from the casting.

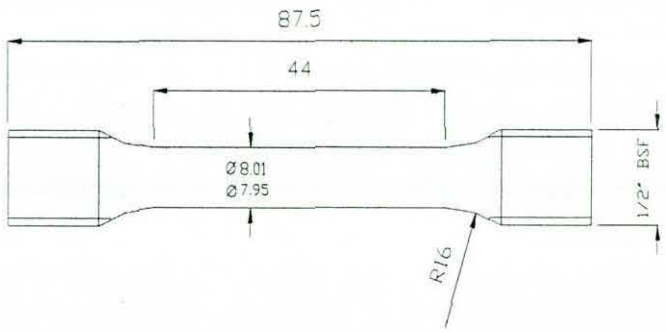


Figure 7-7 Tensile test specimen (All dimensions in mm)

Evaluation of the suitability of the Hounsfield 16 tensile test specimen as the replacement for the RB4 (British and MEL standard) was conducted during the preliminary casting programme. This action had two aims in mind, namely:

(i) it is not feasible to machine screw threads on MMC tensile test specimens, especially on a large scale. When threading was conducted during the casting trial, significant tool wear was experienced, together with frequently broken threads on the fibre reinforced specimens, as shown in figure 7-8. That is why an alternative solution was needed.

(ii) selection of the Hounsfield 16 standard or its equivalent in comparison to the British or MEL recommended test specimen (RB4), doubled the rate of production and the number of specimens that could be extracted from a single casting. Figure 7-9 and 7-12 shows the location of tensile specimens in the casting.

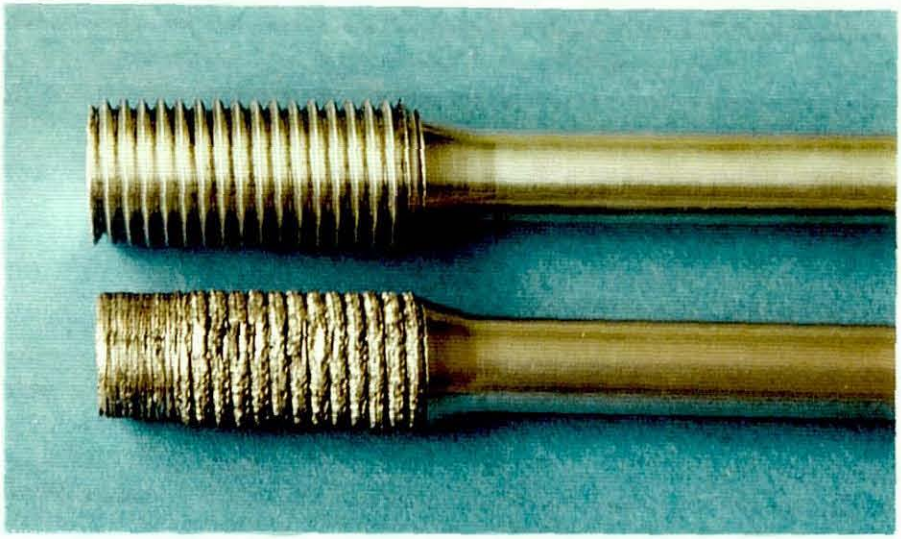


Figure 7-8 Photograph showing the threaded end of RB4 tensile specimens: The alloy specimen (top) and the fibre reinforced specimen (bottom).

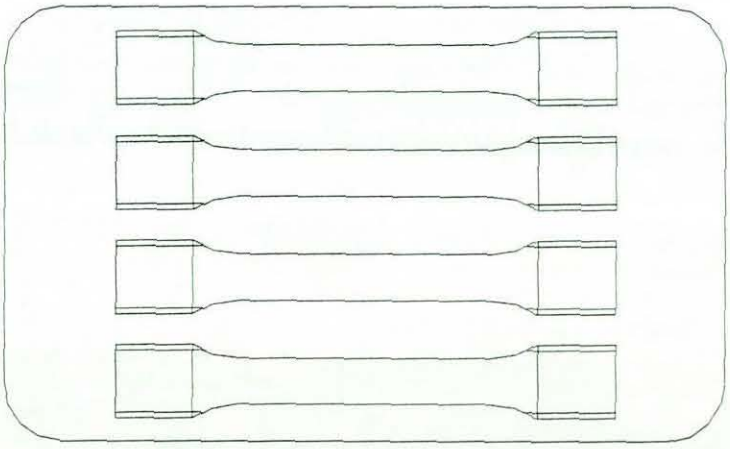


Figure 7-9 The approximate location of the British and MEL recommendation test specimens (RB4) in the casting.

The initial Hounsfield 16 specimens consistently fractured at the radius end of the composite tensile test specimens, as shown in figure 7-10. This was caused by two factors, namely:

(i) *Poor process control*: poor control of machining parameters, i.e. too heavy depth of cut and improper use of cutting tools.

(ii) *Poor specimen design*: unsuitable design of tensile specimen, i.e. too small a radius which subsequently induces stress at that region.

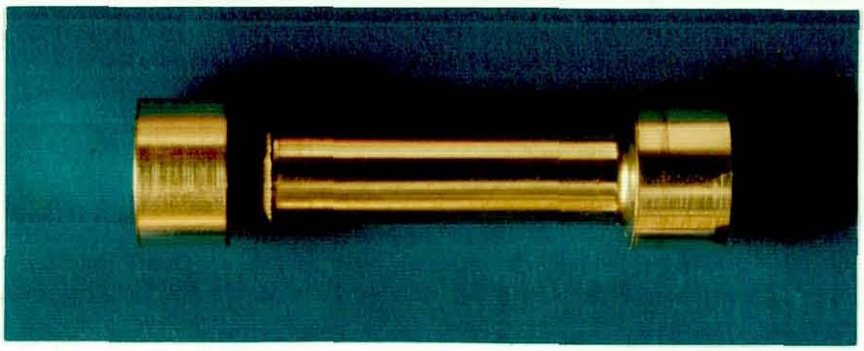


Figure 7-10 Fracture at the radius neck (outside the preferred gauge length)

In order to eliminate the problems of specimen threading and fracture outside the gauge length, a modified Hounsfield 15 tensile test specimen was designed to enable proper tensile testing for the magnesium matrix composite. Figure 7-11 shows the dimensions of the modified test specimen machined from a section of the casting.

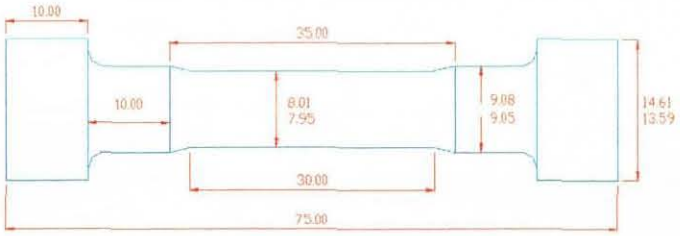


Figure 7-11 Modified Hounsfield 15 tensile test specimen caters for Magnesium matrix composite (All dimension in mm).

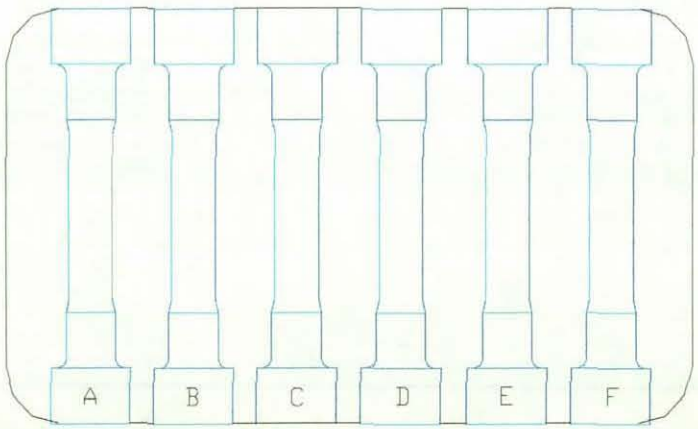


Figure 7-12 The approximate location of the modified Hounsfield 15 tensile test specimen in the casting.

7.8 Hardness Measurement of the Specimens

Hardness was measured to determine and study the influence of reinforcement on the magnesium and the isotropy of fibre distribution. The locations of hardness measurements are shown in figure 7-13. Hardness measurements were conducted on the Rockwell B scale for both the alloys and composites. The preference of Rockwell to Vickers hardness measurement was due to the larger indentation needed to ensure a more consistent measurement on the composite. This is because the area of Vickers hardness indentation is much smaller, in some cases the measurement could be taken from the hard fibre, and this would inevitably cause large variations in the hardness values.

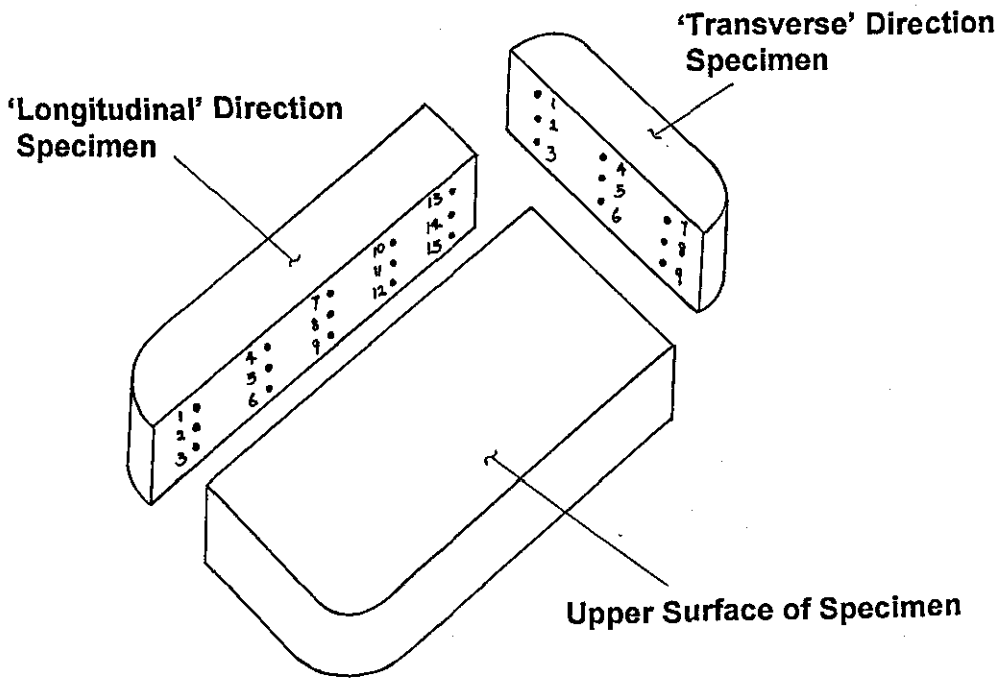


Figure 7-13 Locations of hardness measurements (each dot represents the position of a hardness measurement) taken in both 'Longitudinal' and 'Transverse' directions

7.9 Metallography

Metallographic examinations were carried out to assess the effect of process parameters and fibre additions on the grain size. The procedure for preparing the specimens and microscopic examination are described in the following sections.

7.9.1 The Extraction of Specimens

In the primary casting programme, specimens were sectioned from the casting using a band saw, in both longitudinal and transverse directions (see figure 7-13). This was necessary to obtain a better understanding of the material solidification characteristics, the effect of fibre additions on the

microstructure, and the reaction and infiltration characteristics of magnesium passing through the preform during casting. In the secondary casting programme only a small part of the specimen was cut from each of the cast specimens using a band saw or engineers' lathe.

7.9.2 The Mounting of Specimens

In the secondary casting programme, three specimens of ∅14mm were mounted into a plastic disc. The operation was performed using the Metaserv mounting press. The specimen was placed in the heated mould, resting on the circular support with the relevant surfaces faced down. Before mounting, sufficient epoxy resin (mounting material) was poured into the mould to cover the specimen.

7.9.3 Specimen Preparation

The sectioned and mounted specimens were ground on a stationary abrasive paper on the Buehler Hand Grind. The grinding process was carried out progressively using 240, 320, 400, 600 and 1000 grit silicon carbide papers. The polishing of the specimen was conducted on a rotating disc machine. Graded diamond paste of 6 μm was used as the abrasive and subsequently followed by the 1μm grade.

7.9.4 Specimen Preparation (Etching) to Reveal Grain Structure

The purpose of the etching process is to reveal some of the microstructure characteristics such as grain boundaries, dendrites and microsegregation which cannot be seen by the naked eye. In order to prevent any contamination, the specimen surface must be cleaned by methanol.

Two different alloys were used in the experiments. The cast structure of the magnesium composite is highly complex and different etchants are required to reveal the various features in the different materials. Therefore, three separate etchant compositions were required to reveal the grain boundaries. The three etchants used in this research work were the Nital, Aceticglycol and Acetic picral. The breakdown of the composition and the etching procedure is shown in table 7-7.

No	Etchant Composition	Etching Procedure	Characteristic and Uses
1.	**Nital** 5ml HNO_3 (5%) 95ml methanol (95%)	Immerse specimen for 15 seconds. Wash with methanol and dry.	Reveals grain structure for RZ5 alloy.

2.	**Aceticglycol** 1ml HNO_3 20ml aceticacid 24ml H_2O 60ml ethylene glycol	Immerse specimen face up with gentle agitation for 1-2 seconds. Wash with water, followed by methanol and dry.	Reveals grain structure for RZ5DF alloy.
3.	**Acetic picral** 5ml Acetic acid 5ml 5% picral 105ml H_2O	Immerse specimen in Acetic picral etchant for 2 sec. Followed by a quick 2 sec. dip in to remove staining.	Reveals grain boundaries in magnesium matrix composite.

Table 7-7 Etchants for micro-examination of magnesium alloys and its composites. More specific details regarding the selection of etchant for magnesium casting alloys can be found in Emley [57].

7.9.5 Microscopic and Fracture Surface Examination

An optical microscope was used to study the influence of various process parameters on the cast structure. Magnifications between 10 to 400 times were used in the microscopic examinations. The fracture surfaces from the tensile test specimens of both alloys and MMCs were characterised using a stereoscan 360 electron microscope (SEM). This was equipped with a back scatter detector and was used to generate atomic number contact images of the RZ5DF and RZ5 alloys' microstructures.

This chapter introduced the equipment, production methods and allocation of test specimens for initial, primary and secondary casting programmes. The following chapters, 8 and 9, provide a detailed account of the results and chapter 10 provides a discussion of the primary and secondary programmes.

Chapter 8

Results and Observations from the Primary Casting Programme

8.1 Introduction

This chapter describes the results obtained from the investigations and observations made during the primary casting programme. A comparison between gravity die cast and squeeze cast magnesium alloys and composites is reported. Investigations were carried out to examine different preform systems, preform temperatures, applied pressures and the duration of applied pressure. The optimum conditions for these process variables were determined and the results are reported. This includes the evaluation of preform permeabilities, examination of tensile fracture faces and analysis of alloy distribution at the fibre-matrix interfaces and grain boundaries. Finally, the consistency of casting properties studied in this research is reported.

8.2 A Comparison of the Casting Processes

Comparisons were made between the mechanical properties and metallography of specimens produced using the squeeze infiltration, squeeze casting and gravity die casting processes. A set of process parameters with fixed pouring and die temperature (listed in section 7.6.2.1) was used as a basis for the investigation of the three different casting processes.

In this section, the results obtained from process comparisons between squeeze infiltration, squeeze casting and gravity die casting on the RZ5DF alloy are presented. This includes the examination of the influence of process choice on material strength and also the determination of the influence of zinc and zirconium additions on the cast magnesium properties. Figure 8-1 illustrates the results of the experiments conducted on the Mg-4.2%Zn-RE (RZ5DF) and RZ5 alloys, these results are compared with the Mg-2.5%Zn-RE alloy published previously [165]. The intention was to establish a high strength alloying system for the squeeze casting and squeeze infiltration processes. Based on the results presented in figure 8-1, the UTS for squeeze cast components show significant improvement with a higher zinc content. The results also indicate that components, in particular those with higher zinc content and produced by gravity die casting, have a large variation in UTS.

The comparison of the tensile properties for the RZ5DF alloy castings produced by squeeze infiltration, squeeze casting and gravity die casting, are displayed in figure 8-2. This highlights the significant improvement in strength that fibre reinforcement brings to the alloy. The average UTS of the MMC material is 102% higher than that for the gravity die cast and 31% higher than that for the

squeeze cast material. Detailed tensile properties of specimens produced from the squeeze infiltration, squeeze casting and gravity die casting processes, are displayed in table 1 of appendix B.

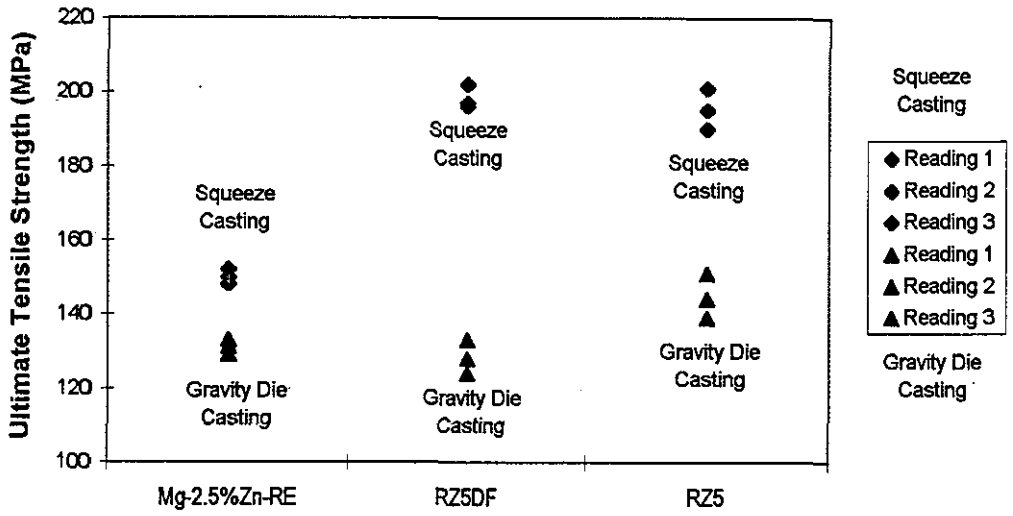


Figure 8-1 The plot of as-cast UTS for squeeze cast (diamonds) and gravity die cast specimens (triangles) cast with constant process parameters (other than applied pressure).

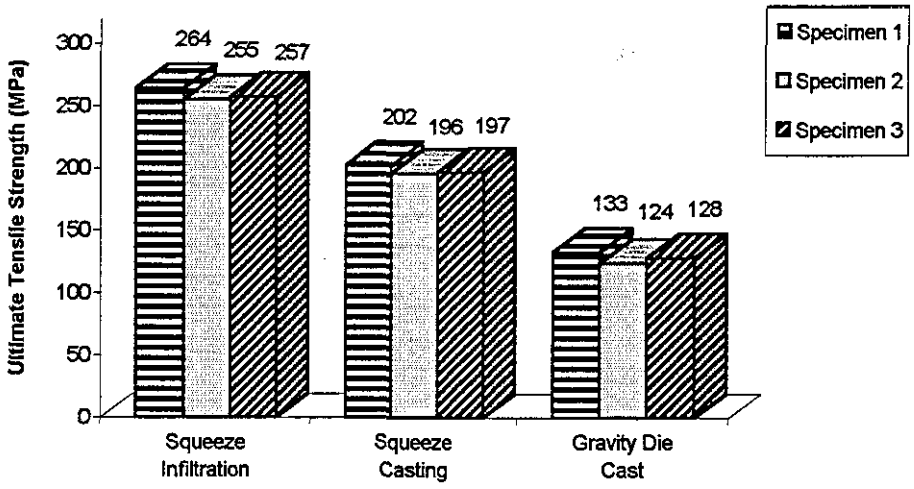


Figure 8-2 The plot of as-cast UTS for gravity die cast, squeeze cast and squeeze infiltrated RZ5DF matrix material, cast under a set of constant process parameters (other than applied pressure).

The hardness results for material produced using the three casting processes are shown in figures 8-3 and 8-4, which compare the hardness along the 'longitudinal' and 'transverse' axis of the cast ingots. The results indicate that the hardness value significantly increases with the presence of 14% volume of Saffil fibres in the alloy. It also showed that the squeeze casting and infiltration processes provide a greater consistency for hardness throughout the alloy cast structure, in comparison to those produced

by the gravity die casting process. Locations of hardness measurements taken in the cast ingots are illustrated in figure 7-13. The results also showed that the hardness values for both squeeze infiltrated (MMC) and squeeze cast (alloy) specimens are more consistent than the gravity die cast specimens. However, such a wide dispersion of hardness values is seen to occur only in the longitudinal direction of the cast specimen. The reasons for this dispersion in hardness values will be explained in section 10.2.

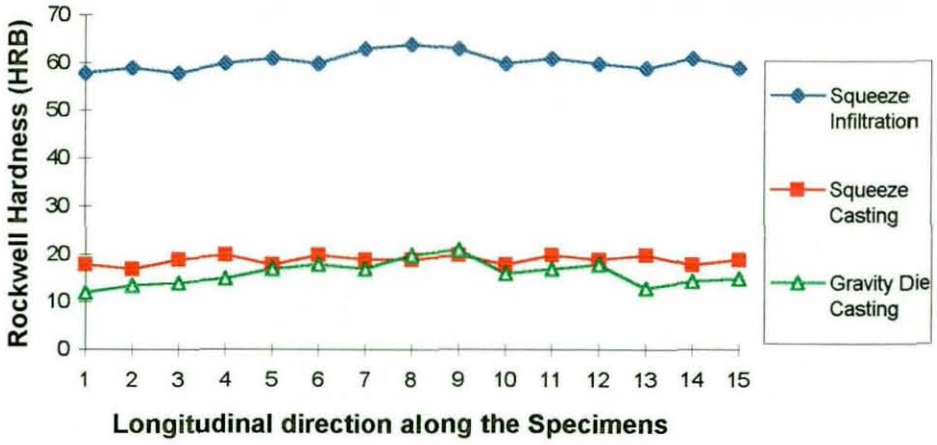


Figure 8-3 The plot of material hardness for gravity die cast, squeeze cast and squeeze infiltrated RZ5DF matrix material, cast under a set of constant process parameters (other than applied pressure).

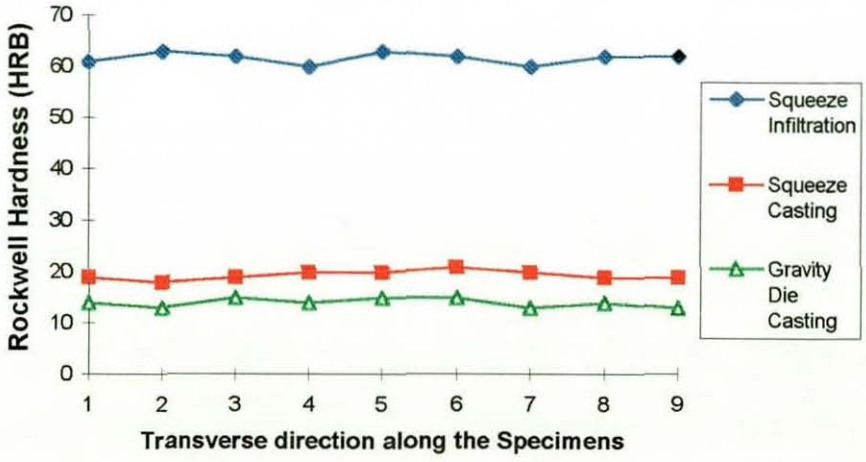
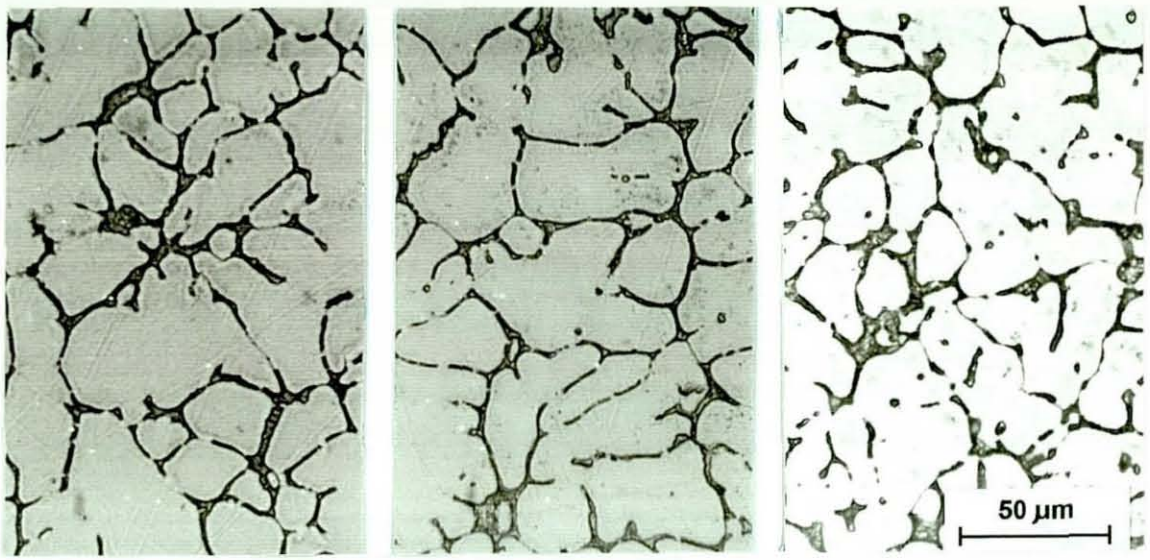


Figure 8-4 The plot of material hardness along the 'transverse' direction on the casting samples, cast under a set of constant process parameters (other than applied pressures)

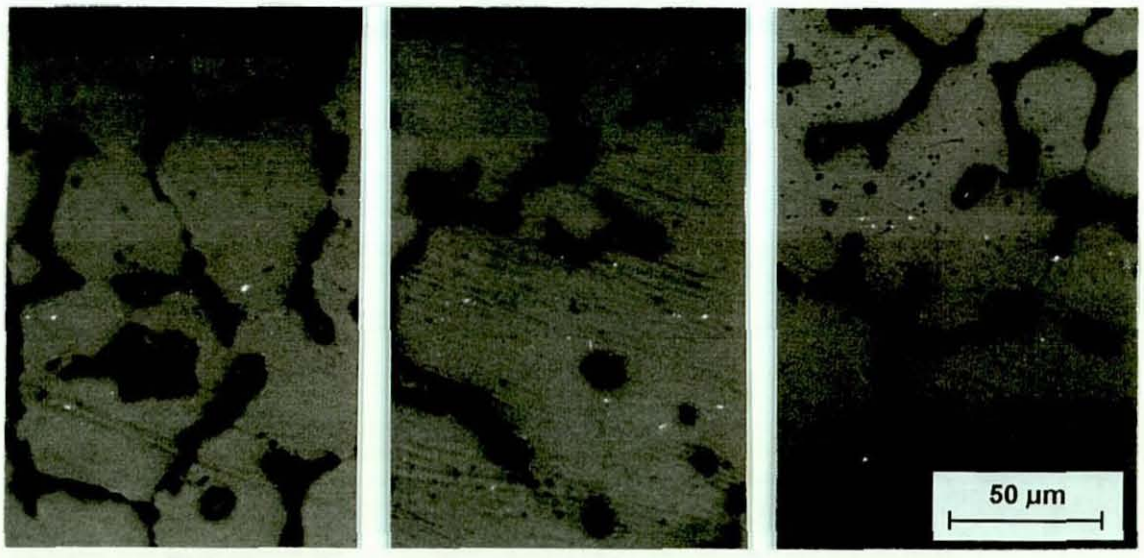
To evaluate the influence of these casting techniques on solidification, microstructural studies were conducted at the edge, quarter and half section of the casting. These studies were intended to develop an understanding, in particular, of the influences on solidification of the die design used in the research. The solidification structures of squeeze cast and gravity die cast material are illustrated in figures 8-5 and 8-6 respectively. The micrographs in figure 8-5 show that squeeze casting produced a

fine and consistent grain structure at the (i) edge, (ii) quarter and (iii) centre of the RZ5DF casting, similar to the observations reported in section 5.4.2.3. Conversely, the slower solidification time associated with the gravity die casting produced variable grain sizes and a coarse structure as seen in figure 8-6.



(i) (ii) (iii)

Figure 8-5 Micrograph of a squeeze casting structure at the (i) edge, (ii) quarter and (iii) centre (RZ5DF alloy).



(i) (ii) (iii)

Figure 8-6 Micrograph of a gravity die casting structure at the (i) edge, (ii) quarter and (iii) centre (RZ5DF alloy).

The surface quality of components produced by squeeze infiltration (MMC), squeeze casting (alloy) and gravity die casting can be seen in figure 8-7. From the photograph, it can be seen that squeeze infiltrated component (i) and squeeze cast component (ii) exhibit a better surface finish compared to those produced by gravity die casting (iii).

(i) (ii) (iii)

Figure 8-7 The surface quality of components produced by (i) squeeze infiltration, (ii) squeeze casting and (iii) gravity die casting.

8.3 The Evaluation of Different Preform Systems

Based on the process settings listed in section 7.6.2.2, several observations were made on the quality and ease of infiltration of the preforms. These observations and results are presented in the following sections and include a comparison of tensile strength, material hardness and metallography.

8.3.1 Examination of the Quality of Various Preform Systems

The quality of the preforms supplied by the manufacturer was examined by SEM to ensure a sound preform (no fibre fracture) prior to squeeze infiltration with the RZ5DF alloy. Such pre-examination is required as fractured fibres are detrimental to the overall MMC mechanical properties. Figures 8-8 to 8-12 show the SEM pictures taken of five different preform systems.

Figure 8-8 SEM micrograph showing the quality of 20% vol. fraction Saffil fibres with 5% silica binder.

Figure 8-9 SEM micrograph showing the quality of 20% vol. fraction Saffil fibres with 5% alumina binder.

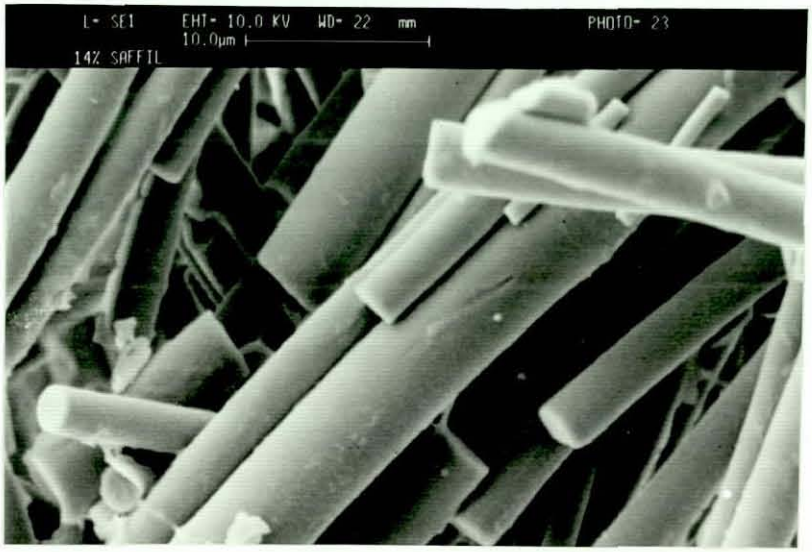


Figure 8-10 SEM micrograph showing the quality of 14% vol. fraction Saffil fibres with 5% silica binder.

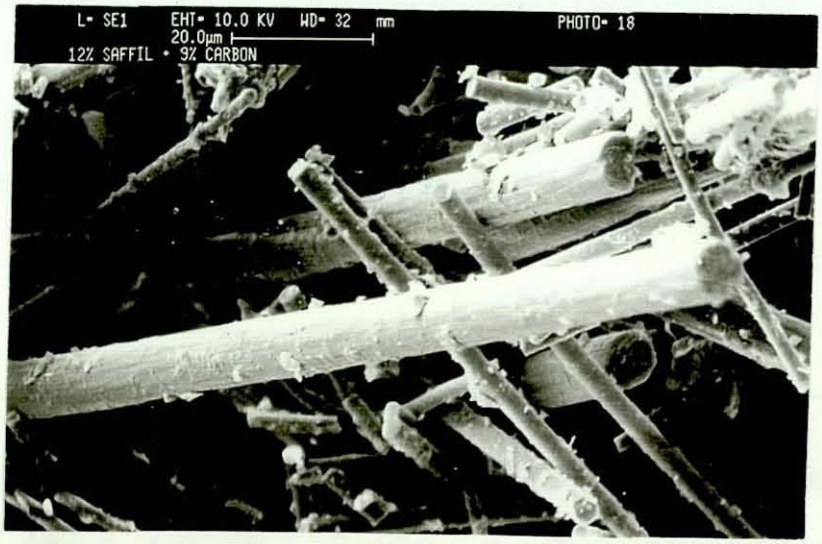


Figure 8-11 SEM micrograph showing the quality of 12% Saffil + 9% carbon vol. fraction fibres with 5% alumino-silicate binder.

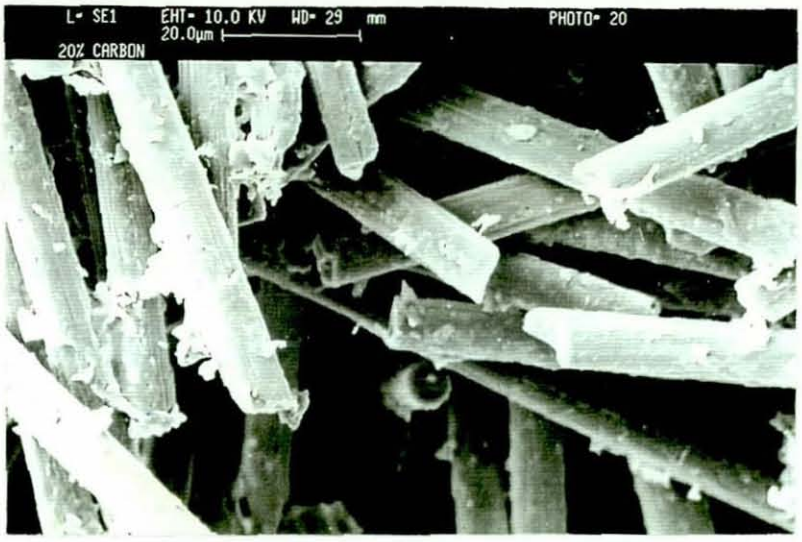


Figure 8-12 SEM micrograph showing the quality of 20% vol. fraction carbon fibres with 5% alumino-silicate binder.

8.3.2 The Effects of Squeeze Infiltration on Different Preform Systems

The effects of squeeze infiltration on different preform systems are described in this section. The cast surface and sectional view of 20% vol. fraction Saffil fibres with 5% silica binder are shown in figures 8-13 and 8-18 respectively. The letters A to F indicate the portion where the specimen was sectioned from the cast ingot shown in figure 7-12. Figure 8-14 shows the incomplete infiltration caused by the low permeability and poor rigidity of 20% vol. Saffil fibre with 5% alumina binder preform, which results in overall deformation of the preform during infiltration. Figure 8-19 shows the sectional view of this preform after infiltration. Examples of sound infiltration of RZ5DF alloy on 14% vol. fraction Saffil fibres with 5% silica binder are shown in figures 8-15 and 8-20.

Figure 8-16 shows the complete infiltration of magnesium alloy on 12% vol. Saffil + 9% carbon fibre with 5% alumina silicate binder. In this case, full infiltration was achieved due to the rigidity of the preform. Figure 8-21 shows the sectional view of this preform system after infiltration with the RZ5DF alloy. Figure 8-17 shows another example of incomplete infiltration caused by poor wetting between the carbon fibres and the material (RZ5DF alloy), which has again caused some deformation to the preform during infiltration. The sectional view of this preform after infiltration is shown in figure 8-22.

Figure 8-13 A typical view of the bottom face of the cast ingot, produced by squeeze infiltrating RZ5DF alloy into 20% vol. fraction Saffil fibre with 5% silica binder.

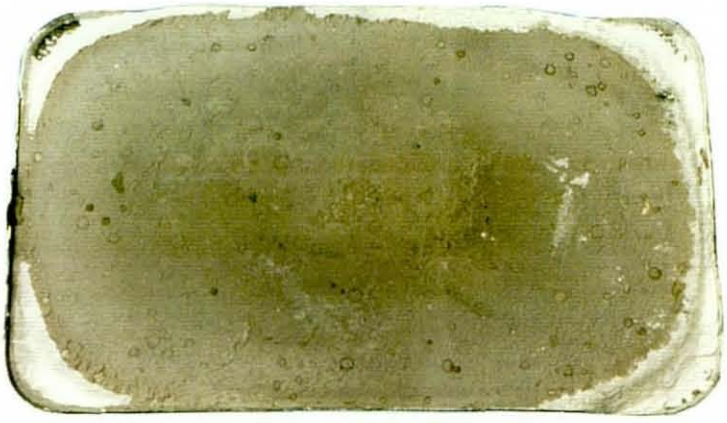


Figure 8-14 A typical view of the bottom face of the cast ingot, produced by squeeze infiltrating RZ5DF alloy into 20% vol. fraction Saffil fibre with 5% alumina binder.

Figure 8-15 A typical view of the bottom face of the cast ingot, produced by squeeze infiltrating RZ5DF alloy into 14% vol. fraction Saffil fibre with 5% silica binder.

Figure 8-16 A typical view of the bottom face of the cast ingot, produced by squeeze infiltrating RZ5DF alloy into 12% Saffil + 9% carbon vol. fraction fibre with 5% alumino-silicate binder.

Figure 8-17 A typical view of the bottom face of the cast ingot, produced by squeeze infiltrating RZ5DF alloy into 20% vol. fraction carbon fibre with 5% alumino-silicate binder.

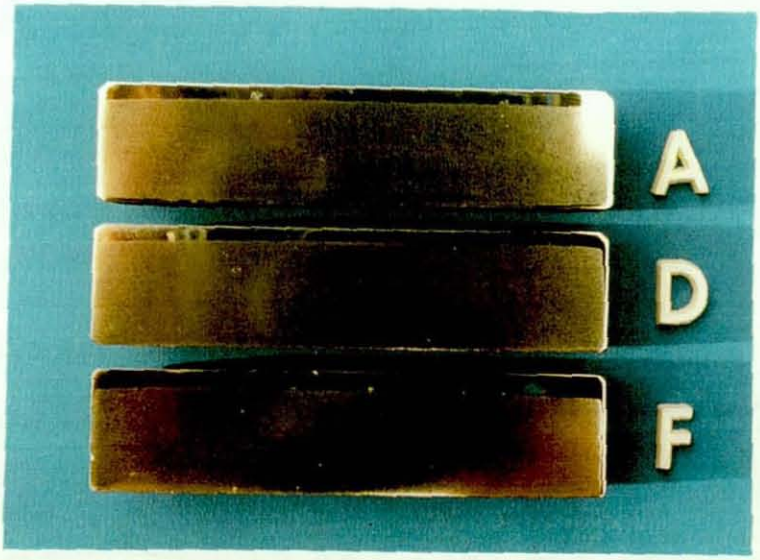


Figure 8-18 The sectional views of RZ5DF alloy infiltration for a 20% vol. fraction Saffil fibre with 5% silica binder.

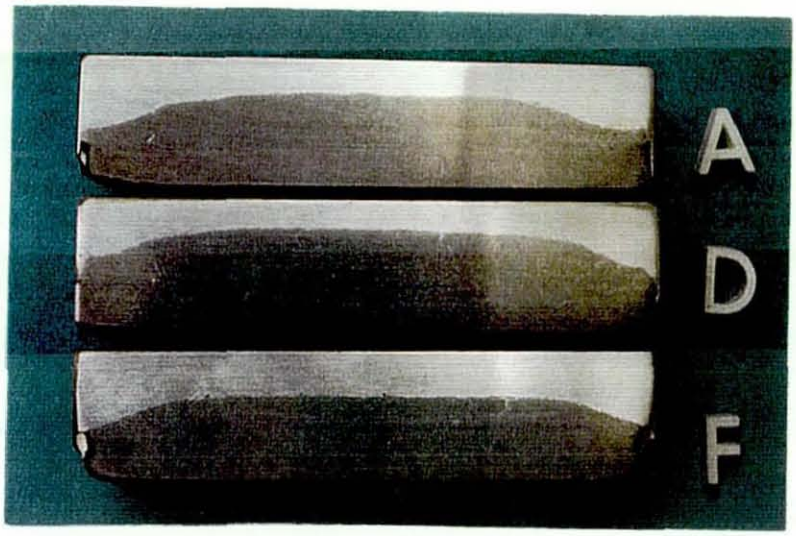


Figure 8-19 The sectional views of RZ5DF alloy infiltration for a 20% vol. fraction Saffil fibre with 5% alumina binder.

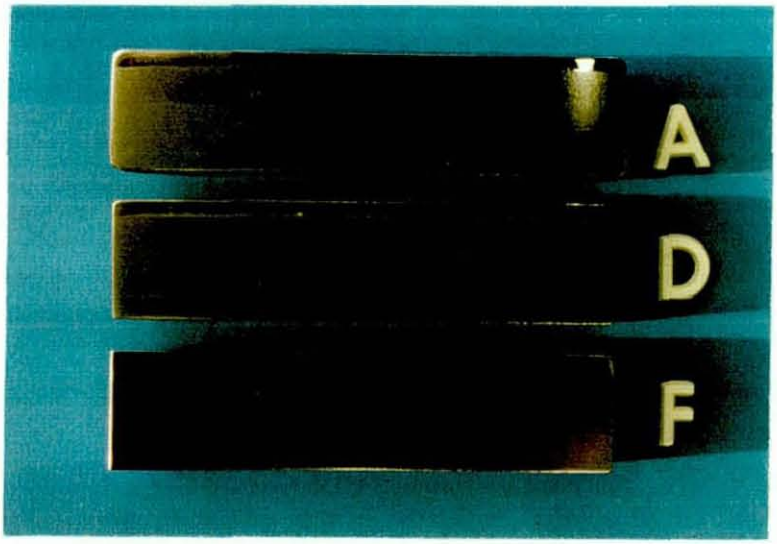


Figure 8-20 The sectional views of RZ5DF alloy infiltration for a 14% vol. fraction Saffil fibre with 5% silica binder.

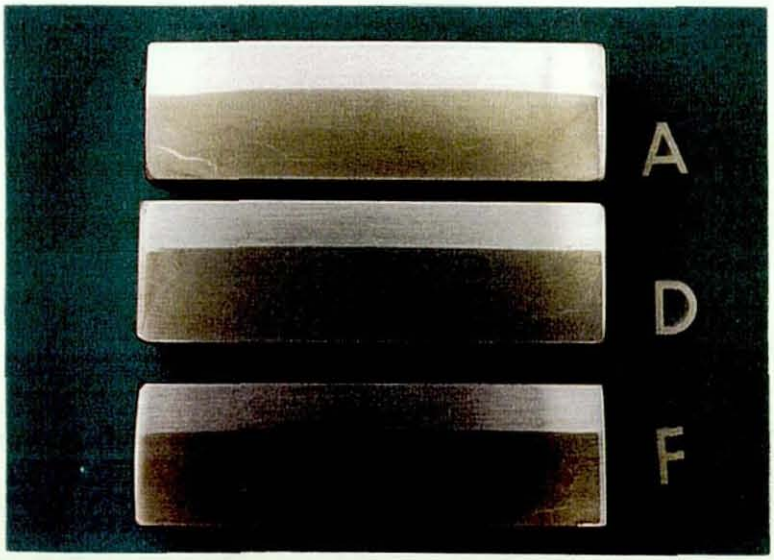


Figure 8-21 The sectional views of RZ5DF alloy infiltration for a 12% Saffil + 9% carbon vol. fraction fibre with 5% alumino-silicate binder.

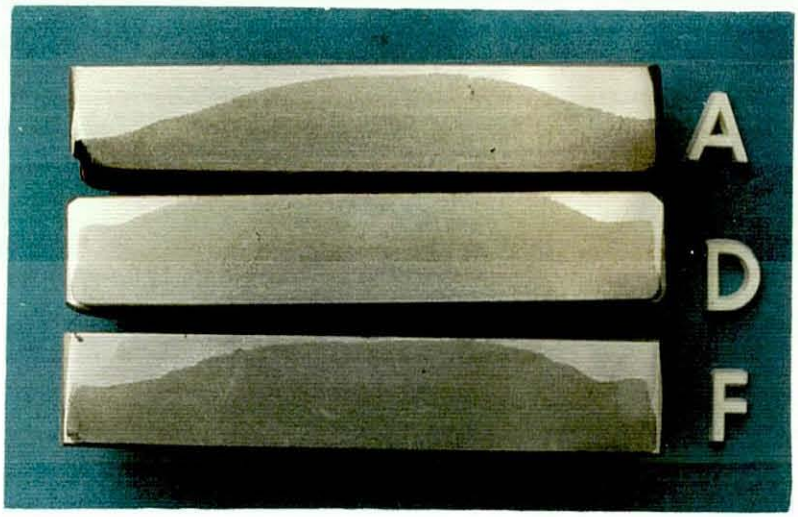
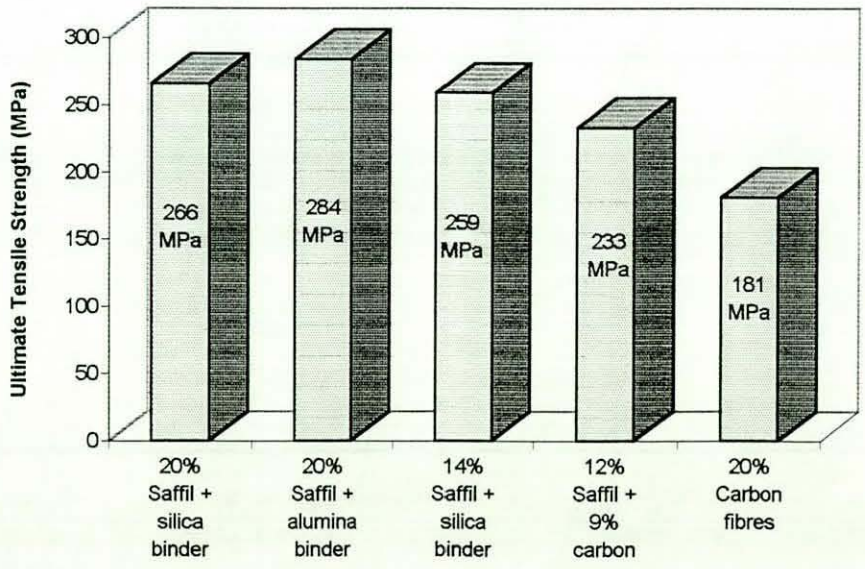


Figure 8-22 The sectional views of RZ5DF alloy infiltration for a 20% vol. fraction carbon fibre with 5% alumino-silicate binder.

8.3.3 The Tensile Properties Magnesium MMC Produced with Different Preform Systems

The tensile properties of magnesium matrix composite produced using the five different preform systems listed in section 6.3.3 have been compared. The results indicate that the 14% vol. fraction Saffil fibre preform proved to be the most promising, in terms of the ease of production and maximum value to strength performance. Figure 8-23 summarises the tensile properties of these preform systems when infiltrated with the RZ5DF alloy. Detailed tensile properties for RZ5DF MMC, produced with different preform systems, are displayed in table 2 of appendix B.



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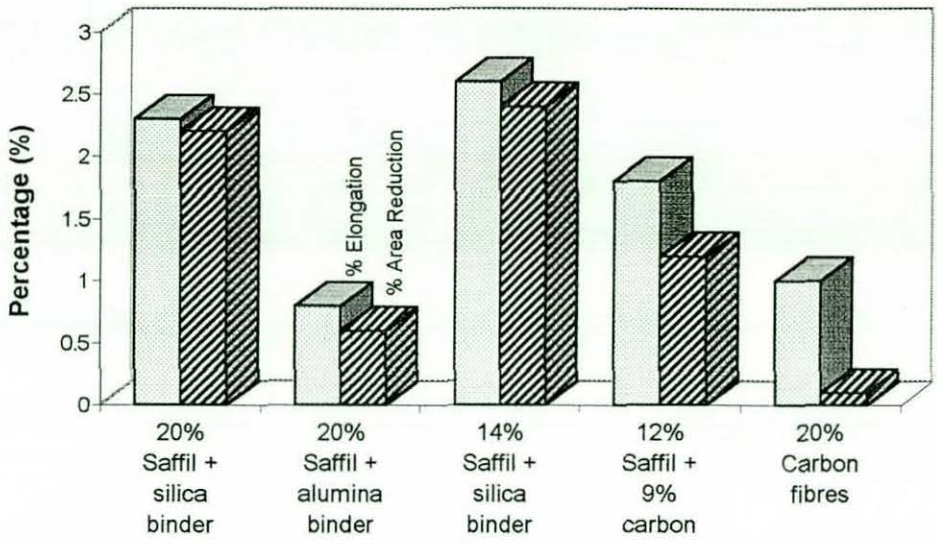


Figure 8-23 The plot of tensile properties for the five different preform systems listed in section 6.3.3 after infiltration with RZ5DF alloy. Cast with constant pouring temperature of 750°C and die temperature of 250°C.

8.3.4 The Hardness Values of Magnesium MMC Produced with Different Preform Systems

The hardness values of the magnesium matrix composites produced using different preform systems were compared. The results indicate a complex correlation between several factors that influence the hardness value of the magnesium matrix composite. Figures 8-24 and 8-25 show the material hardness along the 'longitudinal' and 'transverse' directions for five different preform systems.

The results, in general, show that 20% vol. Saffil with alumina binder provided the highest hardest value for the RZ5DF MMC. This was followed by the 12% vol. Saffil + 9% vol. carbon RZ5DF MMC, and subsequently, by the 20% vol. Saffil with silica binder RZ5DF MMC. Among the five composite systems, the 14% vol. Saffil with silica binder and the 20% vol. carbon with alumino-silicate binder have the lowest hardness. The hardness patterns of the composite systems are similar in both longitudinal and transverse directions. However, the composite system of 20% vol. Saffil with alumina binder showed higher hardness values along its transverse direction. This may be explained by the increased volume of fibres at the location where transverse hardness measurement were taken. The increase in fibre volume caused by deformation of the fibre preform after squeeze infiltration can be seen in figure 8-19.

The results in figures 8-24 and 8-25 show that the lower section of the 12% vol. Saffil and 9% vol. carbon composite (location: 3, 6, 9, 12 and 15) has a higher hardness in relation to the middle and top sections of the specimen. This may be attributed to a minor segregation effect on the smaller diameter (e.g Saffil) fibres, caused by the solidification front which pushes the fibres towards the bottom of the preform during infiltration.

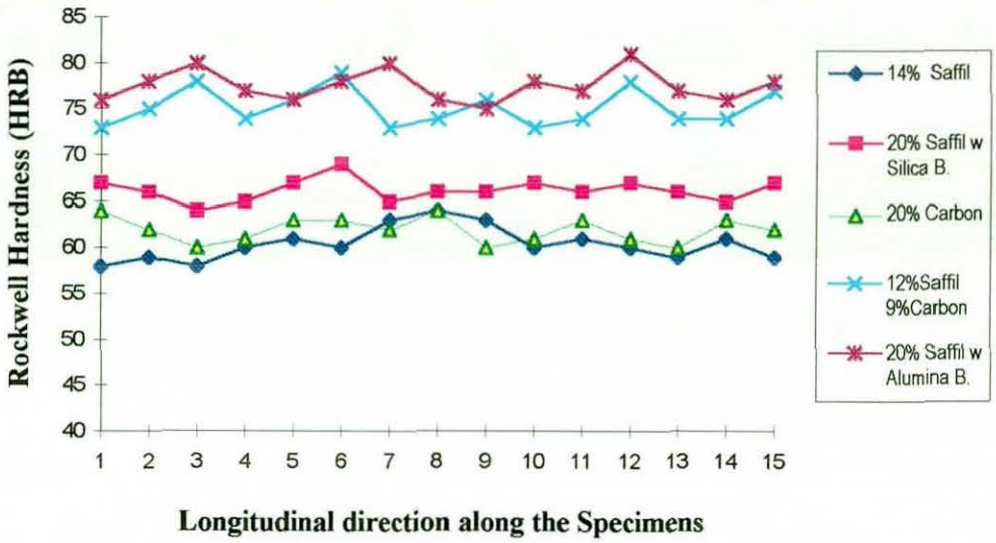


Figure 8-24 The plot of material hardness along the 'longitudinal' direction for five different preform systems. Cast with constant pouring temperature of 750°C and die temperature of 250°C.

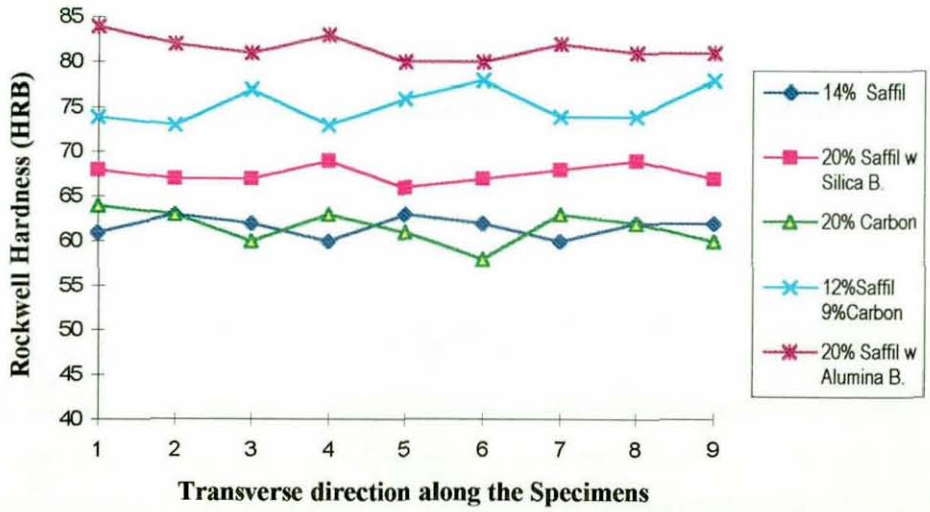


Figure 8-25 The plot of material hardness along the 'transverse' direction for five different preform systems. Cast with constant pouring temperature of 750°C and die temperature of 250°C.

8.3.5 The Microstructural Examination of MMCs Produced by Different Preform Systems

The results of the microscopic examinations of the preforms (listed in section 6.3.3) reinforced with RZ5DF alloy are presented in the sections below.

8.3.5.1 The Evaluation of the Magnesium-Saffil reinforced Composite

Microstructural examinations of magnesium composites reinforced with (i) 20% vol. Saffil + silica binder, (ii) 20% vol. Saffil + alumina binder and (iii) 14% vol. Saffil + silica binder show sound cast structures. However there was evidence of fibre clustering with the higher volume fraction of fibres. Typical examples of the cast structures are shown in figures 8-26, 8-27 and 8-59.

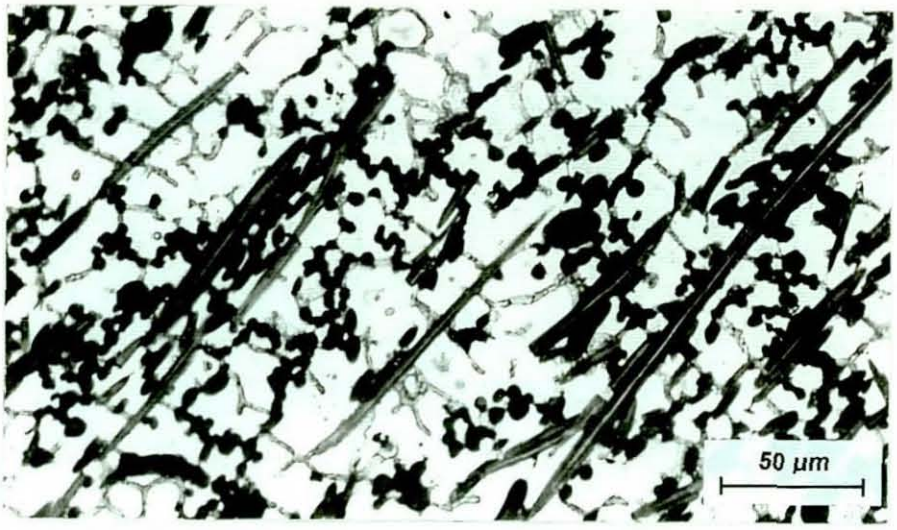


Figure 8-26 Micrograph of the RZ5DF-20% vol. Saffil with 5% silica binder reinforced composite

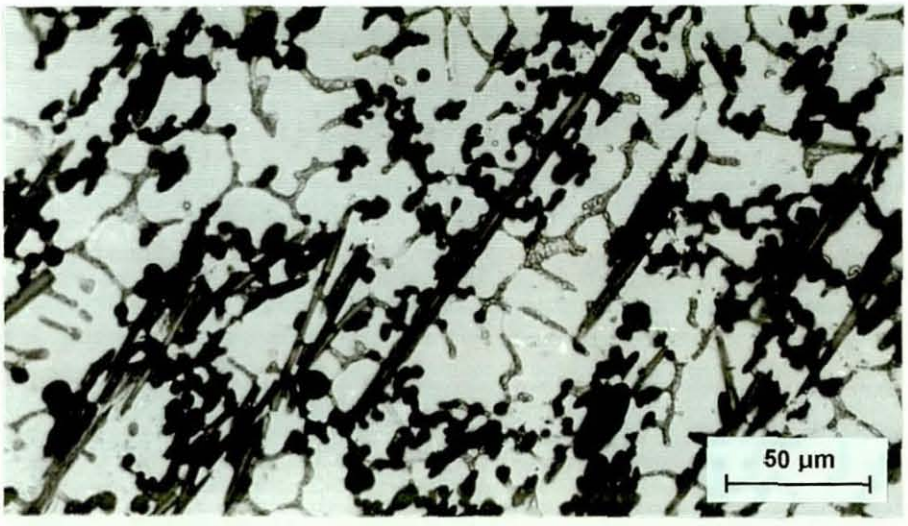


Figure 8-27 Micrograph of the RZ5DF-20% vol. Saffil with 5% alumina binder reinforced composite

8.3.5.2 The Evaluation of the Magnesium-Saffil + Carbon reinforced Composite

Microstructural examinations of magnesium composites reinforced with 12% vol. Saffil + 9% vol. carbon revealed significant shrinkage porosity in the region near the edge of the fabricated composite. Typical examples of the shrinkage porosity within the composite are shown in figure 8-28.

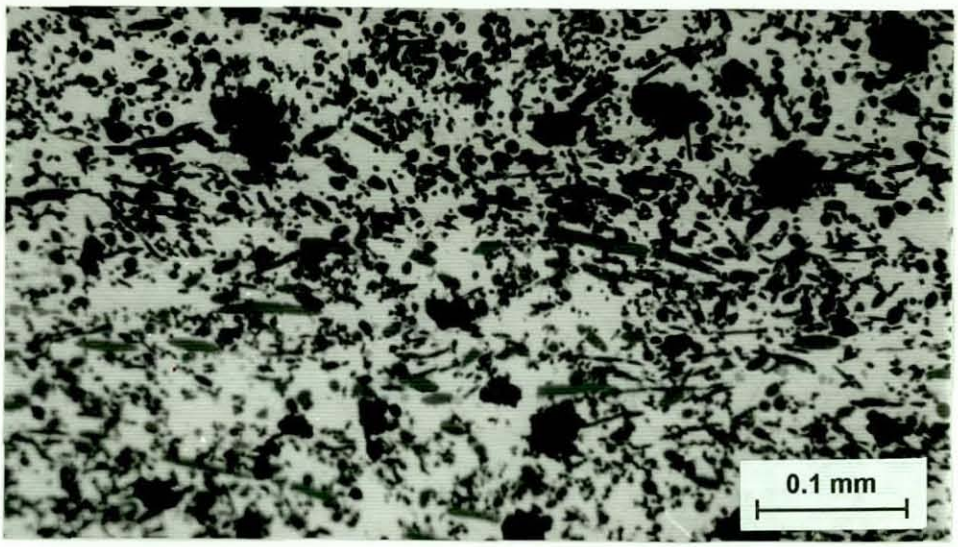


Figure 8-28 Micrograph of the RZ5DF-12% vol. Saffil + 9% vol. carbon reinforced composite, showing the presence of shrinkage porosity in a particular region of the composite specimen.

8.3.5.3 The Evaluation of the Magnesium-Carbon reinforced Composite

Microstructural examination of the carbon reinforced magnesium alloy composite shows that there is significant fibre fracture throughout the composite structure, seen in figure 8-29. This indicates ineffective fibre reinforcement, as the fibre lengths have fallen below the critical value. A discussion of the importance of critical fibre length for fibre reinforcement was presented previously in section 4.4.1. As a consequence, lower tensile properties were achieved, and this can be seen in figure 8-23 (i.e. for cast carbon reinforced magnesium alloy composite).

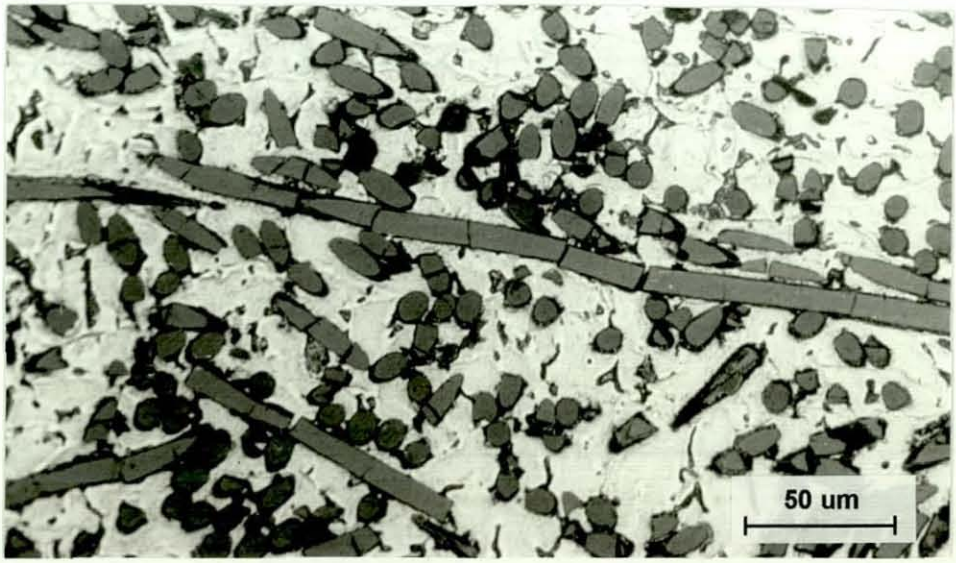


Figure 8-29 Micrograph of the RZ5DF-20% vol. carbon reinforced composite, showing the common feature of fibre fracture.

8.3.5.4 SEM Examination of the Fracture Face of Tensile Test Specimens

Fractographs were produced to study the influence of the composite system on the fracture morphology. Generally, the as-fabricated RZ5DF-Saffil composites show no sign of fibre pullout from the magnesium matrix, indicating strong bonding between the fibre and matrix and therefore effective strengthening of the matrix. A typical structure of a RZ5DF-Saffil MMC is shown in figure 8-40. Preform systems containing carbon fibres were the exception and exhibited fibre pullout. Typical fracture faces of RZ5DF discontinuous carbon fibre and hybrid Saffil + carbon fibre MMC are shown in figures 8-30 and 8-31 respectively.

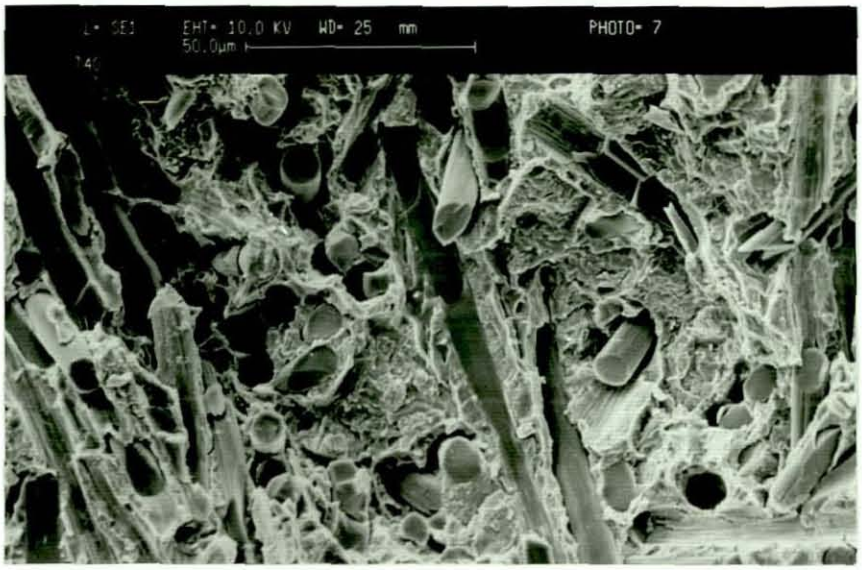


Figure 8-30 Fracture surface of RZ5DF-discontinuous 20% vol. carbon fibre MMC.

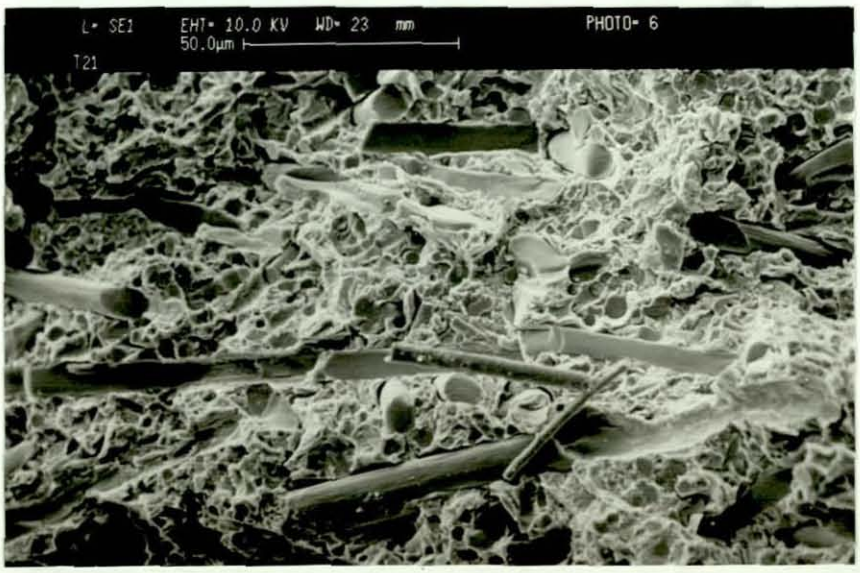


Figure 8-31 Fracture surface of RZ5DF-discontinuous 12% vol. Saffil + 9% vol. carbon fibre MMC.

8.3.6 The Permeability Measurement of Fibre Preforms

A study of fibre preform permeability was conducted to evaluate the effect of permeability on the degree of infiltration. The property of permeability considered in this research refers to the ease with which gases will permeate through a porous fibre network. This property is important as it influences the efficiency with which the melt infiltrates the preform. During infiltration, the metal must first displace the air from within the preform before it can replicate the cavity shape. Failure to do so will result in porosity in the MMC casting, results of which can be seen in figure 8-28. Table 8-1 summarises the results from the study. The permeability meter, test set-up and the preforms used for the tests are shown in section 7.6.2.2.1.

	Preform		Permeability Number
No.	**Fibres**	**Binders**	**Average of three Readings**
1	20% Saffil	Silica	180
2	20% Saffil	Alumina	160
3	14% Saffil	Silica	247
4	12% Saffil + 9% Carbon	Alumino-silicate	83
5	20% Carbon	Alumino-silicate	350

Table 8-1 Results of Permeability measurements for different preform systems. (The greater the permeability number the more permeable the preform is). Measurements were taken over an area of 18mm X 40mm X 15mm thickness, using a large orifice.

The permeability results show that the carbon fibre preform was the most permeable, followed by the preform containing 14% vol. Saffil fibres, 20% vol. Saffil fibres with silica binder, 20% vol. Saffil fibres with alumina binder. The least permeable was the preform containing 12% vol. Saffil + 9% vol. carbon fibres.

8.4 The Evaluation of Suitable Preform Temperature

Several observations can be made based on the process settings listed in section 7.6.2.3 and these observations and results are highlighted in the following sections, which include a comparison of tensile strength, material hardness and metallographical studies.

8.4.1 The Correlation between Preform Temperature and Applied Pressure on the Tensile Properties of Castings

The effects of preform temperature and applied pressure on UTS are summarised in figure 8-32. The results clearly indicate that the use of a high applied pressure (greater than 80 MPa) for squeeze

infiltration is unsuitable. The presence of fractured fibres within the magnesium matrix composite can be seen in figures 8-60 and 8-61, after production with an applied pressure of 100 MPa and 120 MPa respectively. The high applied pressure causes the fibres to fracture during squeeze infiltration and this subsequently reduces the effectiveness of fibres that are intended to strengthen the matrix.

In relation to the influence of preform temperature, the results indicate that the highest preform temperature of 750°C produced more consistent UTS values with different levels of applied pressure. It also shows that the optimum UTS value of 259 MPa was achieved with a preform temperature of 600°C and an applied pressure of 80 MPa. A lower preform temperature of 250°C, and an applied pressure of 80 MPa, produced a UTS value of 201 MPa. The cause of the decrease in UTS values can be seen in figure 8-38, i.e. the clustering of fibres which led to more initiation points for failure within the MMCs, and will be discussed in section 10.4.

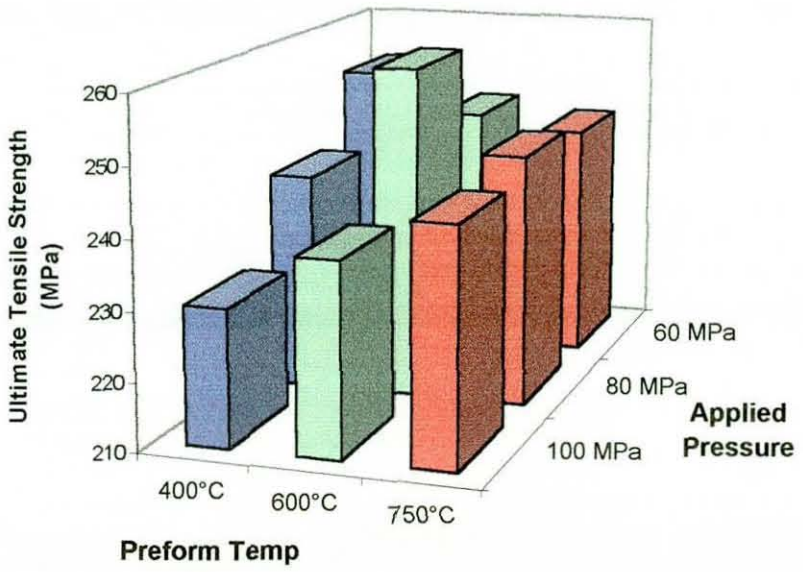


Figure 8-32 The plot of UTS for RZ5DF-14% vol. Saffil MMC produced from various combinations of Applied Pressure and Preform Temperature.

Detailed tensile property results for the squeeze infiltrated RZ5DF-14% vol. Saffil MMC, produced for the investigation of the correlation between applied pressure and preform temperature, are presented in table 3 of appendix B.

8.4.2 The Correlation between Preform Temperature and Applied Pressure and the Hardness of Castings

Hardness measurements were conducted using the Rockwell B scale. Figures 8-33 and 8-34 show the effects of preform temperature on the hardness values. The results show that composites produced with the lowest preform temperature of 400°C and lowest applied pressure of 60 MPa produced a

significant degree of variation in hardness values along the longitudinal direction and, to some extent, along the transverse direction. Such variations in hardness may be explained by the clustering of fibres in the central part of the infiltrated preform shown in section 8-41. At the other extreme, composites produced with the highest values used for the process variables on the preform, i.e. preform temperature of 750°C and applied pressure of 100 MPa caused some variation in hardness values as seen in figures 8-33 and 8-34. The reason may be attributed to the combination of an increase in fluidity (caused by the high preform temperature) and the higher applied pressure with greater energy, which creates movement of fibres within the composite during infiltration.

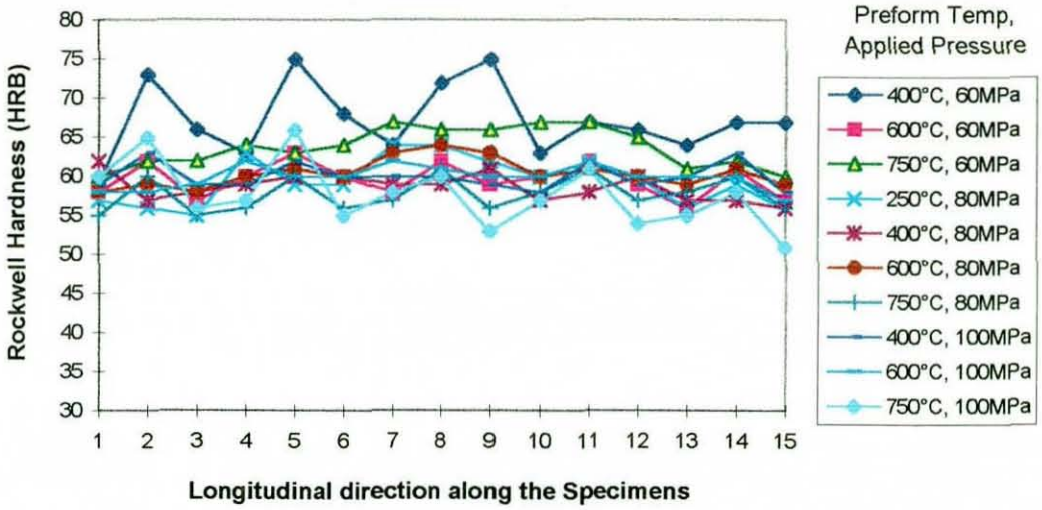


Figure 8-33 The plot of material hardness along the longitudinal direction of the squeeze infiltrated RZ5DF-14% vol. fraction Saffil fibres produced under different combinations of preform temperatures and applied pressures.

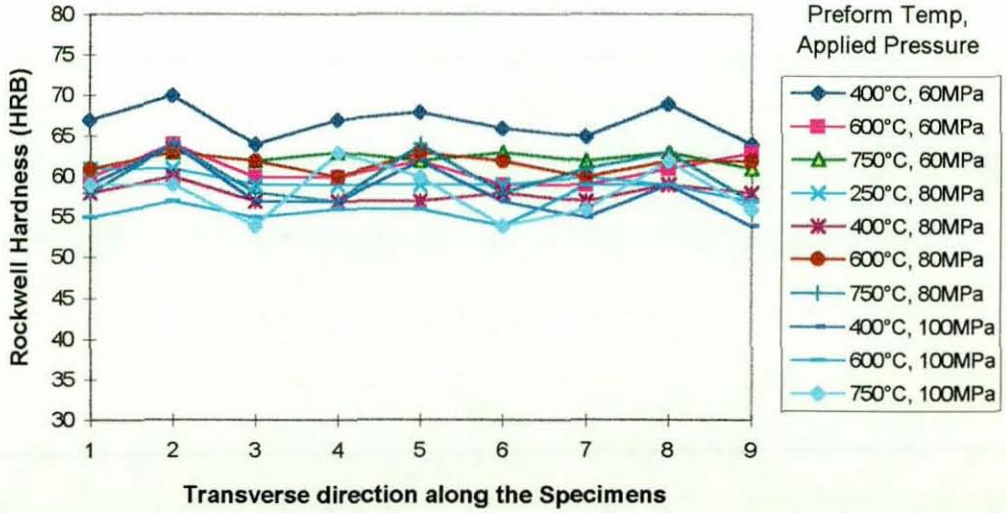


Figure 8-34 The plot of material hardness along the transverse direction of the squeeze infiltrated RZ5DF-14% vol. fraction Saffil fibres produced under different combinations of preform temperatures and applied pressures.

8.4.3 The Correlation between Preform Temperature and Applied Pressure and Cast Microstructure

The preliminary microstructural evaluation of the squeeze infiltrated RZ5DF-14% vol. Saffil fibre interface showed that a lower preform temperature produced more densely packed fibres at the preform surface. The infiltrated region, displayed in figures 8-35 to 8-38, illustrates the significance of preform temperature on the packing of the fibres at the preform surface.

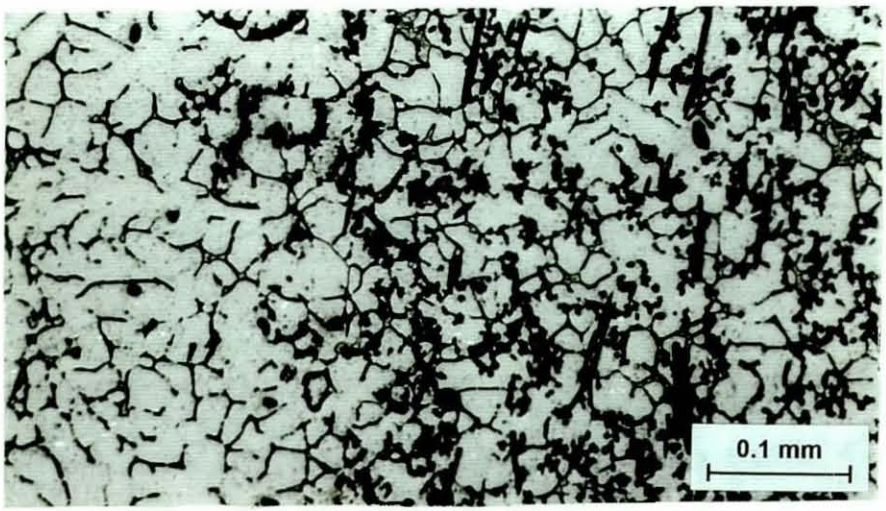


Figure 8-35 A micrograph taken at the preform infiltration region of a squeeze infiltrated specimen produced with a preform temperature of 750°C.

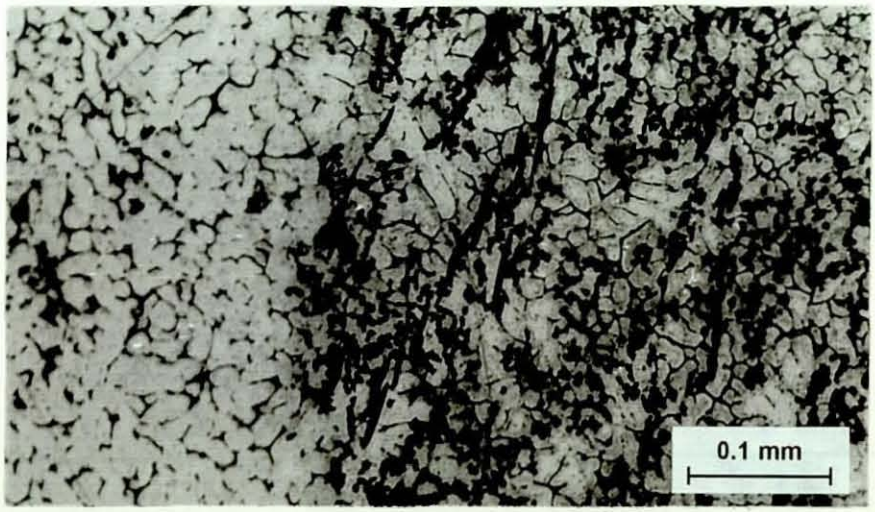


Figure 8-36 A micrograph taken at the preform infiltration region of a squeeze infiltrated specimen produced with a preform temperature of 600°C.

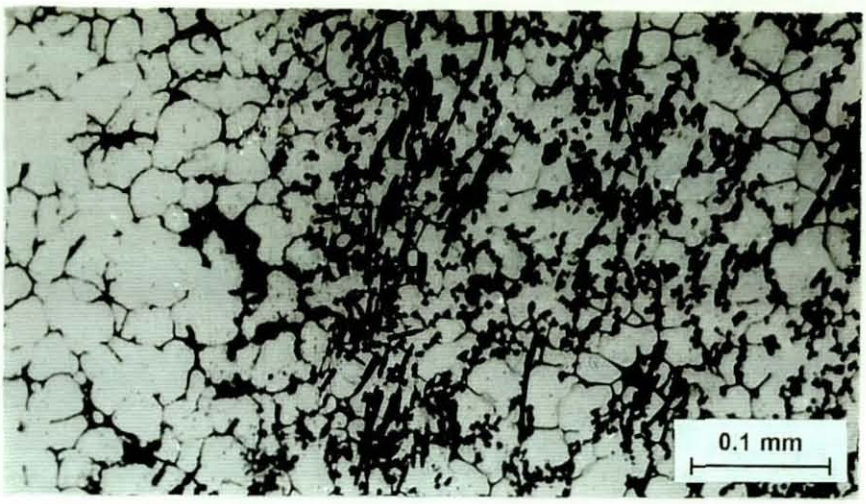


Figure 8-37 A micrograph taken at the preform infiltration region of a squeeze infiltrated specimen produced with a preform temperature of 400°C.

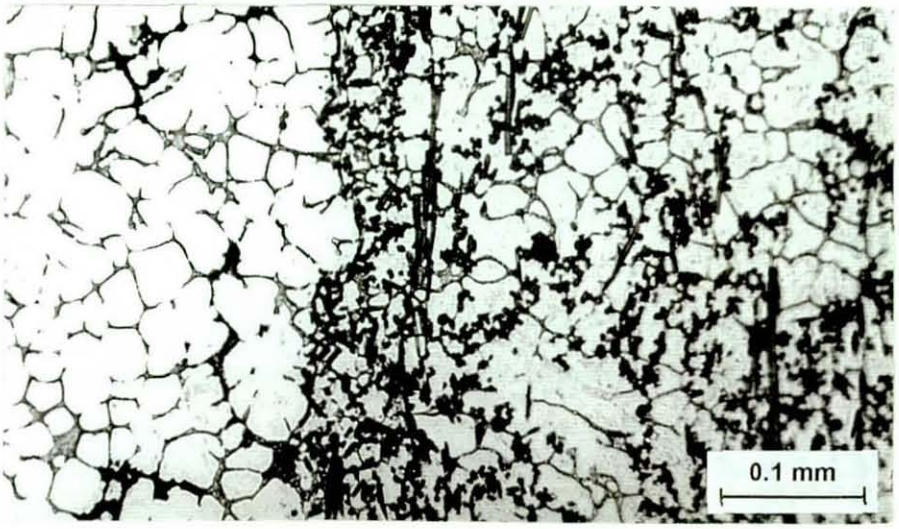


Figure 8-38 A micrograph taken at the preform infiltration region of a squeeze infiltrated specimen produced with a preform temperature of 250°C.

Detailed examination of the distribution of fibres within the infiltrated preform showed an increased incidence of fibre clustering for the specimens squeeze infiltrated with the lower preform temperatures of 400°C and 250°C, as shown in figures 8-37 and 8-38 respectively. The increased clustering of fibres can be seen if this comparison is made against the microstructures produced at the higher preform temperatures of 750°C and 600°C, shown in figures 8-35 and 8-36 respectively.

SEM examinations were conducted of the fracture faces of the tensile test specimens to study the effects of preform temperature and applied pressure in relation to the UTS. The examination showed that a lower preform temperature promotes fibre clustering, seen in figure 8-39. Such clustering usually results in fibres coming in contact with each other and this reduces the tensile properties. This may explain why most MMCs squeeze infiltrated with a 400°C preform temperature have much lower tensile properties. However, specimens squeeze infiltrated with a preform temperature of 400°C and an

applied pressure of 60 MPa were the exceptions because high tensile properties were attained. Such an increase in tensile properties may be attributed to the preform deformation that increases fibre concentration in the central part of the infiltrated preform. Detailed discussion of the fibre concentration is presented in section 8.4.3.1.

The fracture surface examination of MMC samples produced with a 600°C preform temperature revealed an even distribution of fibres throughout the structure, as displayed in figure 8-40. This may explain why MMCs squeeze infiltrated with a 600°C preform temperature and 80 MPa applied pressure had the highest tensile properties.

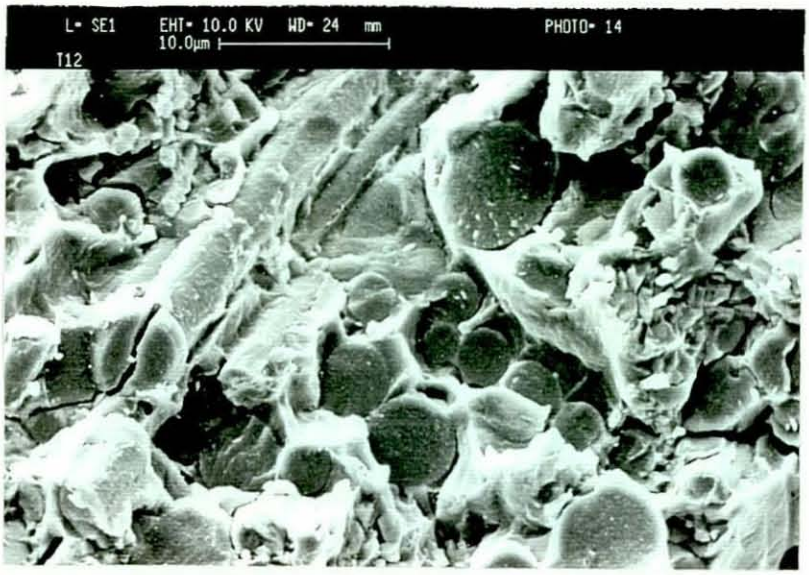


Figure 8-39 SEM micrograph of the fracture face of a squeeze infiltrated RZ5DF-14% vol. Saffil MMC produced with 80 MPa applied pressure and 400°C preform temperature.

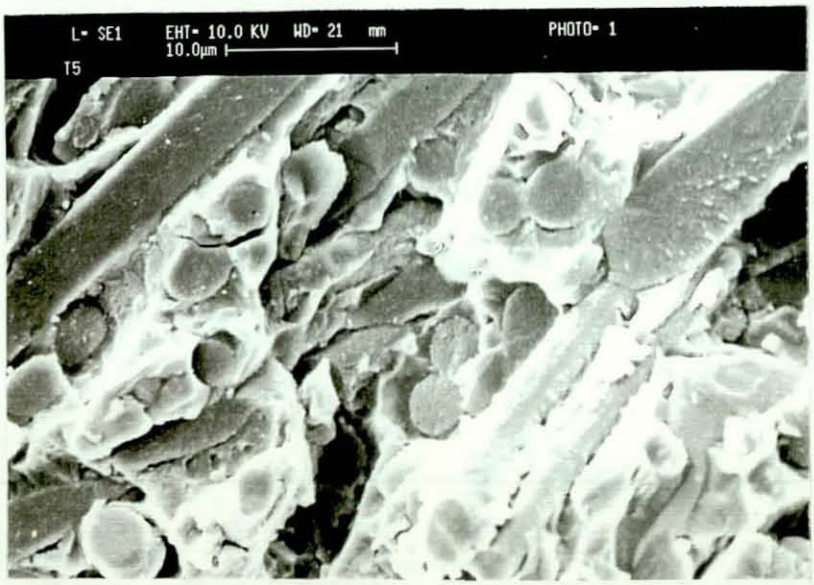


Figure 8-40 SEM micrograph of the fracture face of a squeeze infiltrated RZ5DF-14% vol. Saffil MMC produced with 80 MPa applied pressure and 600°C preform temperature.

8.4.3.1 The Microstructural Evaluation of the Distribution of Fibres in Specimens Squeeze Infiltrated with a Preform Temperature of 400°C and Applied Pressure of 60MPa

Microstructural examinations were conducted on the specimen squeeze infiltrated with a preform temperature of 400°C and an applied pressure of 60 MPa to examine the increase in hardness value at the central region of the specimen, seen in figures 8-33 and 8-34. The micro examination revealed concentration of fibres in the central part of the infiltrated preform, which subsequently caused the rise in UTS and increase in value of hardness. This effect is mainly due to the poor combination of process parameters, i.e. low preform temperature and insufficient applied pressure. The concentration of fibres due to preform deformation is illustrated in figure 8-41.

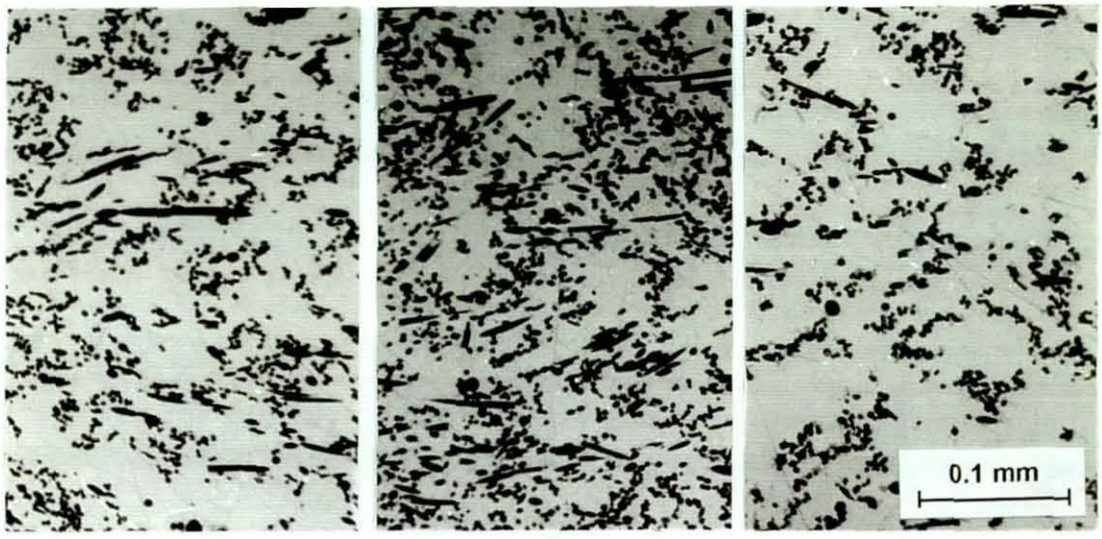


Figure 8-41 Microstructure showing different parts of the squeeze infiltrated RZ5DF-14% vol. fraction Saffil specimen produced with a preform temperature of 400°C and applied pressure of 60 MPa. The sequence is (i) top, (ii) centre and (iii) bottom portion of the fabricated composite.

8.5 The Evaluation of the Effects of Different Applied Pressures

Based on the process settings listed in section 7.6.2.4, several observations were made and are highlighted with the results in the following sections, which include a comparison of tensile strengths, material hardnesses and metallographic structures.

8.5.1 The Evaluation of the Effects of Different Applied Pressures on Cast Tensile Strength

The effects of applied pressure on both the alloys and MMCs were investigated to establish an understanding of the relationship between process parameters and tensile properties. The effects of applied pressure on the tensile properties of squeeze cast RZ5DF alloys and its composites are presented in figures 8-42 and 8-43. Detailed results of the tensile properties are presented in table 4 of appendix B.

For the squeeze cast RZ5DF alloy, the maximum UTS value was achieved with an applied pressure of 100 MPa and the lowest UTS value was achieved with atmospheric pressure 0.1 MPa applied in the gravity die casting process. As shown in figure 8-42, the results indicate a significant increase in UTS value up to 60 MPa applied pressure, after this point, further increase in applied pressure does not provide much improvement in UTS. The effects of applied pressure between 60 MPa and 100 MPa are marginal, producing only a 0.6% increase in UTS value for each 20 MPa increase in applied pressure. At an applied pressure of 120 MPa, there is a 6% decrease in the UTS relative to the specimens produced with an applied pressure of 100 MPa.

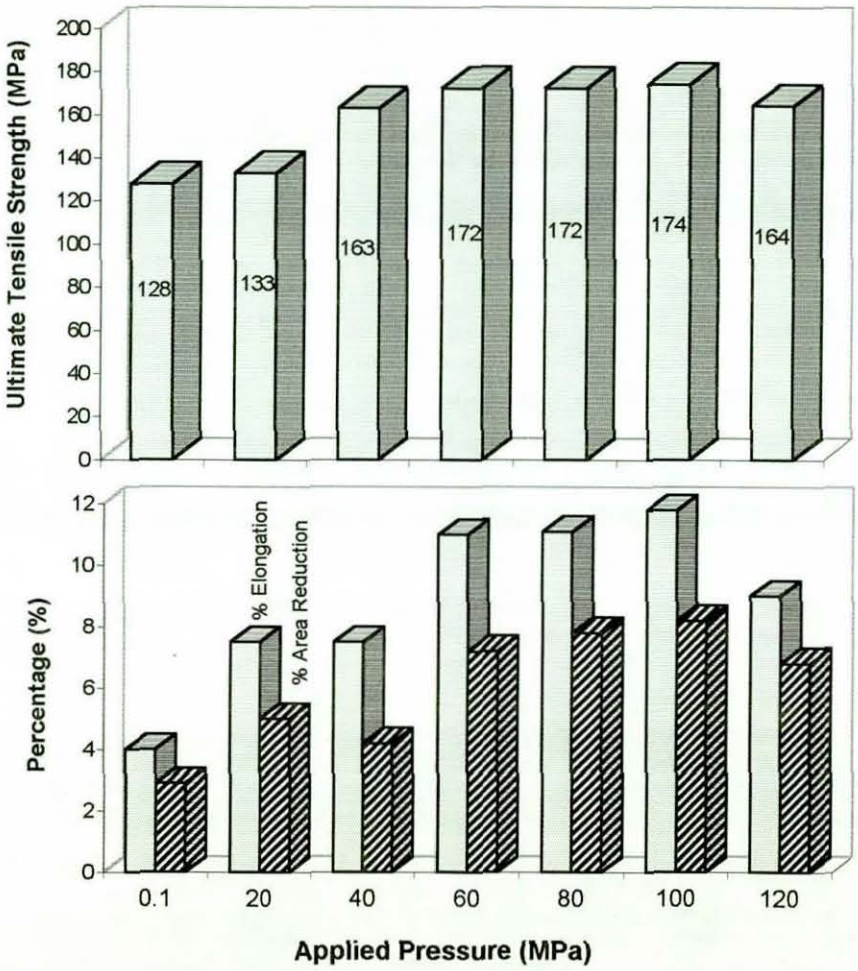


Figure 8-42 The effects of applied pressure on the tensile properties of RZ5DF alloy

For squeeze infiltrated RZ5DF matrix with 14% vol. fraction Saffil fibres, a maximum UTS value of 259 MPa was achieved at an applied pressure of 80 MPa. The lowest UTS value was, again, attained at an applied pressure of 0.1 MPa (atmospheric pressure). The results shown in figure 8-43 indicate a significant increase in UTS when the applied pressure is raised from 0.1 MPa to 80 MPa. Above 80MPa, further increases in applied pressure reduce the UTS values significantly. At an applied pressure of 100 MPa, the UTS values decreases from 259 MPa to 238 MPa and subsequently to 204 MPa with an applied pressure of 120 MPa.

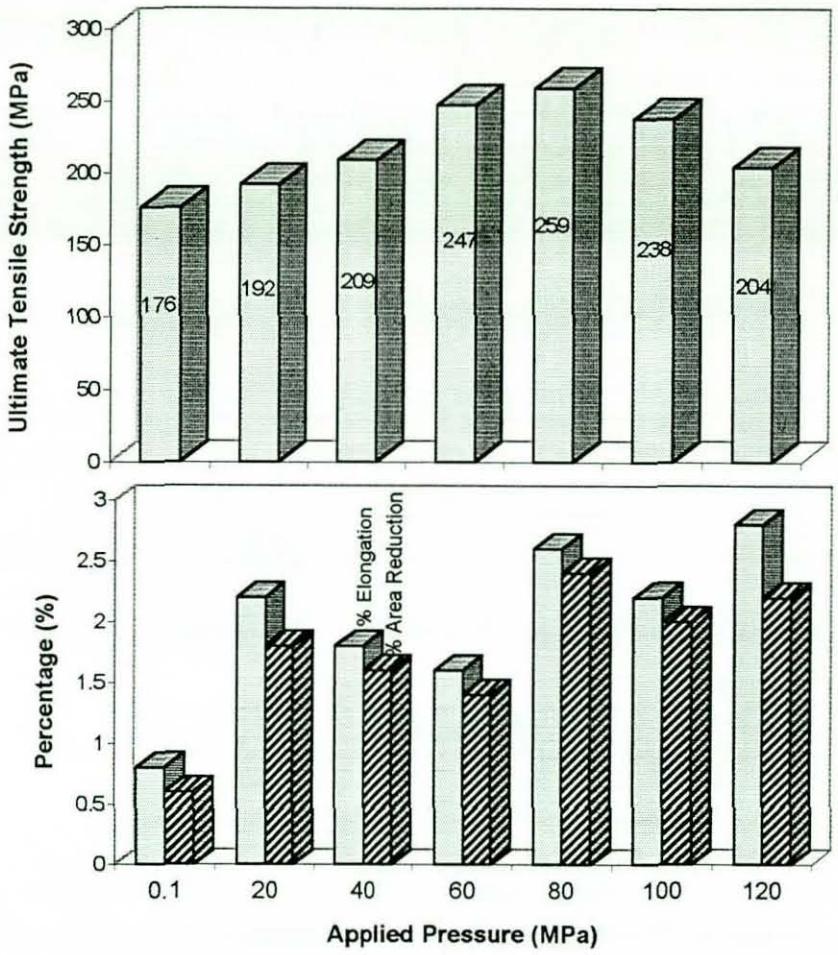


Figure 8-43 The effects of applied squeeze infiltration pressure on the tensile properties of RZ5DF matrix with 14% vol. fraction Saffil fibres

8.5.2 The Evaluation of the Effects of Different Applied Pressures on Cast Hardness Properties

The effects of applied pressure on the hardness values of RZ5DF casting are illustrated in the following sections.

8.5.2.1 The Effects of Applied Pressure on the Hardness Values of the Cast RZ5DF Alloy

The hardness values along the longitudinal and transverse directions of the RZ5DF alloy castings produced with different applied pressures are shown graphically in figures 8-44 and 8-45. The results indicate that the applied pressure has minimum effect on the hardness, since the majority of the cast magnesium alloy hardness values fall within the range of 14 HRB to 20 HRB. However, a detailed examination of figure 8-44 and 8-45 shows that the castings produced with a lower applied pressure (i.e. 20 MPa and 40 MPa) have lower hardness values, this is to be expected since specimens produced at a low applied pressure contain voids and therefore have a lower hardness compared to a sound material.

According to the British Standard BS 891 for Rockwell hardness testing, the next higher or lower hardness scale should be used if the hardness rises above a value of 100 or falls below a value of 20 on any scale in order to avoid inaccuracy. The majority of the hardness values attained for the RZ5DF alloy falls below 20 HRB and, in accordance with the British Standard, the next softer scale should be used, i.e. HRA scale. Since the intention of this experiment was to compare the improvements that fibre reinforcement brings to the alloys, the Rockwell B scale was selected in order to display the hardness values of the MMCs and the alloys using a single scale. Figures 8-81 and 8-82 will illustrate this point.

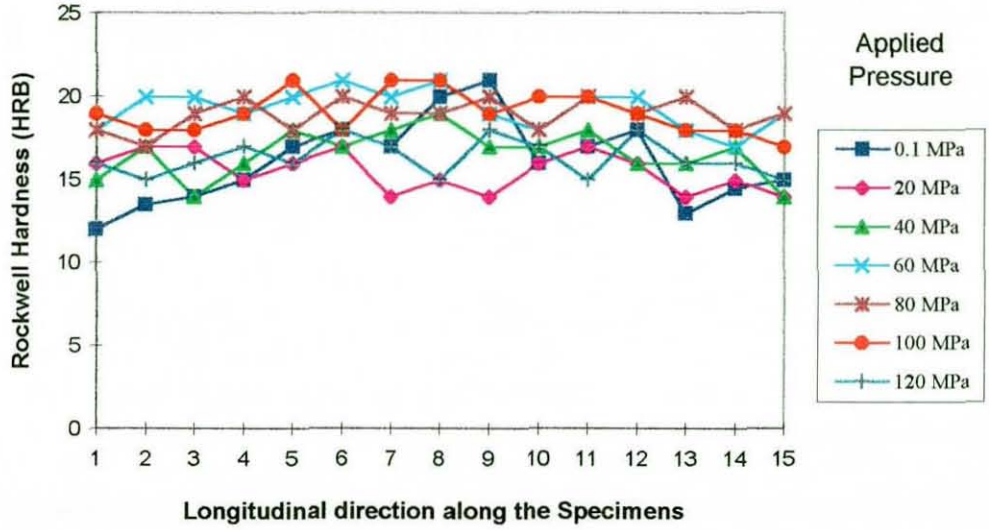


Figure 8-44 The plot of material hardness along the longitudinal direction of the squeeze cast RZ5DF alloy, cast with constant pouring temperature of 750°C and die temperature of 250°C.

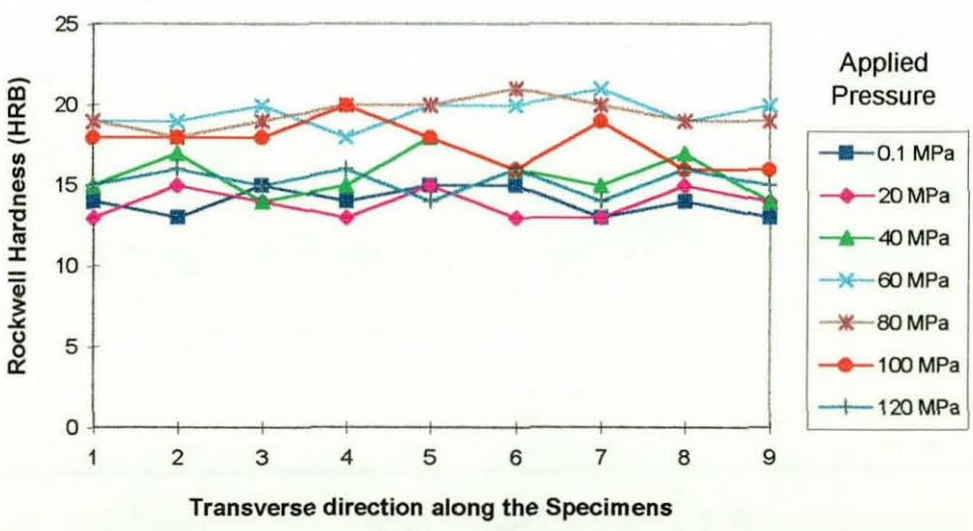


Figure 8-45 The plot of material hardness along the transverse direction of the squeeze cast RZ5DF alloy, cast with constant pouring temperature of 750°C and die temperature of 250°C.

8.5.2.2 The Effects of Applied Pressure on the Hardness Values of the Magnesium MMC

The hardness values of 14% vol. Saffil fibre reinforced RZ5DF alloy, along the longitudinal and transverse directions, produced using different applied pressures are shown graphically in figures 8-46 and 8-47.

The results again suggest that specimens produced at a low applied pressure may contain voids and, as a consequence, have a lower hardness than sound material. The specimens produced with 0.1 MPa and 20 MPa applied pressure may be cited. The voids and shrinkage porosity in these castings was caused by inadequate pressure for the full infiltration of molten magnesium into the porous fibre preform, resulting in lower hardness values.

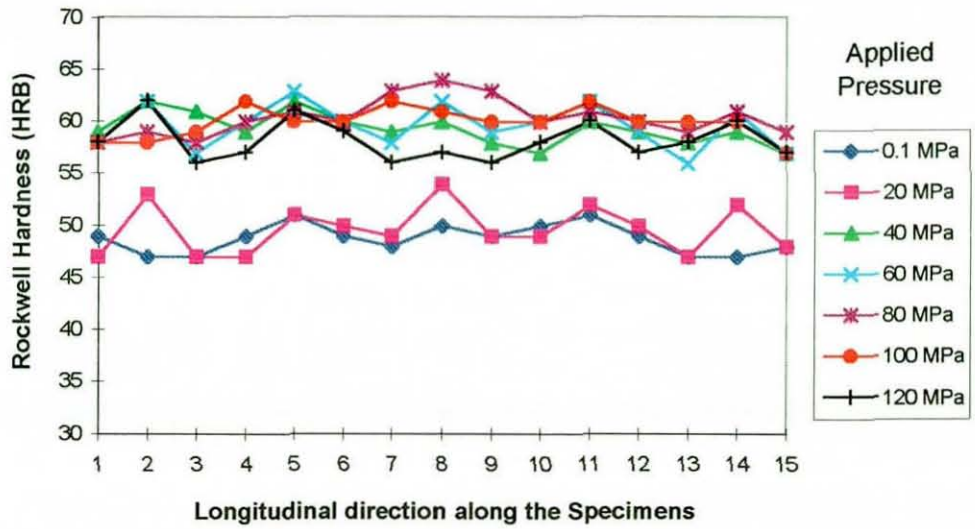


Figure 8-46 The plot of material hardness along the longitudinal direction of the squeeze infiltrated RZ5DF alloy with 14% vol. fraction Saffil fibres, cast with constant pouring temperature of 750°C and die temperature of 250°C.

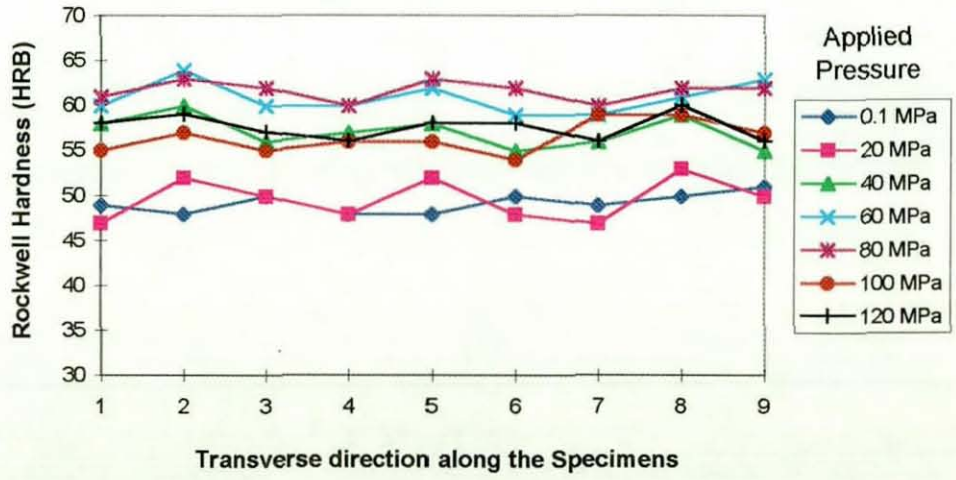


Figure 8-47 The plot of material hardness along the transverse direction of the squeeze infiltrated RZ5DF alloy with 14% vol. fraction Saffil fibres, cast with constant pouring temperature of 750°C and die temperature of 250°C.

8.5.3 The Evaluation of the Effects of Different Applied Pressures on the Microstructure of Cast Specimens

Microstructure examinations were conducted to examine the influence of applied pressure on the cast structure. The microstructures in figures 8-48 to 8-54 indicate that a minimum applied pressure of 40 MPa is required to suppress any form of microporosity. The results also revealed that grain size generally decreases as the applied pressure increases. However, as the applied pressure increases above 100 MPa there is an increased tendency for variation in grain sizes.

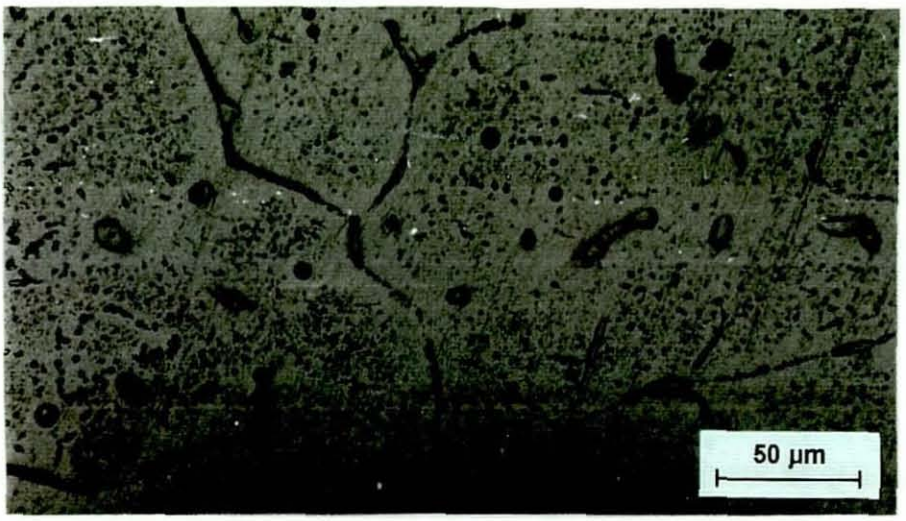


Figure 8-48 Optical microstructure of squeeze cast RZ5DF alloy produced under atmospheric pressure (0.1MPa), providing an average grain size of 127µm.

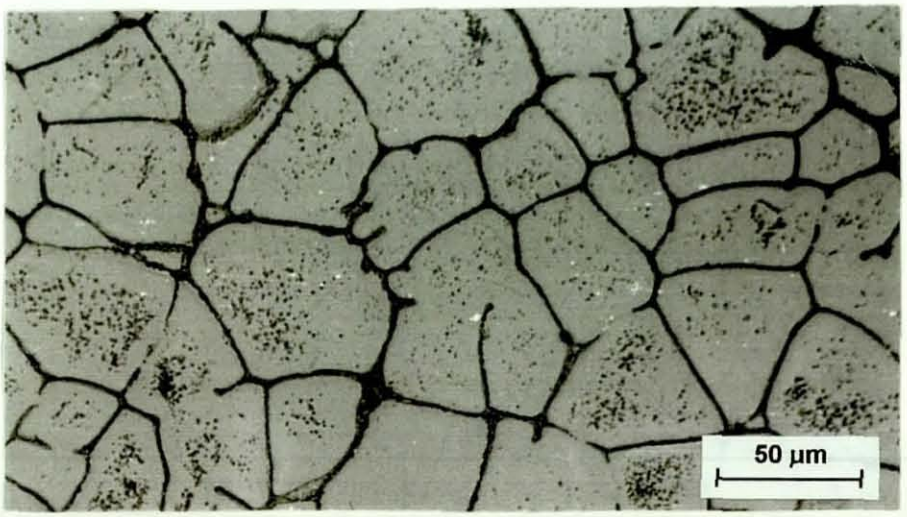


Figure 8-49 Optical microstructure of squeeze cast RZ5DF alloy produced with applied pressure of 20 MPa, providing an average grain size of 32µm.

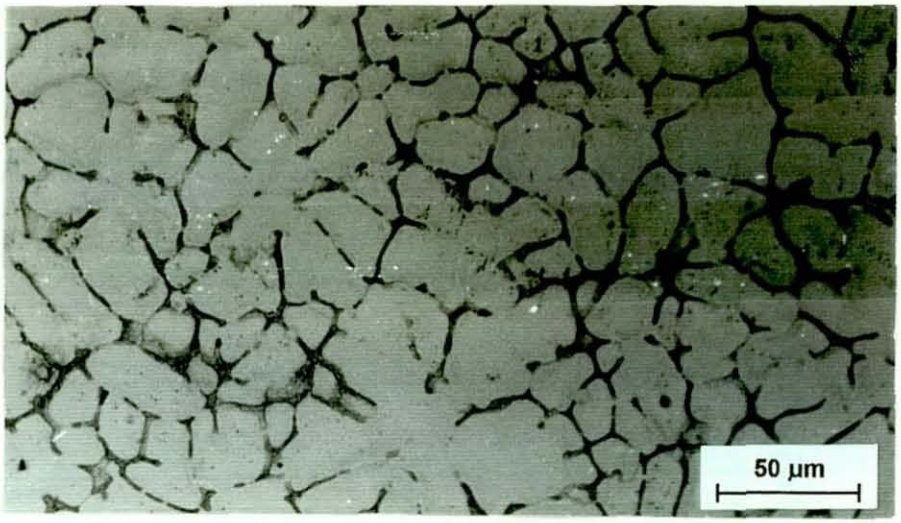


Figure 8-50 Optical microstructure of squeeze cast RZ5DF alloy produced with applied pressure of 40 MPa, providing an average grain size of 23µm.

Figure 8-51 Optical microstructure of squeeze cast RZ5DF alloy produced with applied pressure of 60 MPa, providing an average grain size of 21µm.

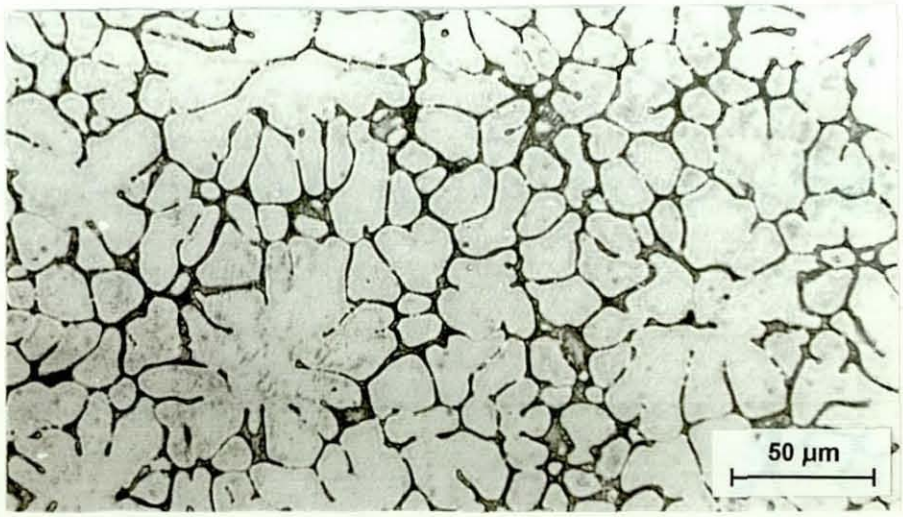


Figure 8-52 Optical microstructure of squeeze cast RZ5DF alloy produced with applied pressure of 80 MPa, providing an average grain size of 20 µm.

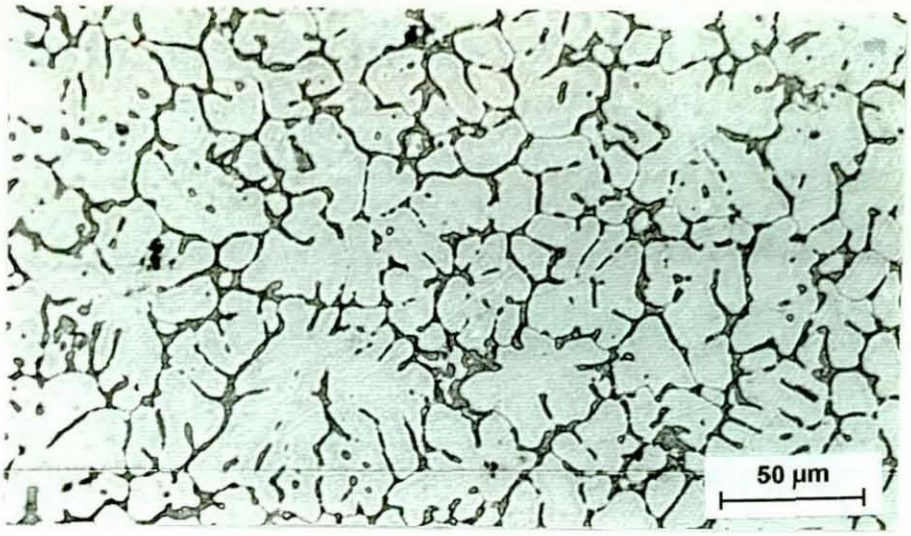


Figure 8-53 Optical microstructure of the squeeze cast RZ5DF alloy produced with applied pressure of 100 MPa, providing an average grain size of 19 µm.

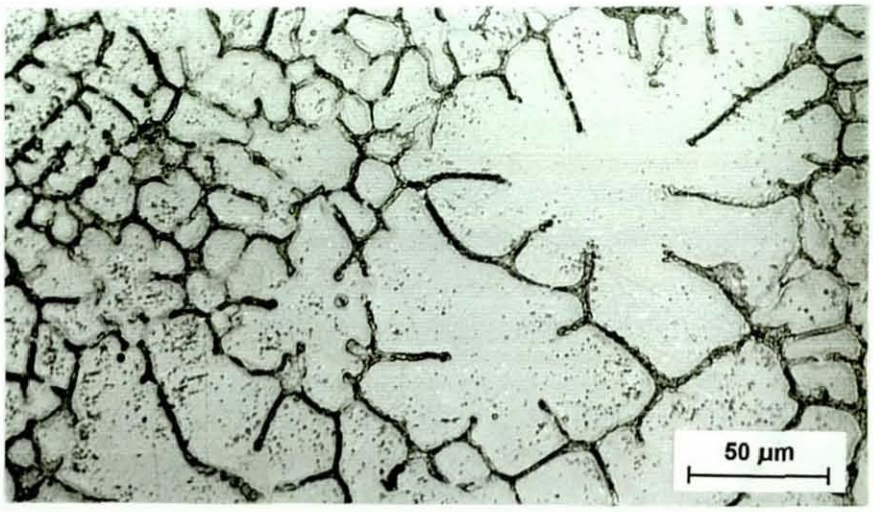


Figure 8-54 Optical microstructure of squeeze cast RZ5DF alloy produced with applied pressure of 120 MPa, providing an average grain size of 35µm.

Figures 8-55 to 8-61 illustrate the influence of applied pressure on the cast structure of RZ5DF alloy reinforced with 14% vol. fraction Saffil fibres. The cast RZ5DF MMC structures shown in figures 8-55, 8-56 and 8-57 reveal signs of microporosity for specimens produced with an applied pressure below 60 MPa. Although the microporosity is not clear in the micrographs (figures 8-56 to 8-57), the microporosity was obvious when adjusting the depth of field under an optical microscope. The microporosity was mainly present at the grain boundaries where it would be expected since those areas were the last regions to solidify. To suppress any form of microporosity, a minimum applied pressure of 60 MPa is therefore required. As applied pressure exceeds 80MPa there is an increased tendency for fibre fracture and clustering of fibres, which can be seen in figures 8-60 and 8-61.

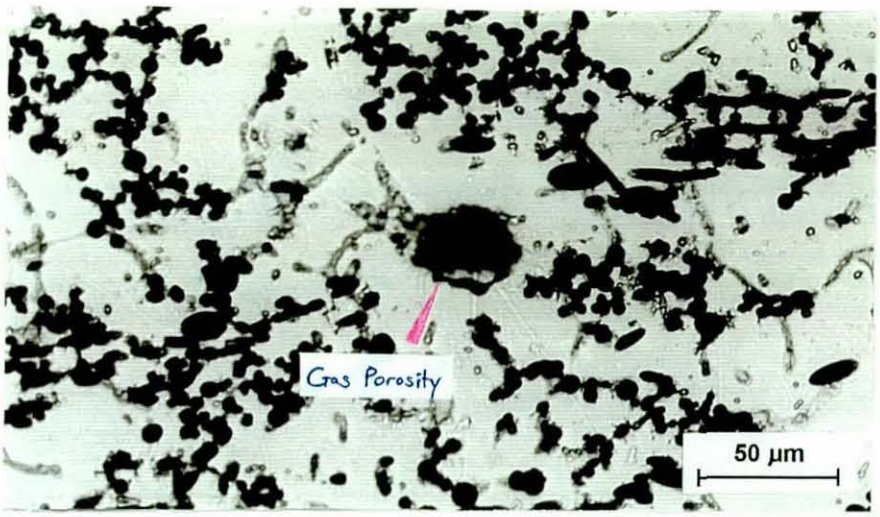


Figure 8-55 Optical microstructure of squeeze infiltrated RZ5DF-14% vol. fraction Saffil fibres produced under atmospheric pressure (0.1MPa), showing significant fibre clustering or segregation to the grain boundaries.

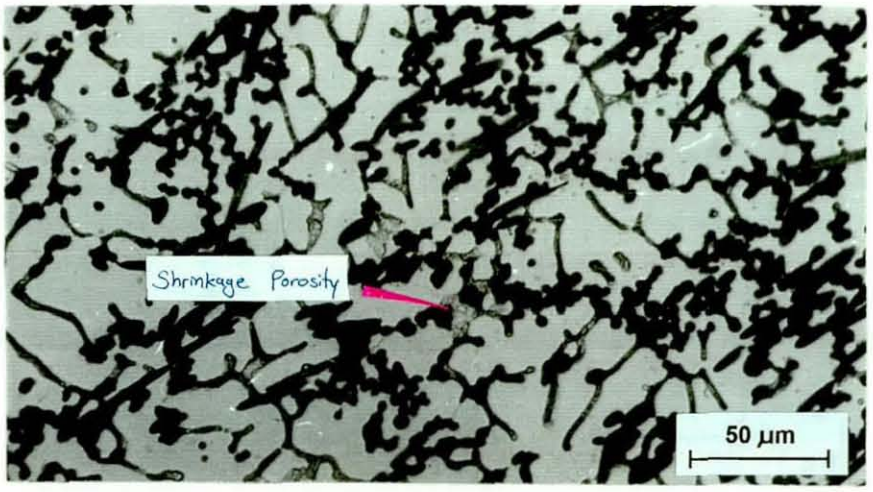


Figure 8-56 Optical microstructure of squeeze infiltrated RZ5DF-14% vol. fraction Saffil fibres produced under applied pressure of 20 MPa, showing signs of shrinkage porosity at the grain boundaries.

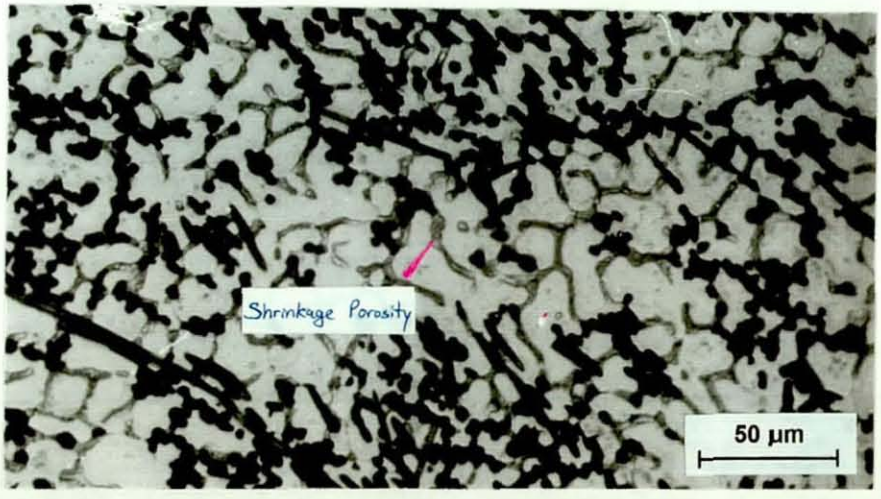


Figure 8-57 Optical microstructure of squeeze infiltrated RZ5DF-14% vol. fraction Saffil fibres produced under applied pressure of 40 MPa, showing signs of shrinkage porosity at the grain boundaries.

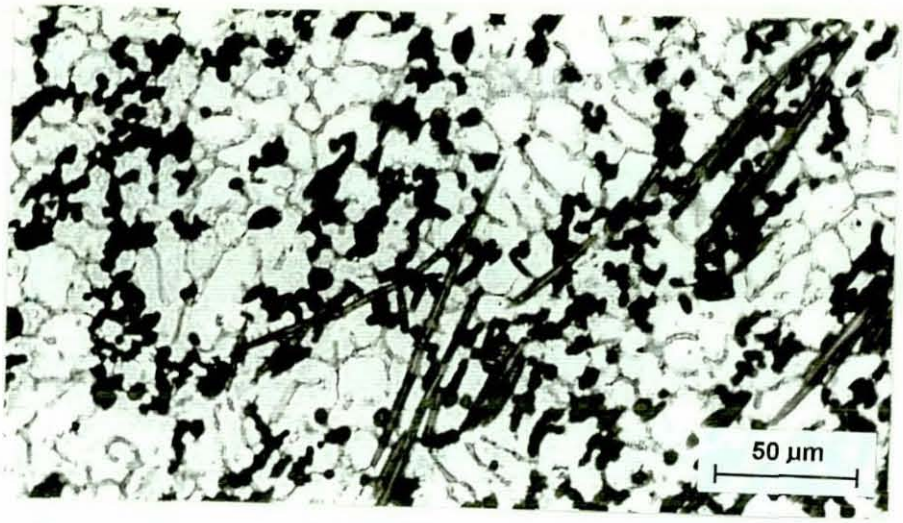


Figure 8-58 Optical microstructure of squeeze infiltrated RZ5DF-14% vol. fraction Saffil fibres produced under applied pressure of 60 MPa.

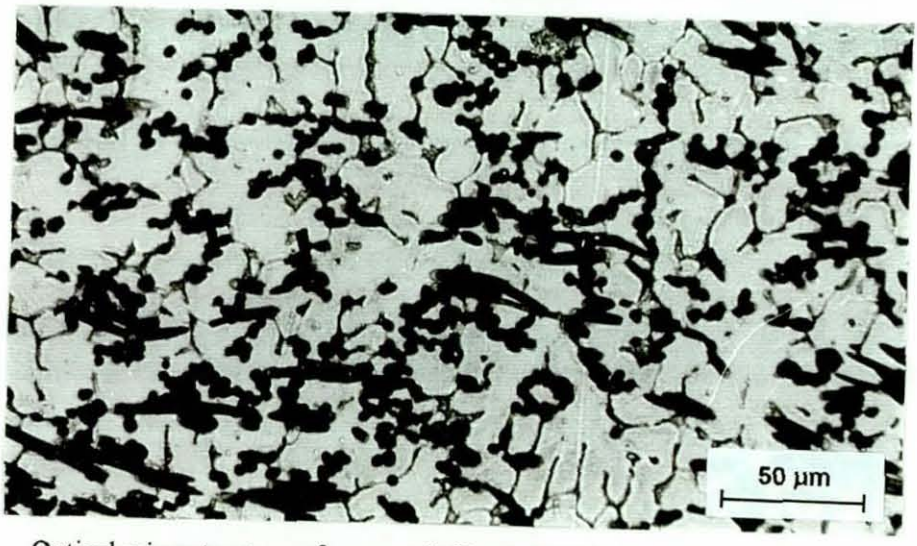


Figure 8-59 Optical microstructure of squeeze infiltrated RZ5DF-14% vol. fraction Saffil fibres produced under applied pressure of 80 MPa.

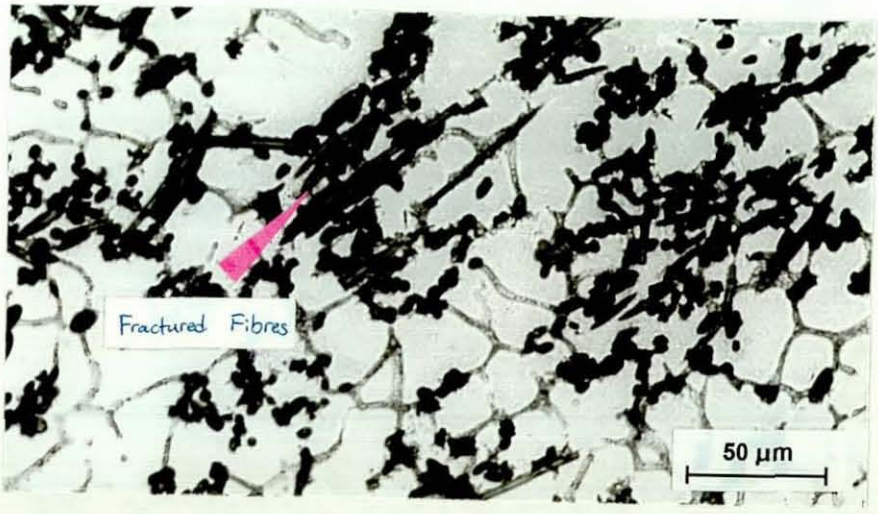


Figure 8-60 Optical microstructure of squeeze infiltrated RZ5DF-14% vol. fraction Saffil fibres produced under applied pressure of 100 MPa, showing signs of fracture and touching fibres.

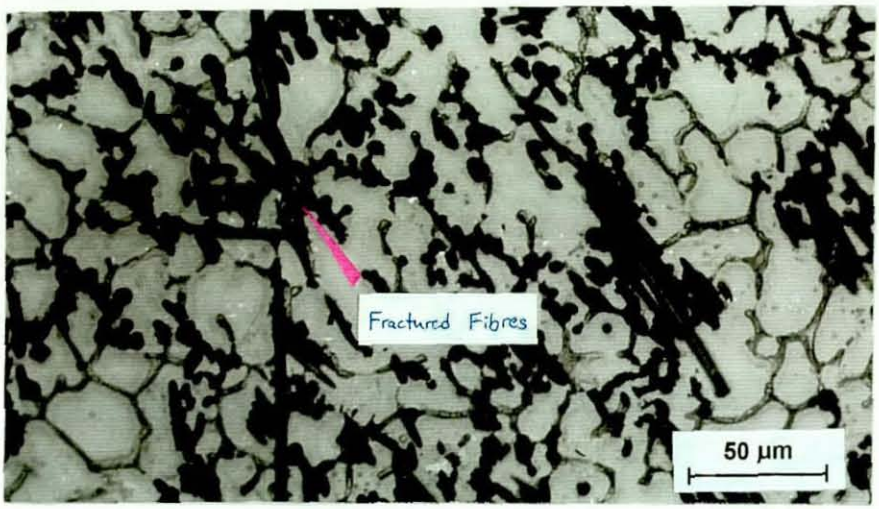


Figure 8-61 Optical microstructure of squeeze infiltrated RZ5DF-14% vol. fraction Saffil fibres produced under applied pressure of 120 MPa, showing signs of fracture and touching fibres.

The fracture surface examination of MMC samples produced at applied pressure levels of 100 MPa and 120 MPa showed signs of fibres that had been fractured, seen in figures 8-62 and 8-63 respectively. These fibre fractures were most probably caused by excessive infiltration pressure rather than by the breakage of the specimen due to the tensile load applied during the destructive tests. Microstructural examination along longitudinal and transverse directions of the cast ingot confirms that the fibres are indeed broken during infiltration. Examples of fractured fibres in the cast structure are shown in figures 8-60 and 8-61. An even distribution of fibres throughout the structure produced with 80 MPa applied pressure can be seen in figures 8-40 and 8-59.

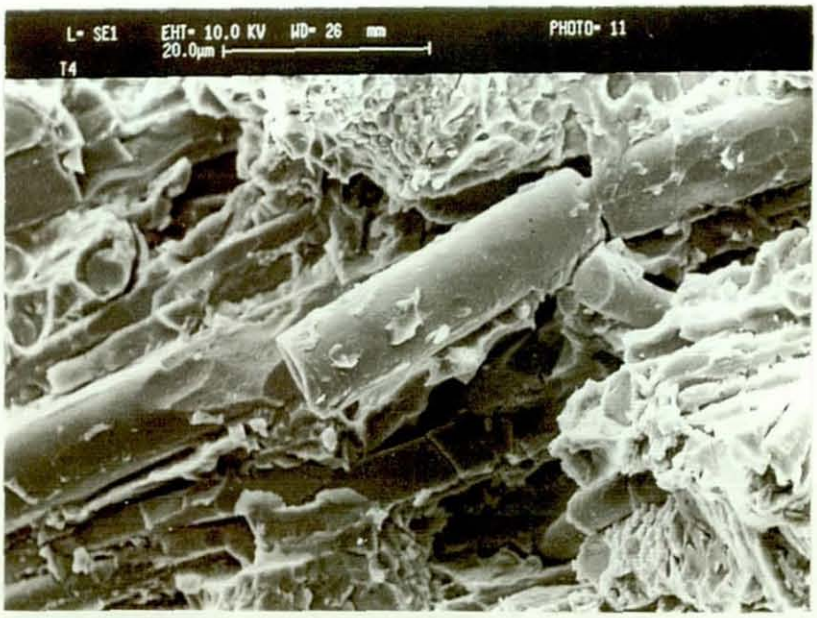


Figure 8-62 SEM micrograph of the fracture face of a squeeze infiltrated RZ5DF-14% vol. fraction Saffil fibres produced under applied pressure of 100 MPa.

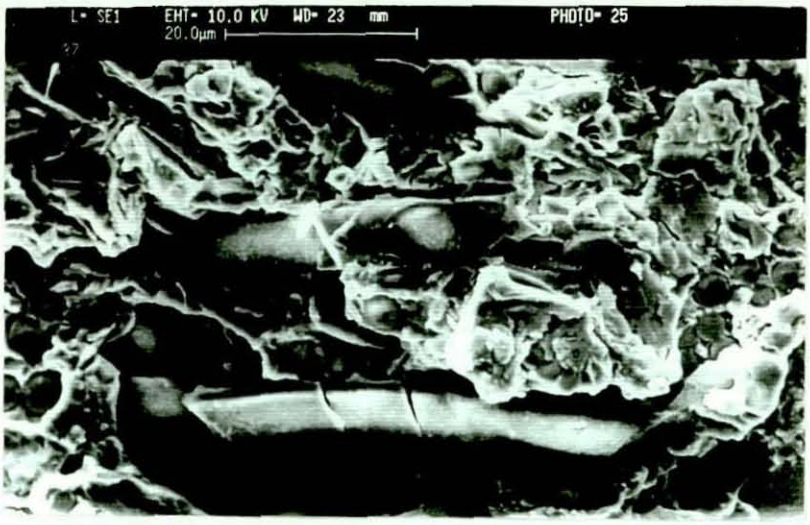


Figure 8-63 SEM micrograph of the fracture face of a squeeze infiltrated RZ5DF-14% vol. fraction Saffil fibres produced under applied pressure of 120 MPa.

8.6 The Evaluation of the Effects of Applied Pressure Duration

Several observations have been made based on the process settings listed in section 7.6.2.5 and these are highlighted along with the results in the following sections, which include a comparison of tensile strengths, material hardnesses and metallographic structures.

8.6.1 The Effects of Applied Pressure Duration on Tensile Properties

Based on the "rule of thumb", the maximum duration of pressure application should be 1 second per millimetre of section thickness [160]. The maximum casting section thickness used in the experiments was 16mm, hence the duration of applied pressure was 16 seconds. However the "rule of thumb" does not apply in this case, as a 'minimum' duration of applied pressure (25 seconds) exists for this particular alloy, as shown in figures 8-64 and 8-65. However, a duration of greater than 25 seconds of applied pressure does not result in any further improvement in tensile properties. Therefore, a duration of 25 seconds of applied pressure was seen to provide the best results, in terms of the maximum tensile properties and the optimum rate of production of the RZ5DF alloy. Figure 8-65 summarises the effects of applied pressure duration on the elongation and the % area reduction of the squeeze cast RZ5DF alloy.

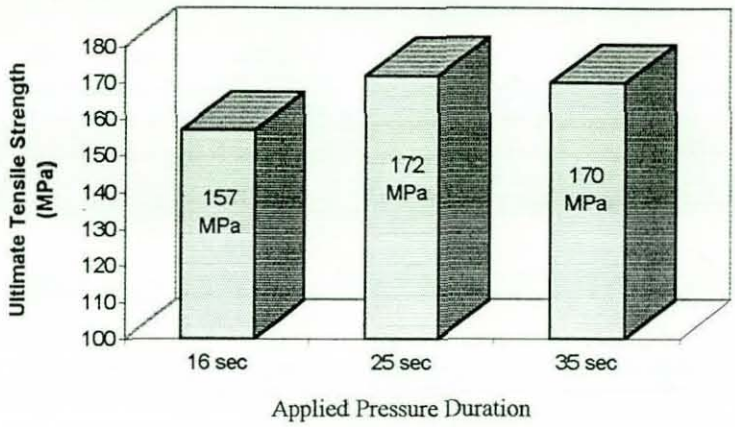


Figure 8-64 The effects of Applied Pressure Duration on the UTS values of RZ5DF alloy produced by squeeze casting. (Average results taken from three specimens).

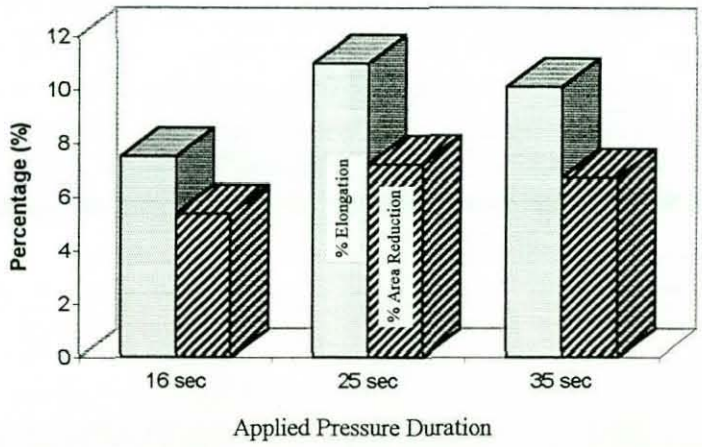


Figure 8-65 The effects of Applied Pressure Duration on the % elongation and % area reduction of RZ5DF alloy produced by squeeze casting. (Average results taken from three specimens).

The influence of applied pressure duration on squeeze infiltrated RZ5DF MMC, as with squeeze casting, shows that there exists a 'minimum' duration of 25 seconds, which is required to ensure that the casting is fully solidified. Figure 8-66 displays the effect of applied pressure duration on the UTS, and figure 8-67 summarises the elongation and area reduction properties.

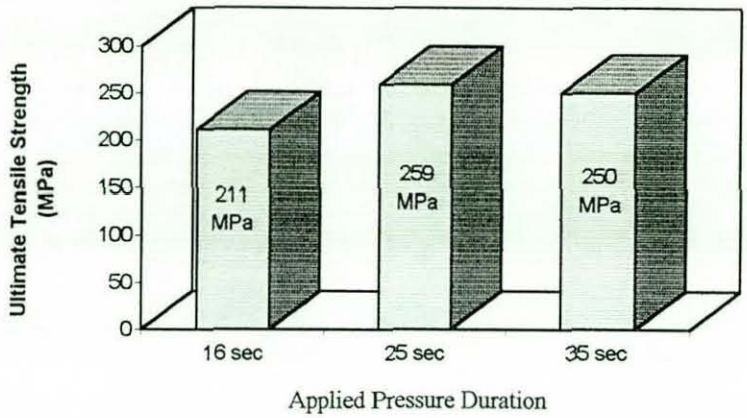


Figure 8-66 The effects of Applied Pressure Duration on the UTS values of RZ5 MMC produced by squeeze infiltration. (Average results taken from three specimens).

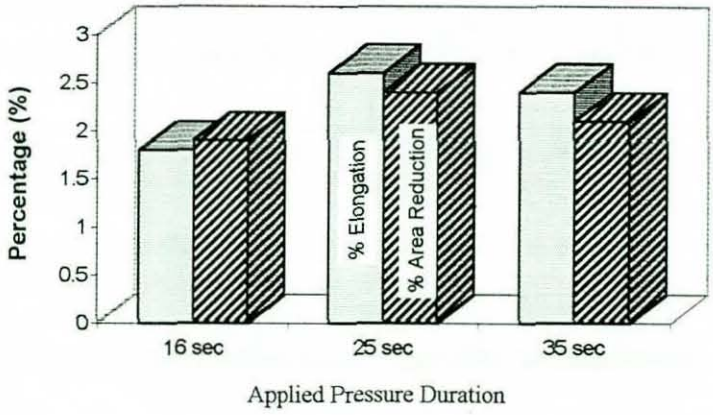


Figure 8-67 The effects of Applied Pressure Duration on the % elongation and % area reduction of RZ5DF MMC produced by squeeze infiltration. (Average results taken from three specimens).

Detailed tensile properties for RZ5DF alloy and its composite, produced with different applied pressure duration, are displayed in tables 5a and 5b of appendix B.

8.7 The Analysis of Alloy Distribution at Fibre-Matrix Interfaces and Grain Boundaries

SEM mapping analysis was conducted to evaluate the alloy distribution in the cast metal. The following sections present the results of the analysis of the cast microstructures of RZ5DF and RZ5 alloys and their composites.

8.7.1 SEM Zinc Mapping Analysis of the Grain Boundaries of the RZ5DF Alloy

Zinc mapping analysis was conducted to assess the distribution of zinc in the cast material. Figure 8-69 presents the zinc mapping analysis for the gravity die cast microstructure. The mapping shows the presence of a significant amount of zinc at the grain boundaries with a small amount of zinc within the

grains. The back scanner micrograph of the gravity die cast RZ5DF alloy and its zinc mapping analysis are presented in figures 8-68 and 8-69 respectively. The back scanner micrograph and zinc mapping analysis of the squeeze cast RZ5DF alloy are shown in figures 8-70 and 8-71 respectively. The zinc mapping indicates that a greater amount of zinc is present within the grains of the squeeze cast material compared to the gravity die cast material, seen in figure 8-71. To establish the degree of distribution of the alloying elements in the squeeze cast RZ5DF alloy, SEM analyses were conducted at both the grain centre and grain boundary. The locations where the SEM analyses were conducted are shown in figure 8-72. The analyses taken at the grain centre and grain boundary are presented in figures 8-73 and 8-74. Energy Dispersive X-Ray (EDX) analysis at the grain centre revealed the presence of a minimum amount of the alloying elements (i.e. zinc and rare earths elements) in the grain. Analysis at the grain boundary showed larger amounts of alloying elements (zinc, cerium and lanthanum).

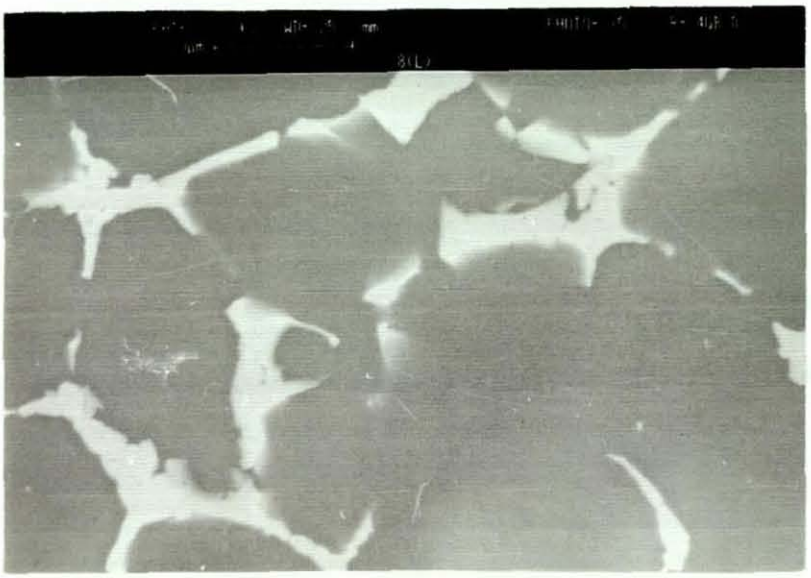


Figure 8-68 Back scanner micrograph of gravity die cast RZ5DF alloy

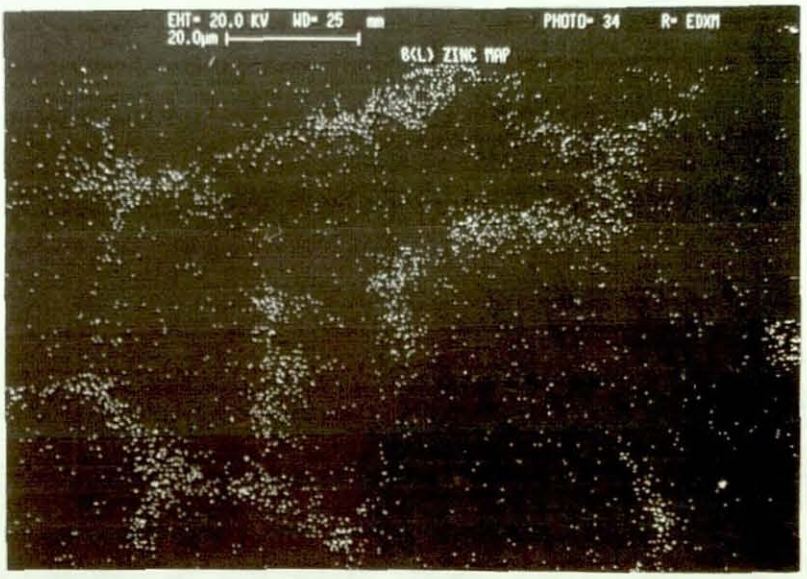


Figure 8-69 SEM zinc mapping on the microstructure of gravity die cast RZ5DF alloy

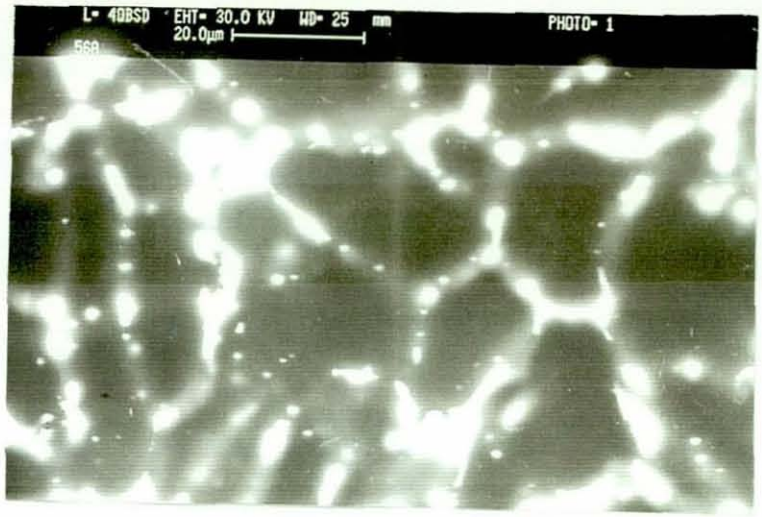


Figure 8-70 Back scanner micrograph of squeeze cast RZ5DF alloy

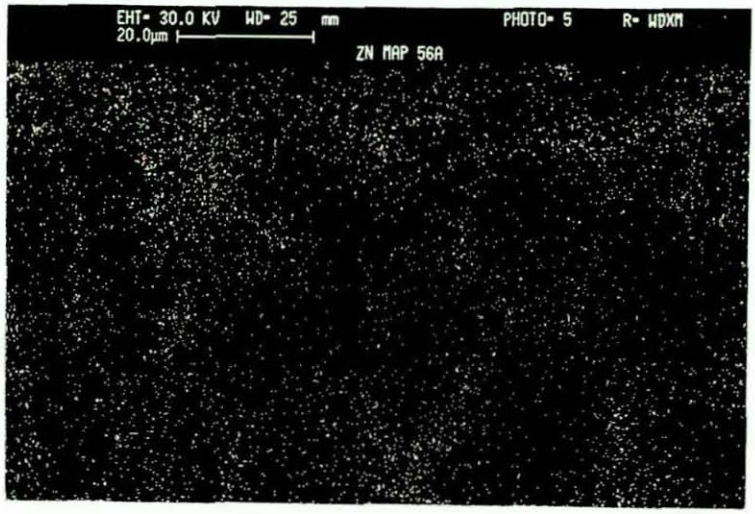


Figure 8-71 SEM zinc mapping on the microstructure of squeeze cast RZ5DF alloy

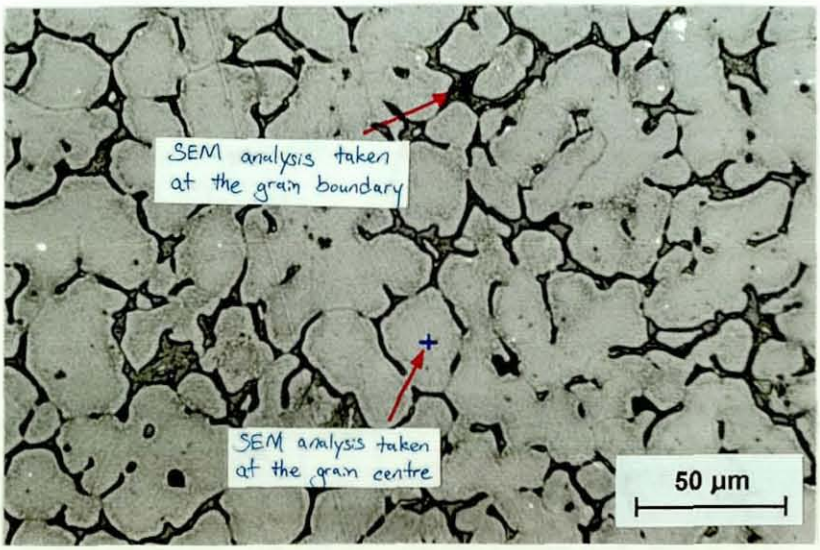


Figure 8-72 Microstructure of RZ5DF alloy, indicating the location where SEM analysis was taken

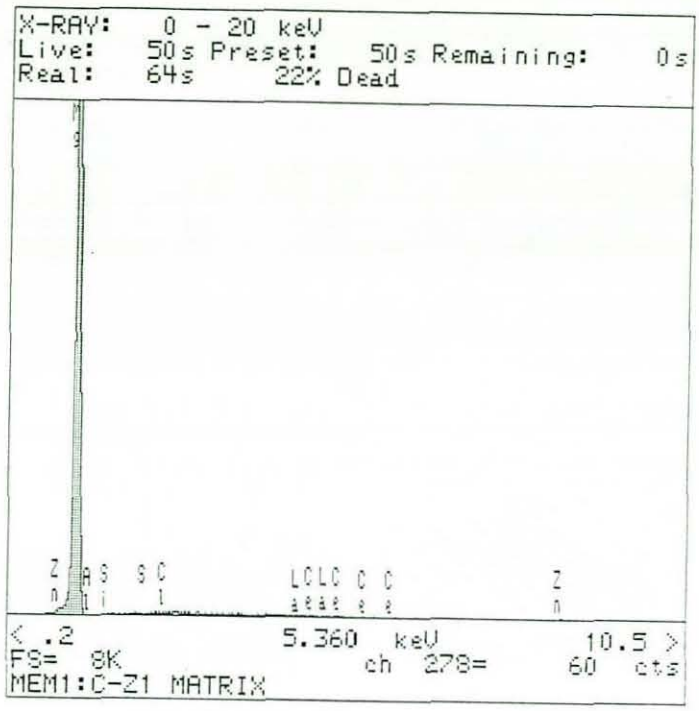


Figure 8-73 EDX analysis taken at the grain centre

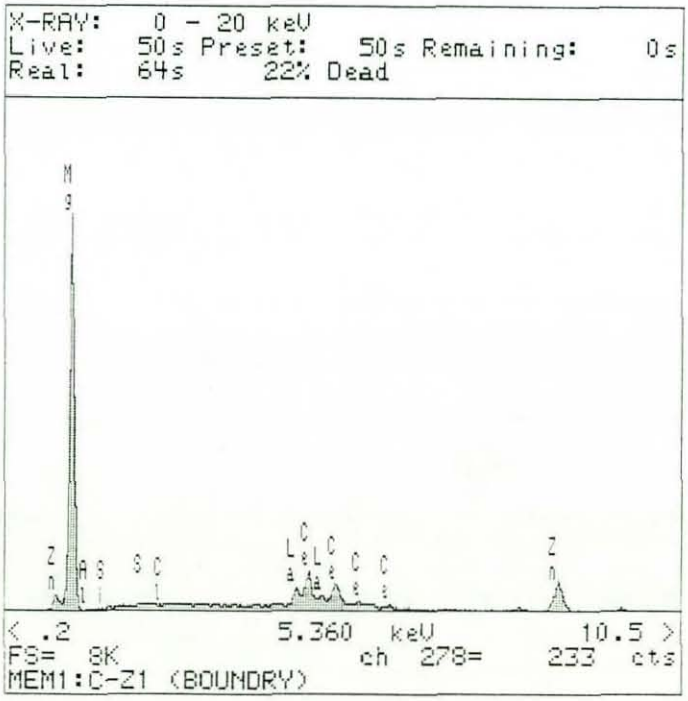


Figure 8-74 EDX analysis taken at the grain boundary

8.7.2 SEM Zinc and Zirconium Mapping Analyses of the Squeeze Cast RZ5 Alloy

Both zinc and zirconium mapping analyses were conducted to assess the distribution of these elements in the cast material. Figure 8-75 presents the microstructure of the squeeze cast RZ5 alloy. The

mapping analyses for zinc and zirconium are illustrated in figures 8-76 and 8-77 respectively. The zinc mapping again shows a greater amount of zinc at the grain boundary relative to the amount present within the grains. From figure 8-77, it may be seen that the distribution of zirconium is primarily within the grain, with very little or no sign of zirconium presence at the grain boundaries. This indicates that the zirconium remains in solid solution after solidification.

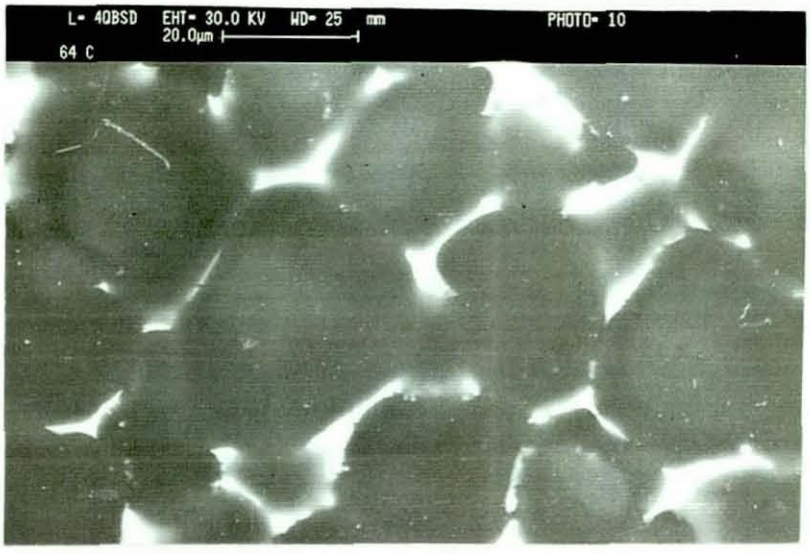


Figure 8-75 Back scanner micrograph of squeeze cast RZ5 alloy.

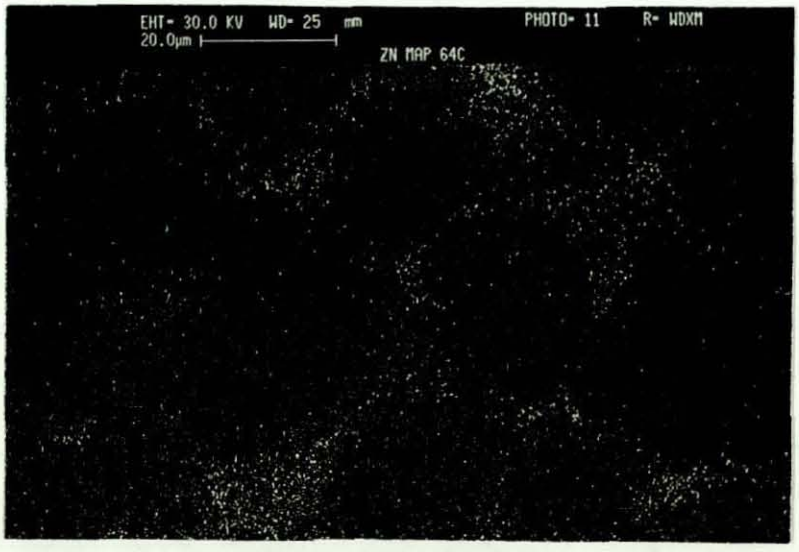


Figure 8-76 SEM zinc mapping on the microstructure of squeeze cast RZ5 alloy

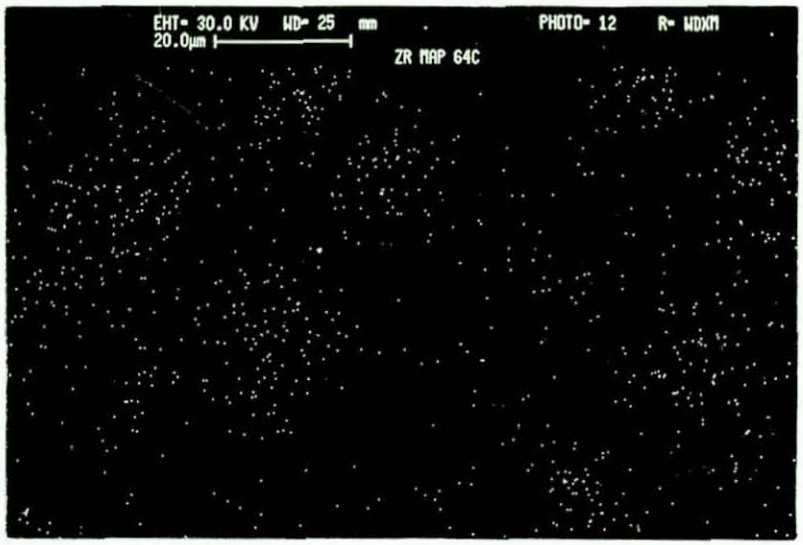


Figure 8-77 SEM zirconium mapping on the microstructure of squeeze cast RZ5 alloy.

8.7.3 SEM Zinc Mapping Analysis for the Fibre-matrix Interface of a RZ5DF-Saffil Fibre Composite

Figure 8-78 presents the zinc mapping analysis for the fibre-matrix interface of a RZ5DF-Saffil fibre composite, the back scanner micrograph is shown in figure 8-79. The mapping again shows that the majority of the zinc is present at the grain boundaries and around the fibres.

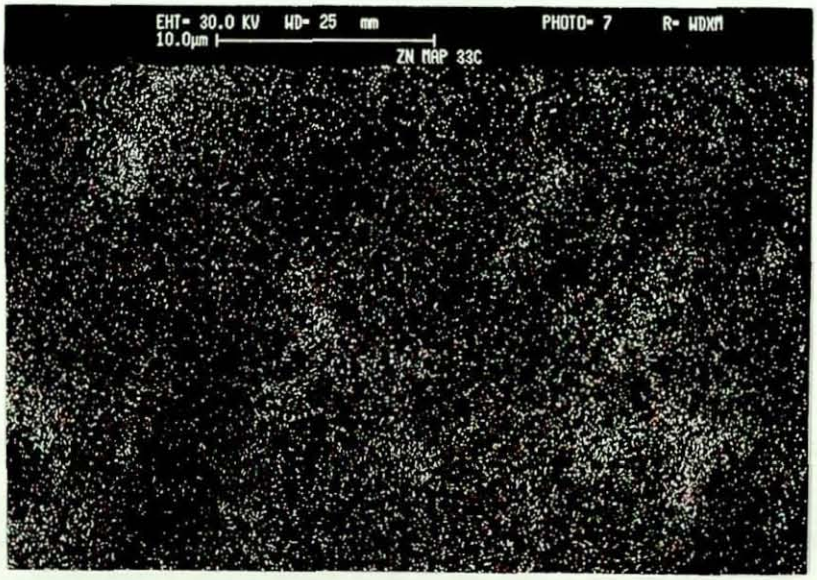


Figure 8-78 SEM zinc mapping on the fibre-matrix interface of RZ5DF-14% vol. Saffil composite

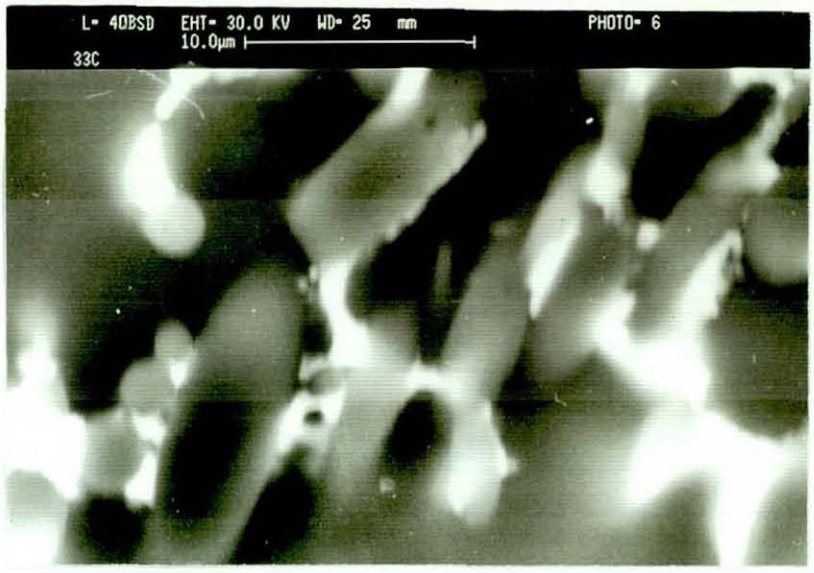


Figure 8-79 Back Scanner micrograph on the fibre-matrix interface of RZ5DF-14% vol. Saffil composite

8.8 The Consistency of Casting Properties in Longitudinal and Transverse directions

Several observations on the process settings listed in section 7.6.2.6 are highlighted together with results in the following sections, which include a comparison of tensile strengths and metallographic structures. A summary of hardness test results taken along the longitudinal and transverse directions for all the castings, conducted during the primary casting programme, is presented in section 8.8.2.

8.8.1 The Evaluation of Tensile Properties in Transverse Direction of the Casting

To be able to extract a higher number of tensile specimens (for effective usage per volume of casting) from a cast ingot without sacrificing experimental consistency, it was necessary to analyse the tensile strength of specimens along the transverse direction of the as-cast ingot. A schematic view showing the approximate location of the tensile specimens in the casting is shown in figure 7-12. Analysis of the tensile properties of the specimens extracted from the casting have been conducted to evaluate the consistency along the transverse direction of the casting ingot. Figure 8-80 shows the transverse tensile properties of RZ5DF alloy produced by (i) squeeze casting and squeeze infiltration with (ii) 14% and (iii) 20% vol. fraction Saffil fibres. The tensile properties along the transverse direction show consistency. Detailed tensile properties of squeeze cast RZ5DF alloy and its composite, produced to evaluate the process consistency, are presented in table 6 of appendix B.

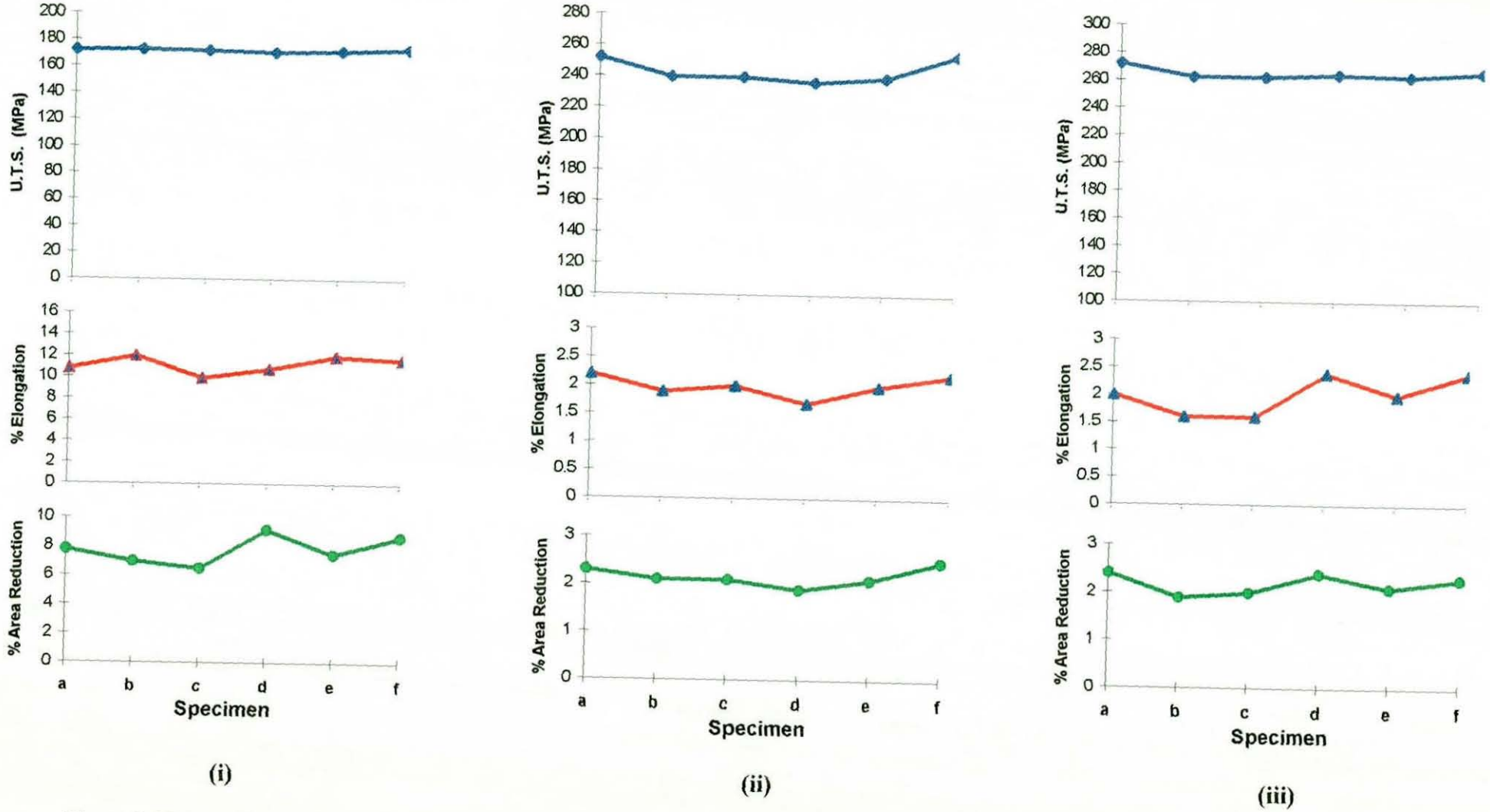


Figure 8-80 Transverse tensile properties of RZ5DF alloy (i) squeeze cast; and squeeze infiltrated with (ii) 14% and (iii) 20% volume fraction Saffil fibres

The ranges in tensile properties for the six specimens from the squeeze cast RZ5DF alloy are: 171 to 174 MPa for UTS, 9.9 to 12% for % elongation and 6.5 to 9.2% for % area reduction. The ranges in tensile properties for the six squeeze infiltrated RZ5DF alloy specimens incorporating a 14% vol. fraction Saffil fibre preform are: 237 to 255 MPa for UTS, 1.7 to 2.2% for % elongation and 1.9 to 2.5% for % area reduction. The ranges for the 20% vol. fraction Saffil fibre preform are: 263 to 272 MPa for UTS, 1.6 to 2.4% for % elongation and 1.9 to 2.4% for % area reduction. The variations in % elongation and % area reduction for MMC specimens are within ± 0.4% and are too small to suggest correlation with UTS. Therefore, for this reason, only the variation between the UTS values will be discussed.

The results presented in figure 8-80 (i) show the UTS values to be very consistent. However, the UTS for the squeeze infiltrated RZ5DF Saffil composite is higher for specimens from the two ends of the cast ingot, as shown in figure 8-80 (ii) and (iii). This increase is due to the presence of a higher fibre content in those regions, which results from minor preform deformation near the die wall. Similar preform deformation was reported in section 8.4.3.1 and figure 8-41.

The results also indicate that the variation in UTS tends to decrease with increased fibre volume fraction. This is because the higher fibre content preforms are more resistant to deformation. Since the UTS values for all three experimental materials and/or composite systems show minimal variation along the transverse direction, the results are consistent enough to support specimen extraction along the transverse direction for the secondary casting programme.

8.8.2 The Summary of Hardness Values Obtained during the Primary Casting Programme

Hardness values reported in sections 8.2 to 8.6 are summarised for the longitudinal and transverse direction in figures 8-81 and 8-82 respectively. The summaries show two distinct zones, those for the alloy and those for the fibre reinforced material. Those with fibre reinforcement show significantly higher hardness, the increases are in the range of 280% to 600% relative to the alloy system.

Specimens A (cast with 12% vol. Saffil + 9% vol. carbon preform system) and B (cast with 20% vol. Saffil with alumina binder) have the highest hardness values. This increase in hardness was due to the presence of higher fibre volume within the preform which was used to produce specimen A. For specimen B, the increased hardness was due to the presence of a higher fibre volume within the reinforced region, as a result of preform deformation during the squeeze infiltration process, as seen in figure 8-19. Conversely, specimens C and D (cast with applied pressure of 0.1 MPa and 20 MPa respectively) have lower hardness values relative to the other fibre reinforced composites. The low hardness was due to the selection of inappropriate process parameters. The importance of process parameters for the squeeze infiltration of magnesium matrix composite was discussed in section 5.6.

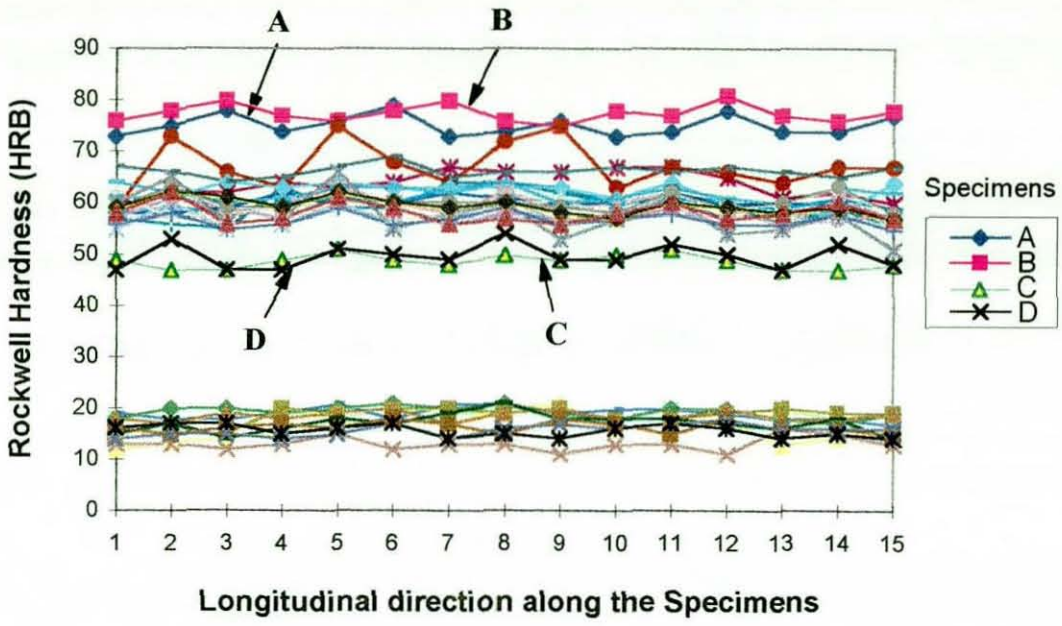


Figure 8-81 Summary of material hardness along the longitudinal direction of squeeze cast RZ5DF alloy and squeeze infiltrated composites with different preform systems.

The hardness values from the transverse direction (figure 8-82) shows a similar pattern to those for the longitudinal values.

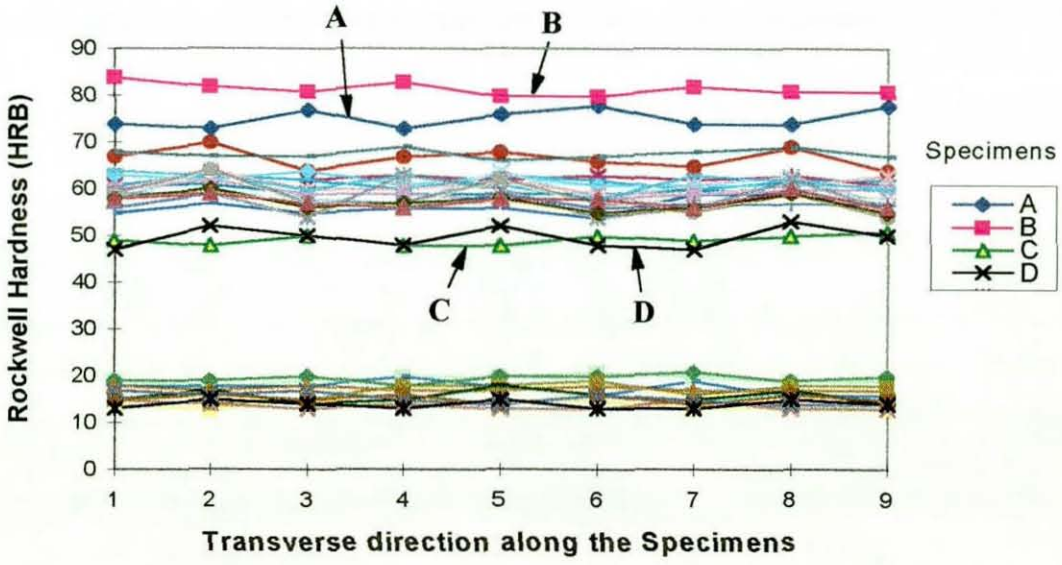


Figure 8-82 Summary of material hardness along the transverse direction of squeeze cast RZ5DF alloy and squeeze infiltrated composites with different preform systems.

8.8.3 Microstructural Evaluation

Microstructural examinations were conducted on the cast structures to study the solidification structures of the squeeze cast components. The effect of solidification is studied by comparing the

grain size throughout the cast structure. The locations of specimens taken for microstructure studies are shown in figure 8-83. The microstructure of the squeeze cast RZ5DF alloy indicates uniform grain structure throughout the cast specimen, illustrated in figure 8-84. This shows that the applied pressure of the squeeze casting process has caused rapid solidification and, in turn, produced a uniform equiaxe cast structure.

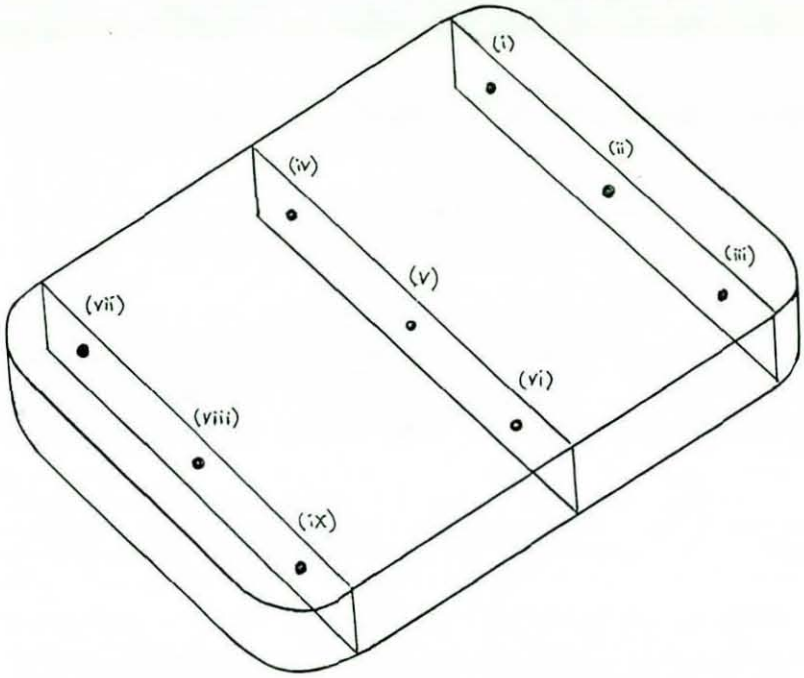
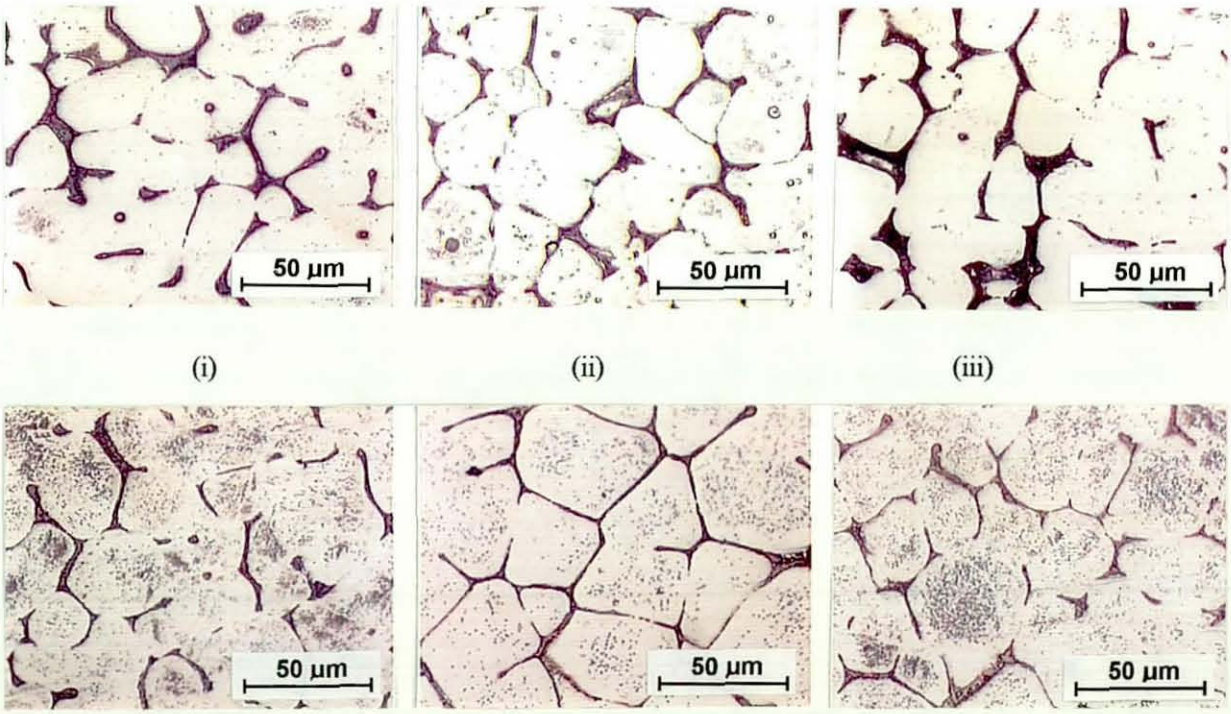


Figure 8-83 Locations of microstructure studies on the cast ingot. The numbers indicate the location where the studies were conducted.



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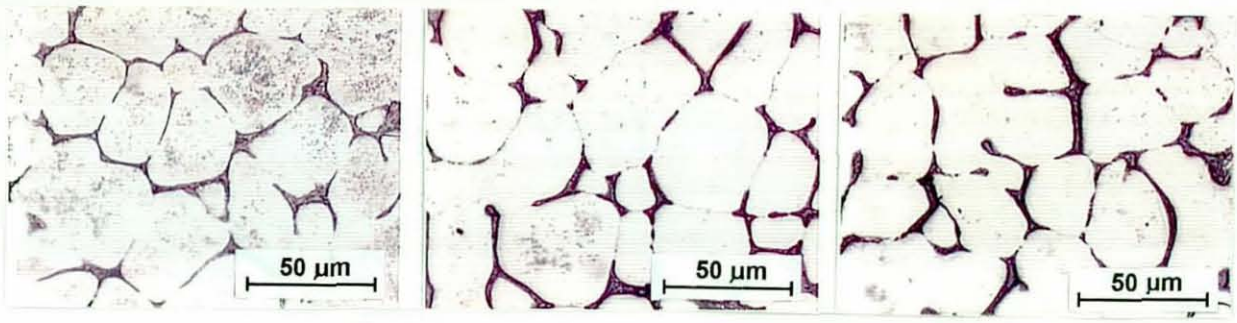
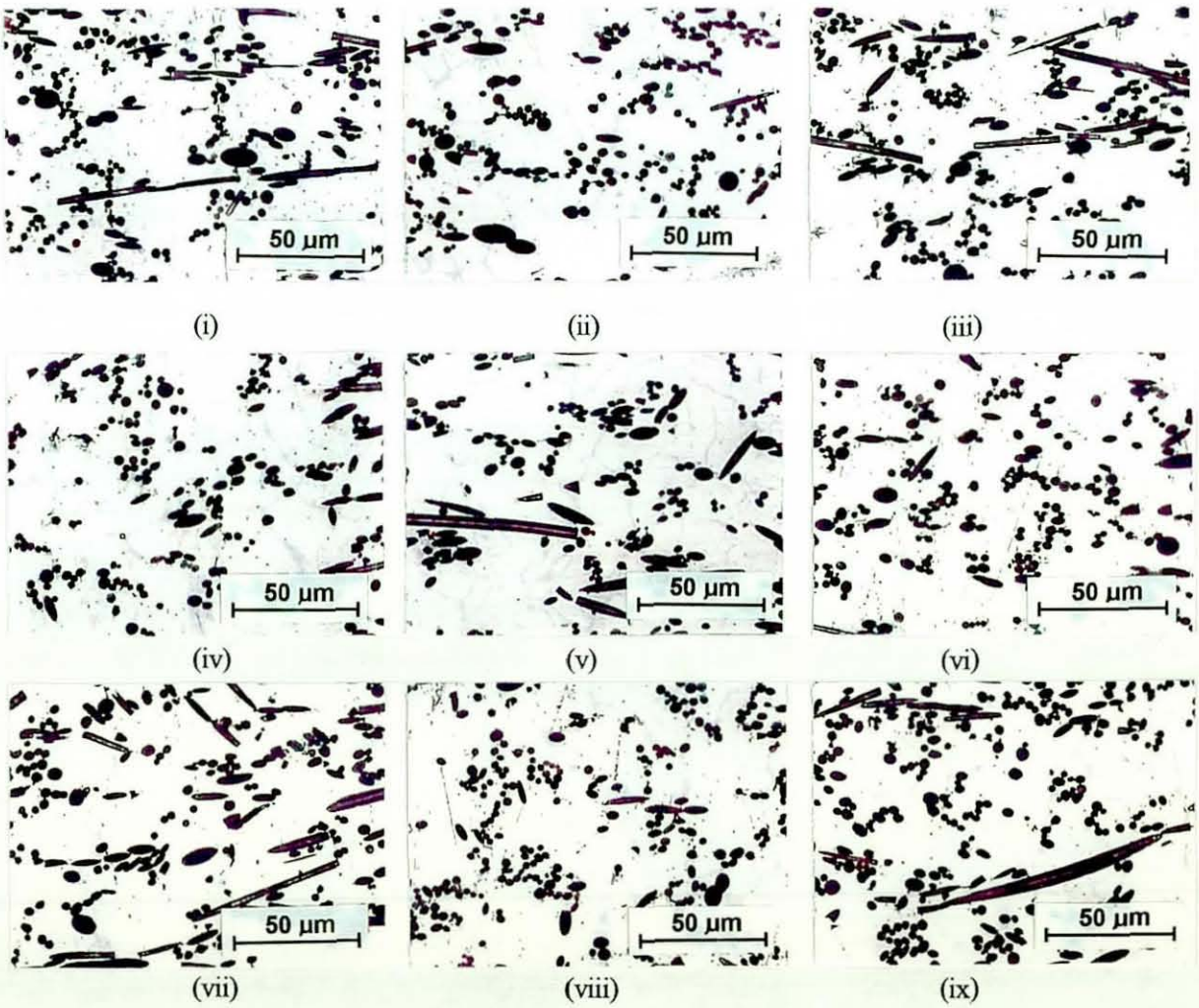


(vii) (viii) (ix)

Figure 8-84 Microstructure of the squeeze cast RZ5DF alloy. Refer to figure 8-83 for each micrograph location on the cast ingot.

The microstructures of the squeeze infiltrated RZ5DF-14% vol. Saffil MMC, presented in figure 8-85, show the fibre distribution throughout the casting to be relatively uniform. This illustrates the advantage of using the fibre preform to control the distribution of the fibre within the composite.



(i) (ii) (iii)

(iv) (v) (vi)

(vii) (viii) (ix)

Figure 8-85 Microstructure of the squeeze infiltrated RZ5DF-14% vol. Saffil MMC. Refer to figure 8-83 for each micrograph location on the cast ingot.

Both microstructural studies on the cast structure show the success of the squeeze casting process in attaining uniform grain structure and fibre distribution (with the aid of a preform). Uniform heating of the die also plays a part in attaining a uniform grain structure throughout the cast specimens.

8.9 Summary

In the primary casting programme, the process parameters and preform system necessary to achieve optimum mechanical properties were established for the RZ5DF alloy and its composite. The following preform system and process variables were now set at a constant level for the secondary casting programme (chapter 9) to investigate the effect of pouring and die temperature on the cast properties.

From the evaluation of suitable preforms for RZ5DF magnesium alloy, 14% vol. Saffil with 5% silica binder preform provides the most promising results, in terms of ease of production and maximum strength. From the evaluation of process variables, it was found that the least effort to achieve maximum performance for squeeze cast RZ5DF alloy was realised with an applied pressure of 60 MPa and at 25 seconds of pressure application. As for the process variables for casting MMC, the highest mechanical properties were achieved with an applied pressure of 80 MPa, a preform temperature of 600°C and squeeze infiltration duration of 25 seconds.

Chapter 9

Results and Observations from the Secondary Experimental Stage

9.1 Introduction

This chapter describes the results obtained from the secondary casting programme. The effects of pouring and die temperature were investigated on the RZ5DF alloy, the RZ5DF reinforced with 14% vol. Saffil fibres, RZ5 alloy and RZ5 reinforced with 14% vol. Saffil fibres. The influence of pouring and die temperatures on the ambient and elevated cast properties are reported in detail. The microstructural examinations of test specimens produced at the various process settings are described. Finally, the results from the evaluation of heat treatment on the alloys and composites are presented.

9.2 The Effect of Pouring and Die Temperatures on the Mechanical Properties and Microstructures of the RZ5DF Alloy

This section describes the observations made on the effects of pouring and die temperature on the mechanical properties and grain size for castings produced with the RZ5DF alloy. The effect of pouring and die temperatures on values of UTS are graphically presented, where individual points on each graph show the average value taken from three specimens.

9.2.1 The Mechanical Properties and Microstructures of the RZ5DF Alloy Tested at Ambient Temperature

The following sections will illustrate the effects of pouring and die temperature on the ambient temperature properties and the microstructures of the RZ5DF alloy. Detailed results of the mechanical properties of squeeze cast RZ5DF alloy specimens, tested at ambient temperature, are displayed in table 1 of appendix C.

9.2.1.1 The Mechanical Properties of the RZ5DF Alloy Tested at Ambient Temperature

The effects of pouring temperature on the values of UTS at different die temperatures are shown in figure 9-1. The graph indicates that, amongst the nine pouring and die temperature combinations, the combination of 780°C pouring temperature and 250°C die temperature produced the highest value for UTS. The figure also shows that a lower but consistent level of UTS was achieved at the die temperature of 275°C.

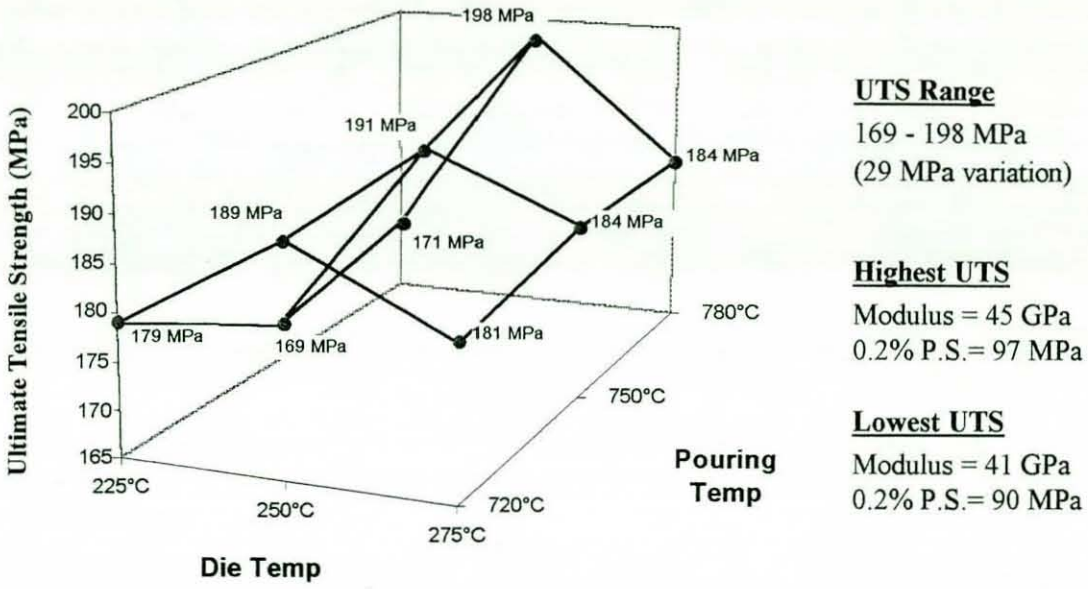


Figure 9-1 The effects of pouring and die temperature on ambient temperature UTS (RZ5DF alloy)

In essence, the overall effects of pouring and die temperatures were UTS values (tested at ambient temperature) with a range of 29 MPa, i.e. a lowest UTS of 169 MPa and a highest UTS of 198 MPa. At the highest value of 198 MPa, the value for Young's modulus was 45 GPa and the 0.2% proof stress was 97 MPa. The lowest UTS values were obtained at a lower die temperature of 225°C, while the mid and highest UTS values were achieved at the die temperature of 275°C and 250°C respectively.

The effects of pouring temperature on % elongation and % area reduction at different die temperatures are shown in table 9-1. The % elongation, for the nine combinations of temperatures, ranges from 8.3 to 12.8%, and the % area reduction ranges from 5 to 7.7%. The results show that the combination of 780°C pouring temperature and 250°C die temperature produced the highest % elongation and the highest % area reduction of 12.8% and 7.7% respectively. At this combination of pouring and die temperatures, the UTS value was also at its highest value of 198 MPa. The results also show that the highest die temperature of 275°C produced a more consistent value of elongation and area reduction.

	225°C		250°C		275°C	
	% Elongation	% Area Reduction	% Elongation	% Area Reduction	% Elongation	% Area Reduction
720°C	10.4%	6.3%	12%	7%	9.3%	6.1%
750°C	8.3%	5%	12%	7.3%	10%	6.4%
780°C	10%	6.2%	12.8%	7.7%	9.7%	5.8%

Table 9-1 The effects of pouring and die temperature on % elongation and % area reduction of RZ5DF alloy specimens tested at ambient temperature

9.2.1.2 The Microstructural Examination of the RZ5DF Alloy Tested at Ambient Temperature

The metallographic examinations were conducted to evaluate the effects of various process parameters (pouring and die temperature) on the squeeze cast RZ5DF microstructure. Microstructural examinations were carried out on specimens selected from those which had the highest, intermediate and lowest UTS values. The combinations were: (i) pouring temperature of 780°C and die temperature of 250°C for the highest UTS value (figure 9-2), (ii) pouring temperature of 720°C and die temperature of 275°C for the intermediate UTS value (figure 9-3), and (iii) pouring temperature of 750°C and die temperature of 225°C for the lowest UTS value (figure 9-4). The results of the microstructural examinations are presented below.

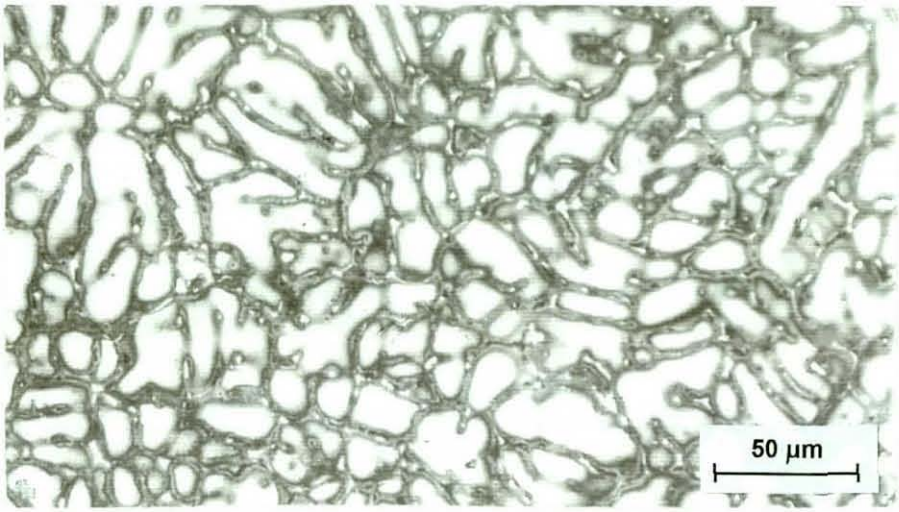


Figure 9-2 Optical microstructure of the squeeze cast RZ5DF alloy with the highest UTS of 198 MPa at ambient temperature, providing an average grain size of 18µm.

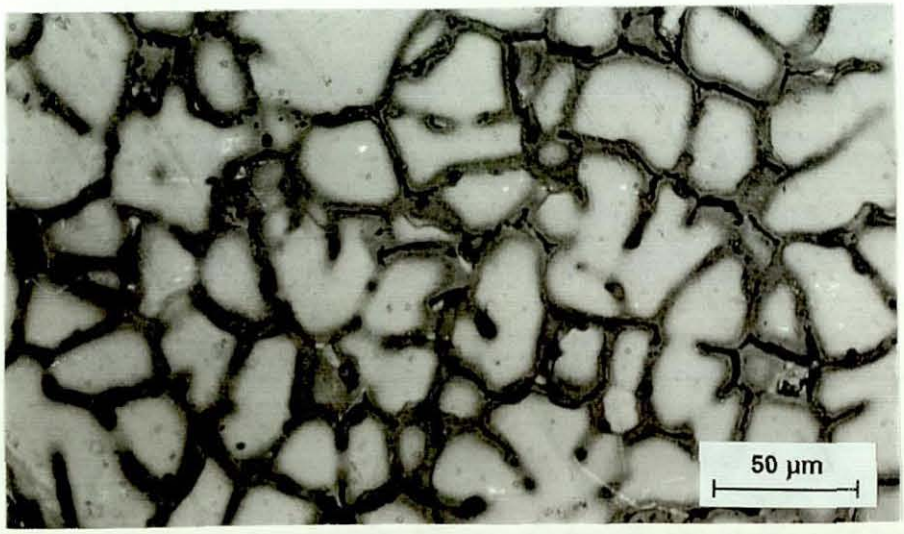


Figure 9-3 Optical microstructure of the squeeze cast RZ5DF alloy with an intermediate UTS of 181 MPa at ambient temperature, providing an average grain size of 28µm.

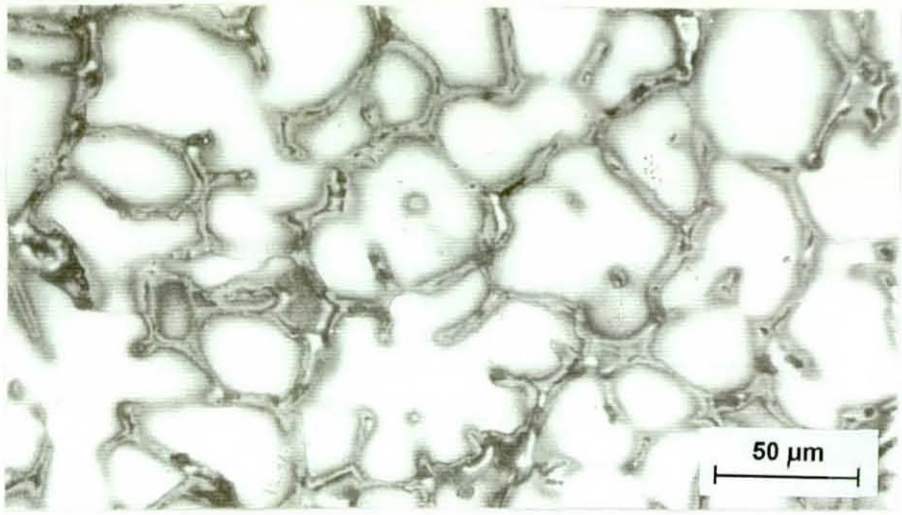


Figure 9-4 Optical microstructure of the squeeze cast RZ5DF alloy with the lowest UTS of 169 MPa at ambient temperature, providing an average grain size of 32µm.

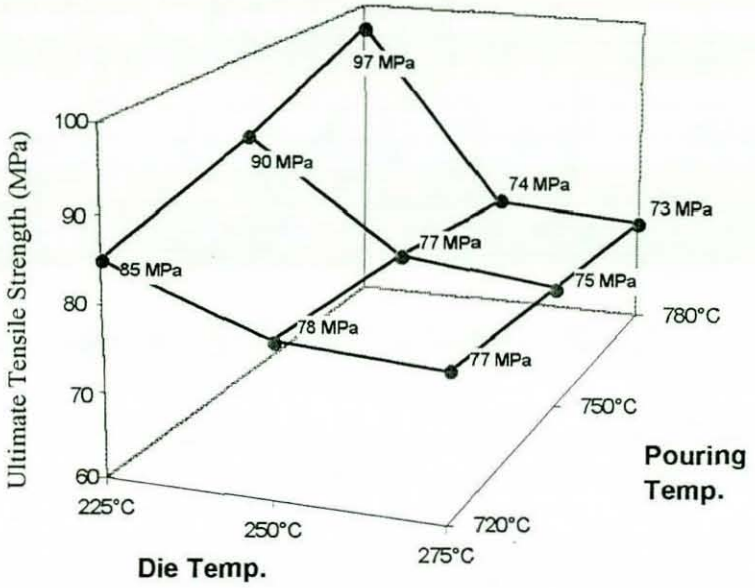
Figure 9-2 shows the smallest grain size produced by a pouring temperature of 780°C and a die temperature of 250°C. Figure 9-4 shows the largest grain size produced by a pouring temperature of 750°C and a die temperature of 225°C. In general, it can be seen from the UTS values associated with the figures that the smaller grain size results in greater strength for the RZ5DF alloy.

9.2.2 The Mechanical Properties and Microstructures of the RZ5DF Alloy Tested at 250°C

The following sections will illustrate the effects of pouring and die temperatures on the elevated temperature properties and microstructures of the RZ5DF alloy. Detailed results of the mechanical properties of squeeze cast RZ5DF alloy specimens, tested at elevated temperature, are displayed in table 2 of appendix C.

9.2.2.1 The Mechanical Properties of the RZ5DF Alloy Tested at 250°C

The effects of pouring and die temperature on values of UTS at an elevated temperature of 250°C are shown in figure 9-5. The chart indicates that, amongst the nine temperature combinations, that of 780°C pouring temperature and 225°C die temperature produced the highest UTS. The results also indicate that the lowest die temperature of 225°C produced the highest UTS values at the elevated temperature.



Range
73 - 97 MPa
(24 MPa variation)

Highest UTS
Modulus = 33 GPa
0.2% P.S. = 55 MPa

Lowest UTS
Modulus = 27 GPa
0.2% P.S. = 63 MPa

Figure 9-5 The effects of pouring and die temperature on the UTS at 250°C (RZ5DF alloy)

In essence, the overall effects of pouring and die temperature were UTS values (tested at 250°C) with a range of 24 MPa, i.e. a lowest UTS of 73 MPa and a highest UTS of 97 MPa. At the highest value of 97 MPa, the value for Young's modulus was 33 GPa and the 0.2% proof stress was 55 MPa.

The effects of pouring temperature on the % elongation and the % area reduction at different levels of die temperature are shown in table 9-2. The % elongation, for the nine combinations of pouring and die temperatures, ranges from values of 36 to 50%, and the % area reduction ranges from values of 36 to 65%. The results indicate a reversed pattern between the relationship of % elongation and % area reduction to UTS at an elevated temperature, where a lowest value of elongation of 36% and a lowest value of area reduction of 36% was associated with the highest value for UTS. The results, similar to those at ambient temperature, show that the highest die temperature of 275°C produced more consistent values for elongation and area reduction.

	225°C		250°C		275°C	
	% Elongation	% Area Reduction	% Elongation	% Area Reduction	% Elongation	% Area Reduction
720°C	50%	59%	47%	62%	50%	58%
750°C	47%	53%	47%	60%	49%	62%
780°C	36%	36%	46%	65%	48%	65%

Table 9-2 The effects of pouring and die temperature on the % elongation and the % area reduction of RZ5DF alloy specimens tested at 250°C.

9.2.2.2 The Microstructural Examination of the RZ5DF Alloy Tested at 250°C

The results presented in figure 9-5, indicate that die temperature had a significant influence on the values for UTS at an elevated temperature, hence microstructural examinations were carried out for three different die temperature specimens. Three specimens were selected for microstructural examination, these were based on the contribution of three different die temperatures (225°C, 250°C and 275°C) cast with a constant pouring temperature of 750°C. The combinations were: (i) pouring temperature of 750°C and die temperature of 225°C for the high UTS value (figure 9-6), (ii) pouring temperature of 750°C and die temperature of 250°C for the intermediate UTS value (figure 9-7), and (iii) pouring temperature of 750°C and die temperature of 275°C for the low UTS value (figure 9-8). The results of the microstructural examinations are presented below.

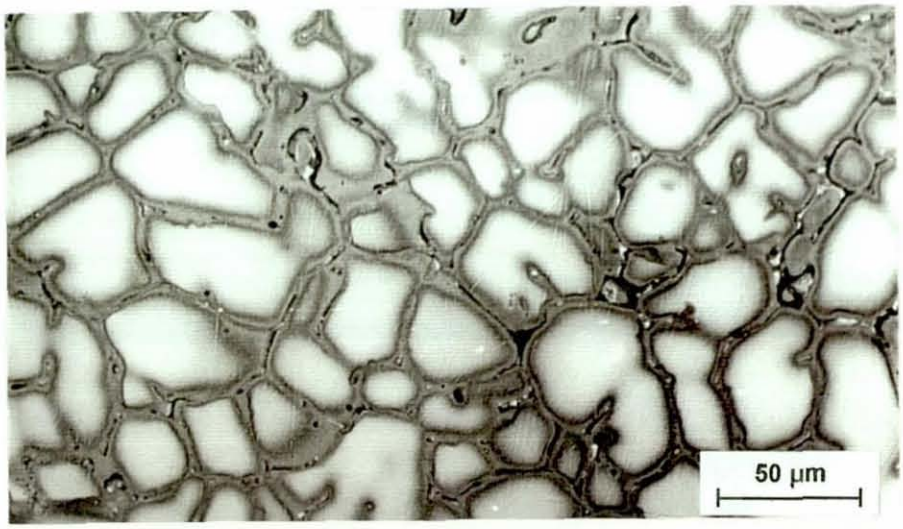


Figure 9-6 Optical microstructure of the squeeze cast RZ5DF alloy with the highest UTS of 90 MPa at an elevated temperature, providing an average grain size of 27µm.

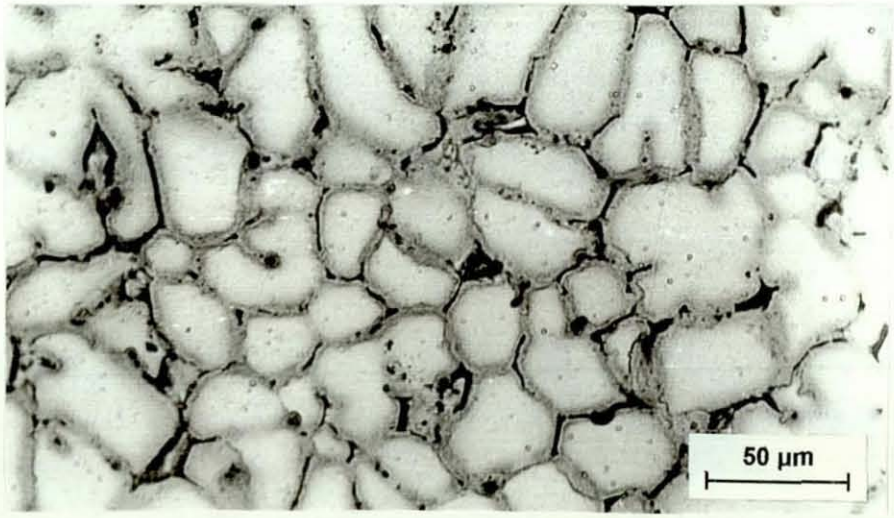


Figure 9-7 Optical microstructure of the squeeze cast RZ5DF alloy with an intermediate UTS of 77 MPa at an elevated temperature, providing an average grain size of 27µm.

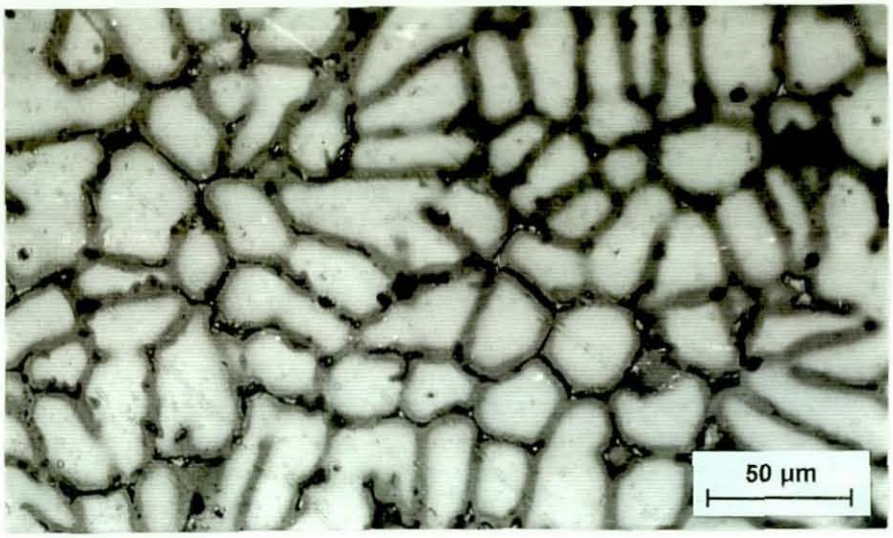


Figure 9-8 Optical microstructure of the squeeze cast RZ5DF alloy with the lowest UTS of 75 MPa at an elevated temperature, providing an average grain size of 25µm.

It can be seen, from figures 9-6 to 9-8, that the grain size ranges from 25 to 27 µm following exposure to elevated temperatures. However, a comparison of the UTS values associated with the figures reveals that the smaller grain size resulted in a reduction in strength for the RZ5DF alloy at elevated temperatures, which was the opposite effect to the relationship between strength and grain size at ambient temperatures.

9.3 The Effect of Pouring and Die Temperatures on the Mechanical Properties and Microstructures of the RZ5DF-14% vol. Saffil MMC

This section describes the observations made on the influence of pouring and die temperature on the mechanical properties and grain size for castings produced with the RZ5DF-14% vol. Saffil MMC. The influences of pouring and die temperatures on UTS are graphically presented. Individual points on each graph show the average value taken from three specimens.

9.3.1 The Mechanical Properties and Microstructures of the RZ5DF-14% vol. Saffil MMC Tested at Ambient Temperature

The following sections will illustrate the effects of pouring and die temperatures on the ambient temperature properties and the microstructures of the RZ5DF-14% vol. Saffil MMC. Detailed results of the mechanical properties of squeeze infiltrated RZ5DF-14% vol. Saffil MMC specimens, tested at ambient temperature, are presented in table 3 of appendix C.

9.3.1.1 The Mechanical Properties of the RZ5DF-14% vol. Saffil MMC Tested at Ambient Temperature

The influence of pouring temperature on UTS at different die temperatures is presented graphically in figure 9-9, indicating that, amongst the nine temperature combinations, that of 780°C pouring temperature and 275°C die temperature produced the highest value for UTS. The figure also shows that a lower level of UTS results was achieved at the die temperature of 225°C.

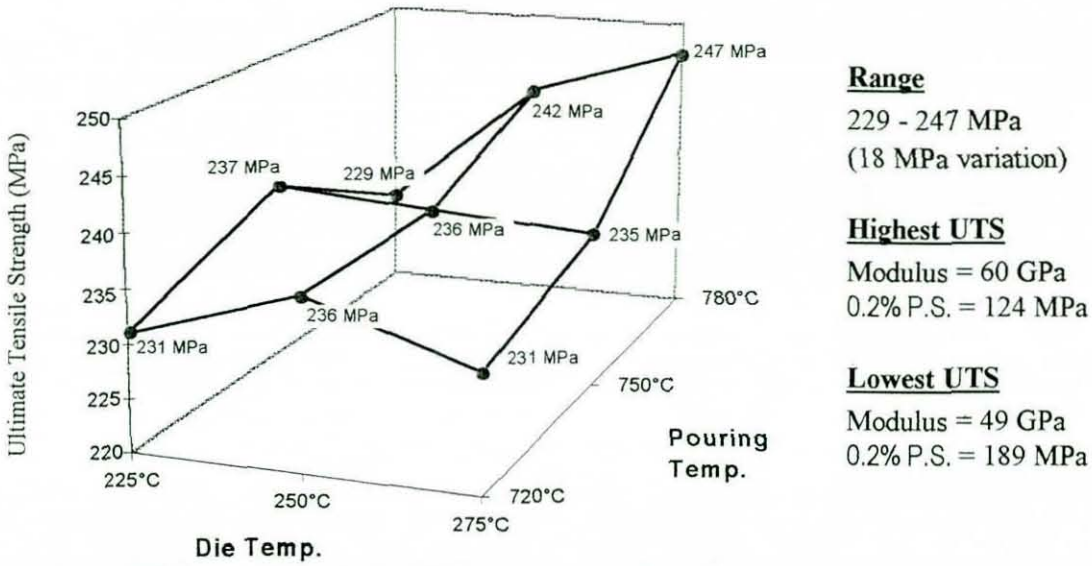


Figure 9-9 The effects of pouring and die temperature on the UTS at ambient temperature (RZ5DF-14% vol. Saffil MMC)

In essence, the overall effects of pouring and die temperature were UTS values (tested at ambient temperature) with a range of 18 MPa, i.e. a lowest UTS of 229 MPa and a highest UTS of 247 MPa. At the highest value of 247 MPa, the value for Young's modulus was 60 GPa and the 0.2% proof stress was 124 MPa.

The influence of pouring temperature on % elongation and % area reduction at different levels of die temperature are shown in table 9-3.

	225°C		250°C		275°C	
	% Elongation	% Area Reduction	% Elongation	% Area Reduction	% Elongation	% Area Reduction
720°C	2%	2.2%	2.2%	2.2%	2.3%	2.3%
750°C	2.1%	2.2%	1.7%	1.8%	2.1%	2%
780°C	1.6%	1.9%	1.5%	1.7%	2.5%	2.6%

Table 9-3 The influence of pouring and die temperature on % elongation and % area reduction of RZ5DF-14% vol. Saffil MMC specimens tested at ambient temperature.

The results from table 9-3, show that % elongation ranges from 1.5 to 2.5%, and that % area reduction ranges from 1.7 to 2.6%. The results also show that the combination of 780°C pouring temperature and 275°C die temperature produced the highest % elongation and the highest % area reduction of 2.5% and 2.6% respectively. At this combination of pouring and die temperatures, the UTS value was also at its highest value of 247 MPa.

9.3.1.2 The Microstructural Examination of the RZ5DF-14% vol. Saffil MMC Tested at Ambient Temperature

Metallographic examinations were conducted to evaluate the influence of various process parameters (pouring and die temperature) on the squeeze infiltrated RZ5DF-14% vol. Saffil MMC microstructure. Microstructural examinations were carried out on specimens selected from those which produced the highest, intermediate and lowest UTS values. The combinations were: (i) pouring temperature of 780°C and die temperature of 275°C for the highest UTS value (figure 9-10), (ii) pouring temperature of 780°C and die temperature of 250°C for the intermediate UTS value (figure 9-11), and (iii) pouring temperature of 780°C and die temperature of 225°C for the lowest UTS value (figure 9-12). The results of the microstructural examinations are presented below.

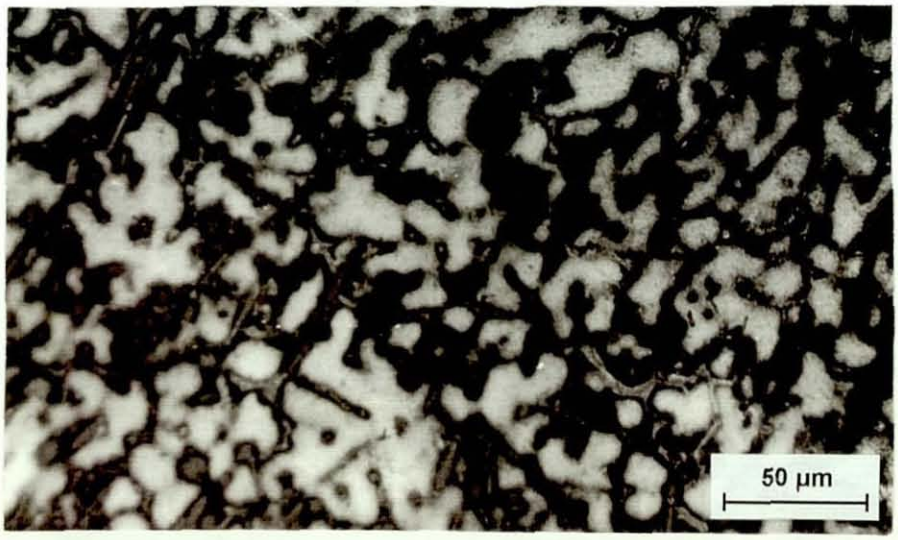


Figure 9-10 Optical microstructure of the squeeze infiltrated RZ5DF-14% vol. Saffil MMC with the highest UTS of 247 MPa at ambient temperature, providing an average grain size of 24µm.

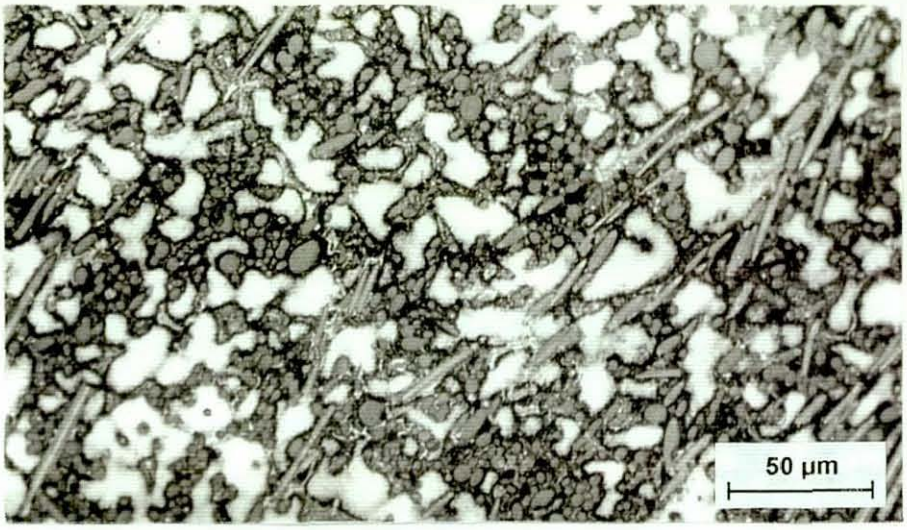


Figure 9-11 Optical microstructure of the squeeze infiltrated RZ5DF-14% vol. Saffil MMC with an intermediate UTS of 242 MPa at ambient temperature, providing an average grain size of 24µm.

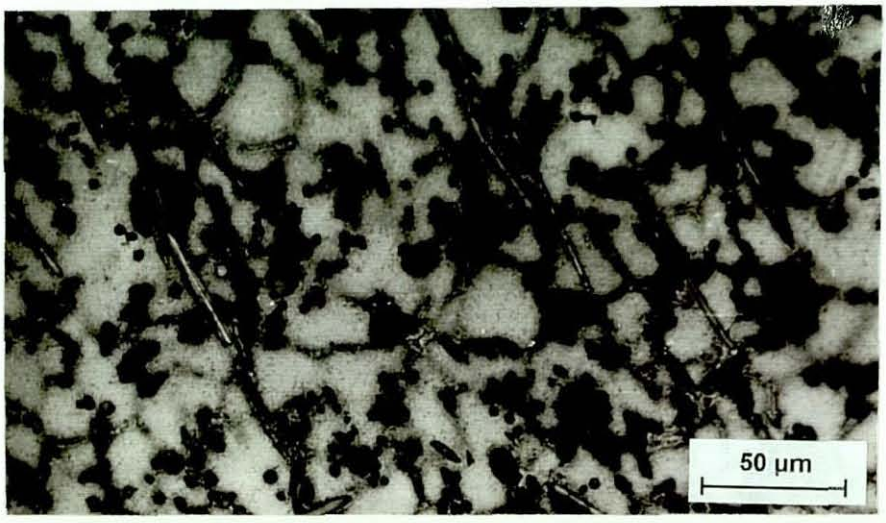


Figure 9-12 Optical microstructure of the squeeze infiltrated RZ5DF-14% vol. Saffil MMC with the lowest UTS of 229 MPa at ambient temperature, providing an average grain size of 27µm.

Figure 9-10 shows the smallest grain size produced by a pouring temperature of 780°C and die temperature of 275°C. Figure 9-12 shows the largest grain size produced by a pouring temperature of 780°C and die temperature of 225°C. The smaller grain size was associated with greater strength for the RZ5DF-14% vol. Saffil MMC.

9.3.2 The Mechanical Properties and Microstructures of the RZ5DF-14% vol. Saffil MMC Tested at 250°C

The following sections will describe the effects of pouring and die temperature on the elevated temperature properties and microstructures of the RZ5DF-14% vol. Saffil MMC. Detailed results of

the mechanical properties of squeeze infiltrated RZ5DF-14% vol. Saffil MMC specimens, tested at elevated temperature, are presented in table 4 of appendix C.

9.3.2.1 The Elevated Properties of RZ5DF-14% vol. Saffil MMC Tested at 250°C

The effects of pouring and die temperature on UTS at an elevated temperature (250°C) are graphically shown in figure 9-13. The chart indicates that the combination of 780°C pouring temperature and 250°C die temperature produced the highest UTS amongst the three pouring temperatures. The results again indicate that the highest temperature gradient with the combination of the highest pouring temperature of 780°C and lowest die temperature of 225°C produced the lowest UTS.

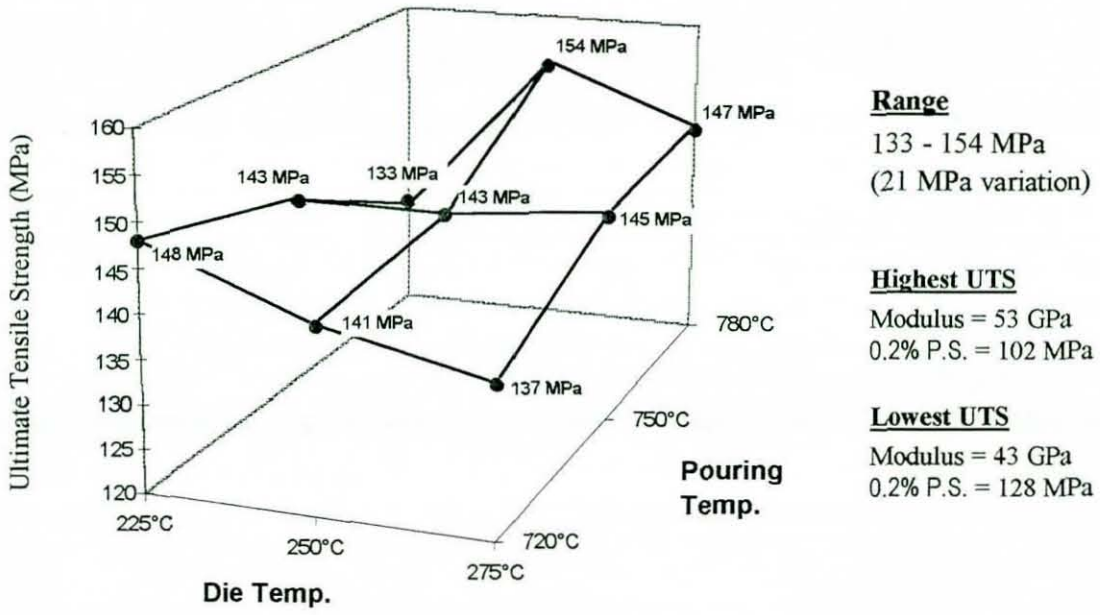


Figure 9-13 The effects of pouring and die temperature on UTS at 250°C (RZ5DF-14% vol. Saffil MMC)

In essence, the overall effects of pouring and die temperature were UTS values (tested at 250°C) with a range of 21 MPa, i.e. a lowest UTS of 133 MPa and a highest UTS of 154 MPa. At the highest value of 154 MPa, the value for Young's modulus was 53 GPa and the 0.2% proof stress was 102 MPa at 250°C.

The influence of pouring temperature on the % elongation and the % area reduction at different levels of die temperature are shown in table 9-4. The % elongation ranges from values of 3.1 to 4.5% and the % area reduction ranges from values of 2.2 to 3.8%. The results show a reversed relationship of % elongation and % area reduction with UTS at an elevated temperature, where a lowest value of elongation of 3.1% and a lowest value of area reduction of 2.2% corresponds with the highest value for UTS.

	225°C		250°C		275°C	
	% Elongation	% Area Reduction	% Elongation	% Area Reduction	% Elongation	% Area Reduction
720°C	3.9%	3.5%	4.5%	3.6%	3.6%	3.2%
750°C	3.7%	2.7%	3.2%	2.4%	4.2%	3.8%
780°C	3.5%	2.5%	3.1%	2.2%	3.7%	3%

Table 9-4 The effects of pouring and die temperature on % elongation and % area reduction of RZ5DF-14% vol. Saffil MMC specimens tested at 250°C.

9.3.2.2 The Microstructural Examination of the RZ5DF-14% vol. Saffil MMC Tested at 250°C

Metallographic examinations were conducted to evaluate the influence of pouring and die temperature on the squeeze infiltrated RZ5DF-14% vol. Saffil MMC microstructures which were exposed to a 250°C test condition. Microstructural examinations were carried out on specimens selected from those which have given the highest, intermediate and lowest UTS values. The combinations were: (i) pouring temperature of 780°C and die temperature of 250°C for the highest UTS value (figure 9-14), (ii) pouring temperature of 780°C and die temperature of 275°C for the intermediate UTS value (figure 9-15), and (iii) pouring temperature of 780°C and die temperature of 225°C for the lowest UTS value (figure 9-16). The results of the microstructural examinations are presented below.

Figure 9-14 Optical microstructure of the squeeze infiltrated RZ5DF-14% vol. Saffil MMC with the highest UTS of 154 MPa at 250°C, providing an average grain size of 26µm.

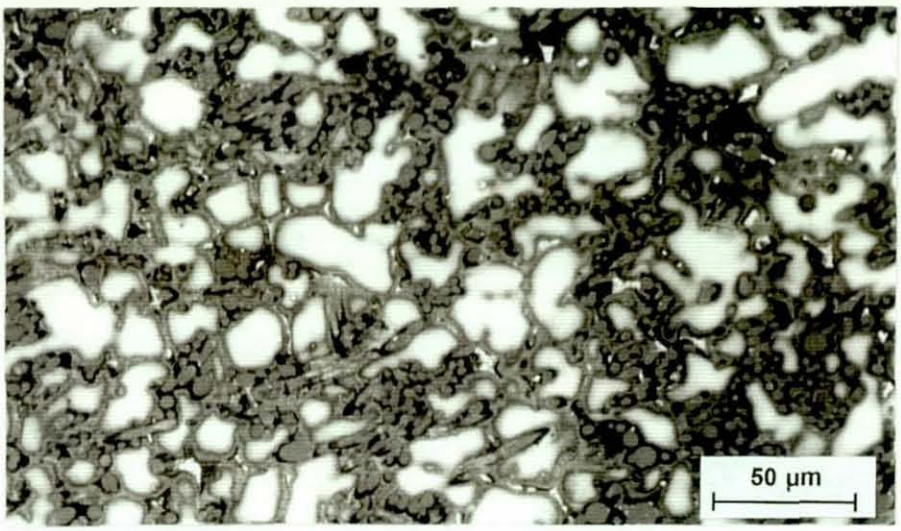


Figure 9-15 Optical microstructure of the squeeze infiltrated RZ5DF-14% vol. Saffil MMC with an intermediate UTS of 147 MPa at 250°C, providing an average grain size of 26µm.

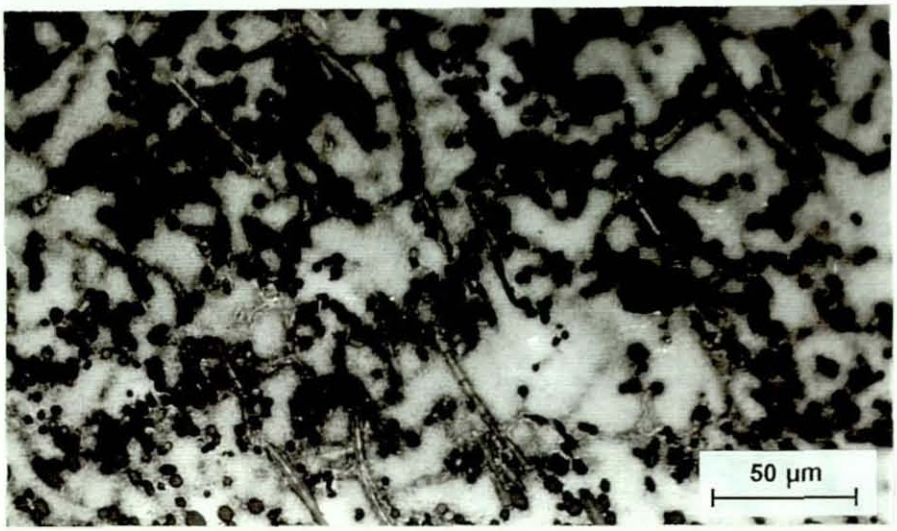


Figure 9-16 Optical microstructure of the squeeze infiltrated RZ5DF-14% vol. Saffil MMC with the lowest UTS of 133 MPa at 250°C, providing an average grain size of 27µm.

It can be seen, from the figures above, that the pouring and die temperature had no influence on the grain sizes for RZ5DF-14% vol. Saffil MMC at an elevated temperature. The micrographs, presented in figures 9-14 to 9-16, show no difference in grain size, as the average grain size only ranges from 26µm to 27µm.

9.4 The Effect of Pouring and Die Temperatures on the Mechanical Properties and Microstructures of the RZ5 Alloy

This section describes the observations made on the influence of pouring and die temperature on the mechanical properties and grain size for the castings produced with the RZ5 alloy. The difference between the RZ5 and RZ5DF alloys (studied in section 9.2) was the presence of zirconium which acts as a grain refiner for the Mg-Zn-RE alloy. The effects of pouring and die temperatures on the UTS

values are graphically presented. Individual points on each graph show the average value taken from three specimens.

9.4.1 The Mechanical Properties and Microstructures of the RZ5 Alloy Tested at Ambient Temperature

The following sections will illustrate the effect of pouring and die temperature on the ambient temperature properties and the microstructures of the RZ5 alloy. Detailed results of the mechanical properties of squeeze cast RZ5 alloy specimens, tested at ambient temperature, are presented in table 5 of appendix C.

9.4.1.1 The Mechanical Properties of the RZ5 Alloy Tested at Ambient Temperature

The influence of pouring temperature on UTS at different die temperatures is presented graphically in figure 9-17, which indicates that the combination of 750°C pouring temperature and 250°C die temperature produced the highest value for UTS. A lower but consistent level of UTS was achieved at the die temperature of 275°C.

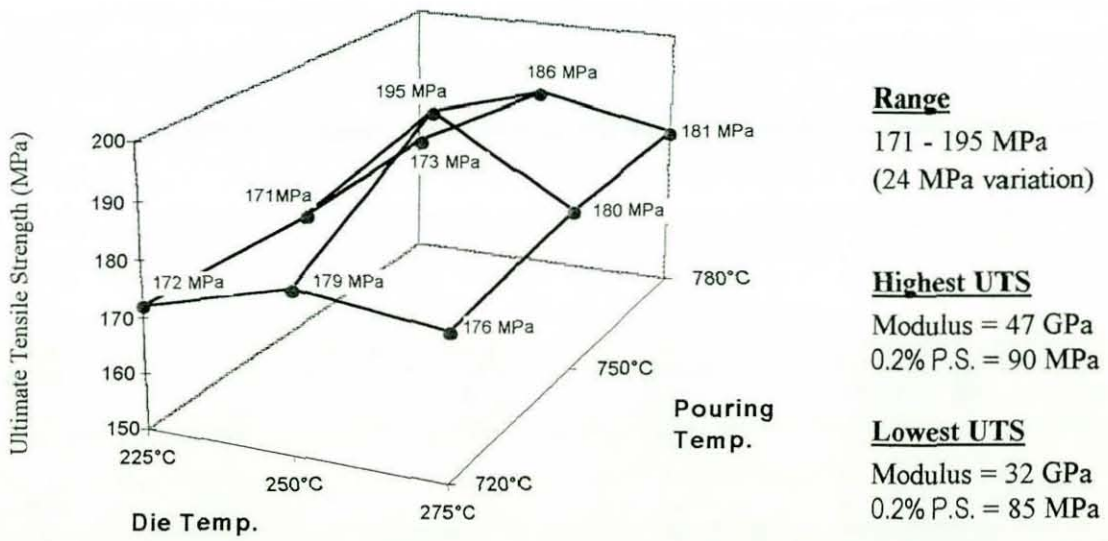


Figure 9-17 The effects of pouring and die temperature on UTS at ambient temperature (RZ5 alloy)

In essence, the influence of pouring and die temperatures were UTS values (tested at ambient temperature) with a range of 24 MPa, i.e. a lowest UTS of 171 MPa and a highest UTS of 195 MPa. At the highest value of 195 MPa, the value for Young's modulus was 47 GPa and the 0.2% proof stress was 90 MPa.

The influence of pouring temperature on % elongation and % area reduction at different levels of die temperature are shown in table 9-5. The % elongation ranges from 8.5 to 11%, and the % area

reduction from 5 to 7.2%. The results show that the combination of 750°C pouring temperature and 250°C die temperature produced the highest % elongation and the highest % area reduction of 11% and 7.2% respectively. At this combination of pouring and die temperatures, the UTS value was also at its highest of 195 MPa. The results also show that the lowest die temperature of 225°C produced a more consistent elongation and area reduction value.

	225°C		250°C		275°C	
	% Elongation	% Area Reduction	% Elongation	% Area Reduction	% Elongation	% Area Reduction
720°C	8.6%	5.4%	9.7%	6.2%	8.6%	5%
750°C	8.5%	5.4%	11%	7.2%	10.3%	6.7%
780°C	9.7%	5.9%	10.7%	6.7%	9.3%	5.3%

Table 9-5 The effects of pouring and die temperature on % elongation and % area reduction of RZ5 alloy specimens tested at ambient temperature.

9.4.1.2 The Microstructural Examination of the RZ5 Alloy Tested at Ambient Temperature

The metallographic examinations were conducted to evaluate the effects of various process parameters (pouring and die temperature) on the squeeze cast RZ5 microstructure. Microstructural examinations were carried out on specimens selected from those which produced the highest, intermediate and lowest UTS values. The combinations were: (i) pouring temperature of 750°C and die temperature of 250°C for the highest UTS value (figure 9-18), (ii) pouring temperature of 780°C and die temperature of 275°C for the intermediate UTS value (figure 9-19), and (iii) pouring temperature of 750°C and die temperature of 225°C for the lowest UTS value (figure 9-20). The results of the microstructural examinations are presented below.

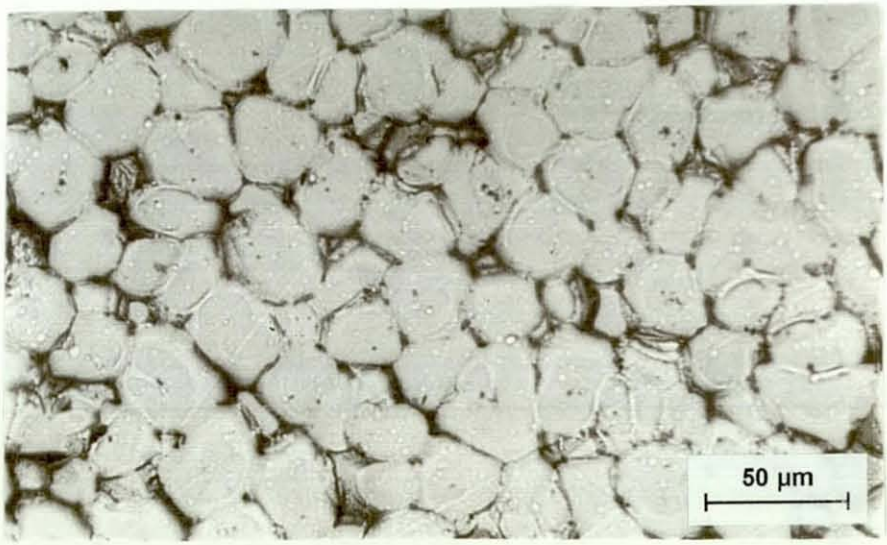


Figure 9-18 Optical microstructure of the squeeze cast RZ5 alloy with the highest UTS of 195 MPa at ambient temperature, providing an average grain size of 21µm.

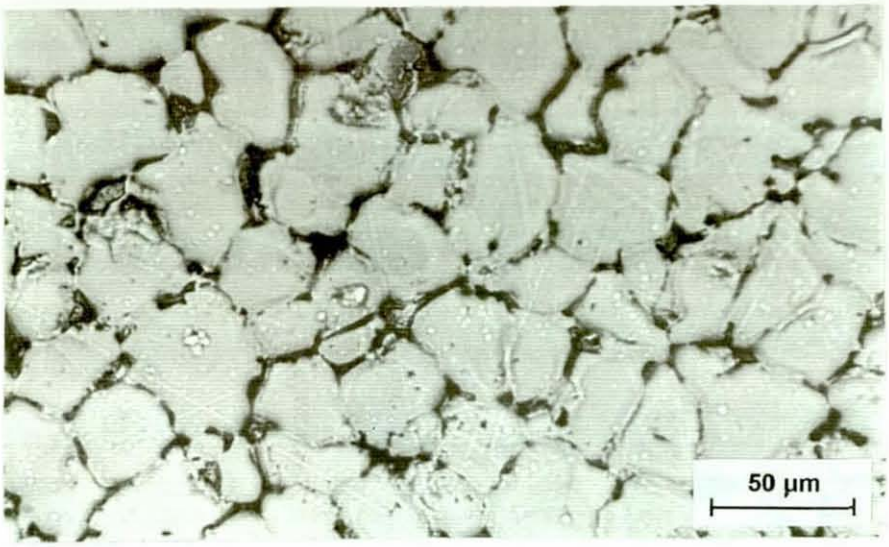


Figure 9-19 Optical microstructure of the squeeze cast RZ5 alloy with an intermediate UTS of 181 MPa at ambient temperature, providing an average grain size of 25µm.

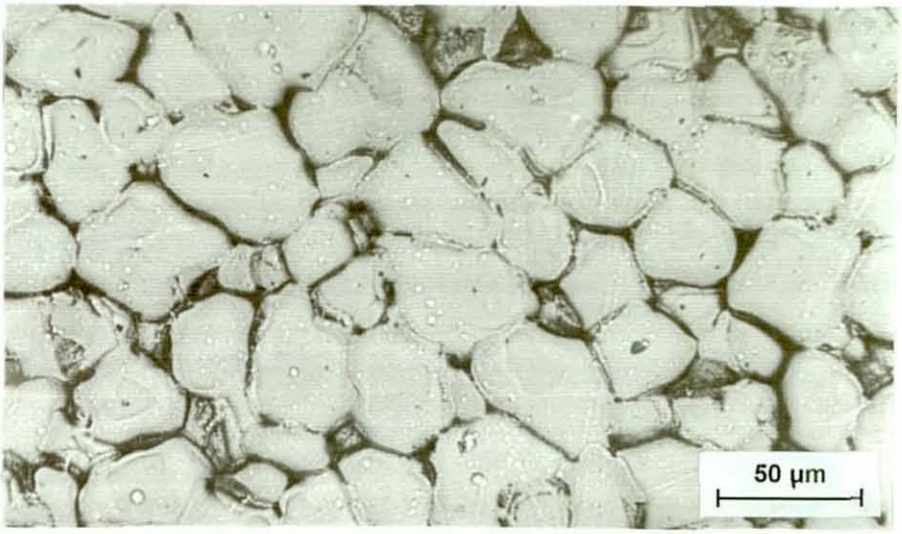


Figure 9-20 Optical microstructure of the squeeze cast RZ5 alloy with the lowest UTS of 171 MPa at ambient temperature, providing an average grain size of 26µm.

Figure 9-18 shows the smallest grain size produced by a pouring temperature of 750°C and die temperature of 250°C. Figure 9-20 shows the largest grain size produced by pouring temperature of 750°C and die temperature of 225°C. In general, it can be seen from the UTS values associated with the figures that the smaller grain size resulted in greater strength for RZ5 alloy.

9.4.2 The Mechanical Properties and Microstructures of the RZ5 Alloy Tested at 250°C

The following sections will illustrate the effect of pouring and die temperature on the elevated temperature properties and microstructures of the RZ5 alloy. Detailed results of the mechanical properties of squeeze cast RZ5 alloy specimens, tested at elevated temperature, are presented in table 6 of appendix C.

9.4.2.1 The Mechanical Properties of the RZ5 Alloy Tested at 250°C

The effects of pouring and die temperature on UTS at an elevated temperature of (250°C) are presented graphically in figure 9-21, which indicates that the combination of 720°C pouring temperature and 225°C die temperature produced the highest value for UTS. The results also indicate that the lowest die temperature of 225°C produced a higher level of UTS at the elevated temperature.

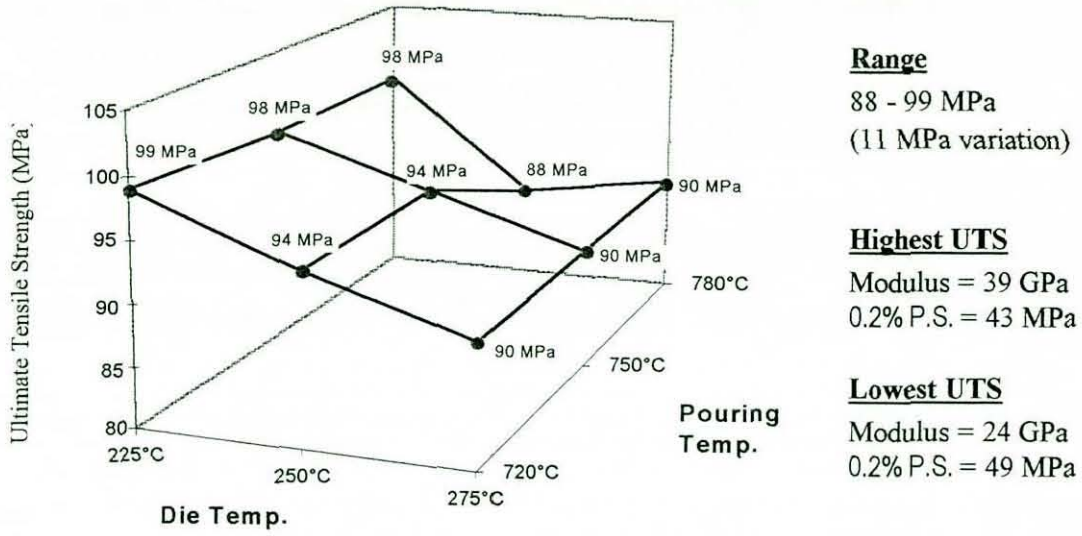


Figure 9-21 The effects of pouring and die temperature on the UTS at 250°C (RZ5 alloy)

In essence, the overall effects of pouring and die temperature were UTS values (tested at 250°C) with a range of 11 MPa, i.e. a lowest UTS of 88 MPa and a highest UTS of 99 MPa. At the highest value of 99 MPa, the value for Young's modulus was 39 GPa and the 0.2% proof stress was 43 MPa.

The effects of pouring temperature on the % elongation and the % area reduction at different levels of die temperature are presented in table 9-6. The % elongation ranges from 34 to 46%, and the % area reduction ranges from of 41 to 66%. The results show that, in general, the intermediate pouring temperature of 750°C and/or intermediate die temperature of 250°C produced more consistent values for the elongation and area reduction.

	225°C		250°C		275°C	
	% Elongation	% Area Reduction	% Elongation	% Area Reduction	% Elongation	% Area Reduction
720°C	42%	63%	45%	64%	34%	41%
750°C	45%	62%	44%	66%	46%	65%
780°C	39%	49%	44%	57%	41%	55%

Table 9-6 The effects of pouring and die temperature on the % elongation and the % area reduction of RZ5 alloy specimens tested at 250°C.

9.4.2.2 The Microstructural Examination of the RZ5 Alloy Tested at 250°C

The effects of pouring and die temperature on the squeeze cast RZ5 microstructure were evaluated using metallographic examinations following exposure to the 250°C test condition/environment. Specimens were selected from those that produced the highest, intermediate and lowest UTS values. The combinations were: (i) pouring temperature of 720°C and die temperature of 225°C for the highest UTS value (figure 9-22), (ii) pouring temperature of 720°C and die temperature of 275°C for the intermediate UTS value (figure 9-23), and (iii) pouring temperature of 780°C and die temperature of 250°C for the lowest UTS value (figure 9-24). The results of the microstructural examinations are presented below.

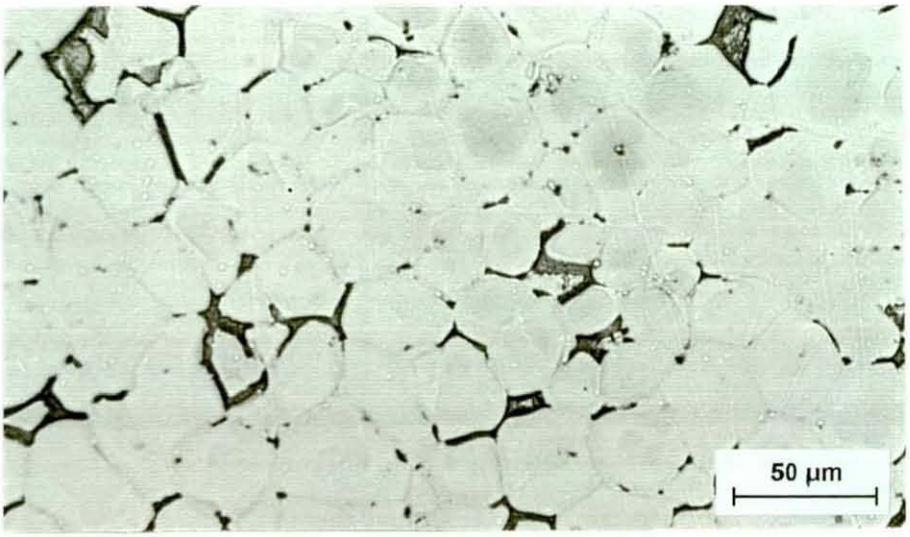


Figure 9-22 Optical microstructure of the squeeze cast RZ5 alloy with the highest UTS of 99 MPa at an elevated temperature, providing an average grain size of 23µm.

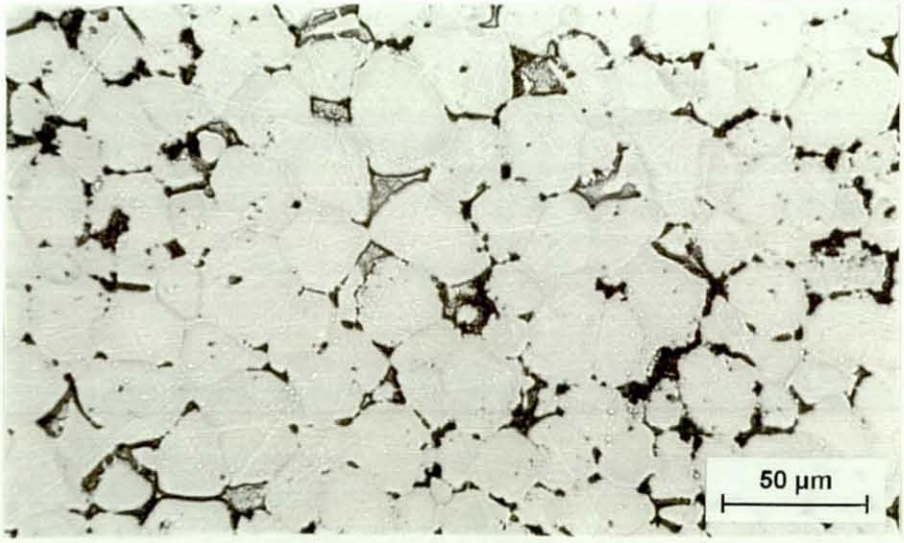


Figure 9-23 Optical microstructure of the squeeze cast RZ5 alloy with an intermediate UTS of 90 MPa at an elevated temperature, providing an average grain size of 23µm.

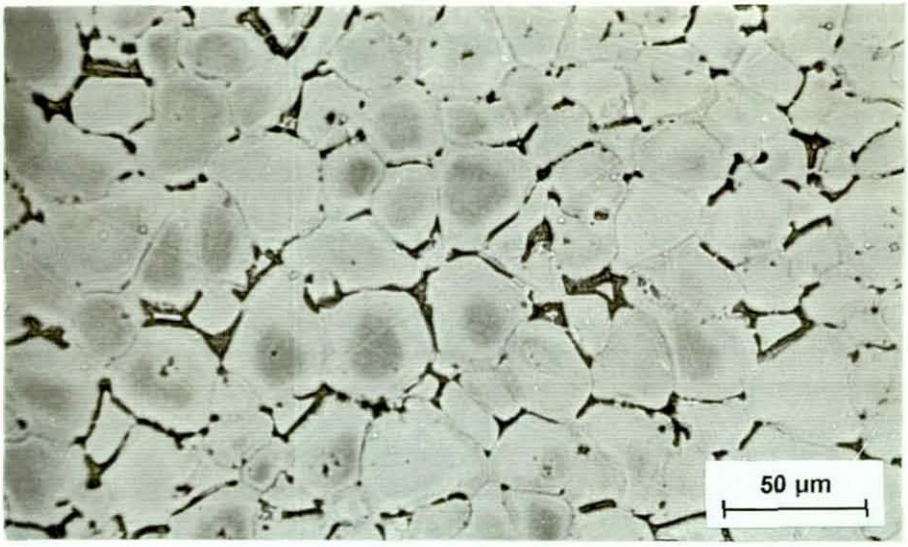


Figure 9-24 Optical microstructure of the squeeze cast RZ5 alloy with the lowest UTS of 88 MPa at an elevated temperature, providing an average grain size of 22µm.

It can be seen, from figures 9-22 to 9-24, that the microstructures show a more consistent grain size of 22 to 23 µm following exposure to elevated temperatures. However, a comparison of the UTS values associated with the figures reveals that the smaller grain size resulted in a reduction in strength for the RZ5 alloy at elevated temperatures, which was the opposite effect to the relationship between strength and grain size at ambient temperatures.

9.5 The Effect of Pouring and Die Temperatures on the Mechanical Properties and Microstructures of the RZ5-14% vol. Saffil MMC

This section describes the observations made on the effects of pouring and die temperature on the mechanical properties and grain size for castings produced with the RZ5-14% vol. Saffil MMC. The effects of pouring and die temperatures on values of UTS are graphically presented, where individual points show the average value taken from three specimens.

9.5.1 The Mechanical Properties and Microstructures of the RZ5-14% vol. Saffil MMC Tested at Ambient Temperature

The following sections will illustrate the effects of pouring and die temperature on the ambient temperature properties and the microstructures of the RZ5-14% vol. Saffil MMC. Detailed results of the mechanical properties of squeeze infiltrated RZ5-14% vol. Saffil MMC specimens, tested at ambient temperature, are presented in table 7 of appendix C.

9.5.1.1 The Mechanical Properties of the RZ5-14% vol. Saffil MMC Tested at Ambient Temperature

The effects of pouring temperature on values for UTS at different die temperatures are presented in figure 9-25. The graph shows that two combinations of pouring temperature and die temperature (780°C with 250°C, and 780°C with 275°C) produced the highest value for UTS. The lowest UTS value was produced at the pouring temperature of 780°C and die temperature of 225°C.

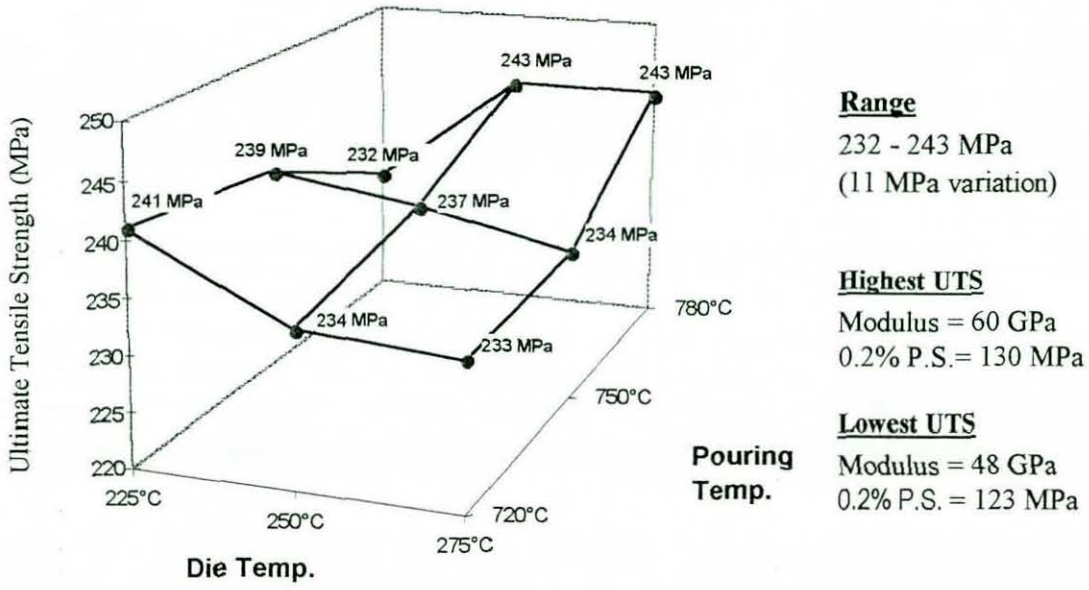


Figure 9-25 The effects of pouring and die temperature on the UTS at an ambient temperature (RZ5-14% vol. Saffil MMC)

In essence, the overall effects of pouring and die temperatures were UTS values (tested at ambient temperature) with a range of 11 MPa, i.e. a lowest UTS of 232 MPa and a highest UTS of 243 MPa. At the highest value of 243 MPa, the value for Young's modulus was 60 GPa and the 0.2% proof stress was 130 MPa.

The effects of pouring temperature on % elongation and % area reduction at different levels of die temperature are shown in table 9-7. The % elongation ranges from 2 to 3%, and the % area reduction ranges from 1.7 to 2.2%. The results show that the combination of 780°C pouring temperature and 250°C die temperature produced the highest % elongation and the highest % area reduction of 3% and 2.2% respectively. At this combination of pouring and die temperatures, the UTS value was also at its highest of 243 MPa.

	225°C		250°C		275°C	
	% Elongation	% Area Reduction	% Elongation	% Area Reduction	% Elongation	% Area Reduction
720°C	2.5%	2%	2%	1.7%	2.2%	2%
750°C	2.3%	1.9%	2.3%	2%	2.4%	1.8%
780°C	2.2%	1.8%	3%	2.2%	2.7%	2.1%

Table 9-7 The effects of pouring and die temperature on the % elongation and % area reduction of RZ5-14% vol. Saffil MMC specimens tested at ambient temperature.

9.5.1.2 The Microstructural Examination of the RZ5-14% vol. Saffil MMC Tested at Ambient Temperature

Metallographic examinations were conducted to evaluate the effects of various process parameters (pouring and die temperature) on the microstructure of the squeeze infiltrated RZ5-14% vol. Saffil MMC microstructure. Microstructural examinations were carried out on specimens selected from those which produced gave the highest, intermediate and lowest UTS values. The combinations were: (i) pouring temperature of 780°C and die temperature of 250°C for the highest UTS value (figure 9-26), (ii) pouring temperature of 750°C and die temperature of 225°C for the intermediate UTS value (figure 9-27), and (iii) pouring temperature of 780°C and die temperature of 225°C for the lowest UTS value (figure 9-28). The results of the microstructural examinations are presented below.

Figure 9-26 Optical microstructure of the squeeze infiltrated RZ5-14% vol. Saffil MMC with the highest UTS of 243 MPa at ambient temperature, providing an average grain size of 24µm.

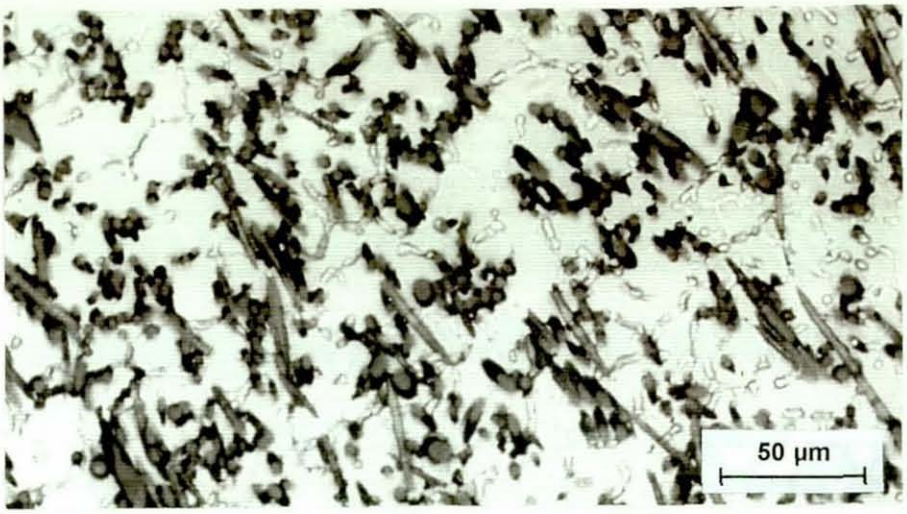


Figure 9-27 Optical microstructure of the squeeze infiltrated RZ5-14% vol. Saffil MMC with an intermediate UTS of 239 MPa at ambient temperature, providing an average grain size of 25µm.

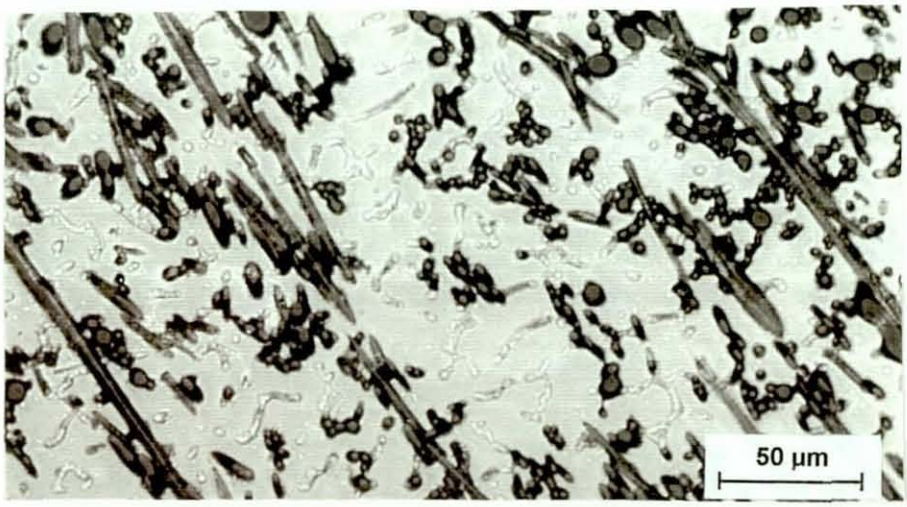


Figure 9-28 Optical microstructure of the squeeze infiltrated RZ5-14% vol. Saffil MMC with the lowest UTS of 232 MPa at ambient temperature, providing an average grain size of 25µm.

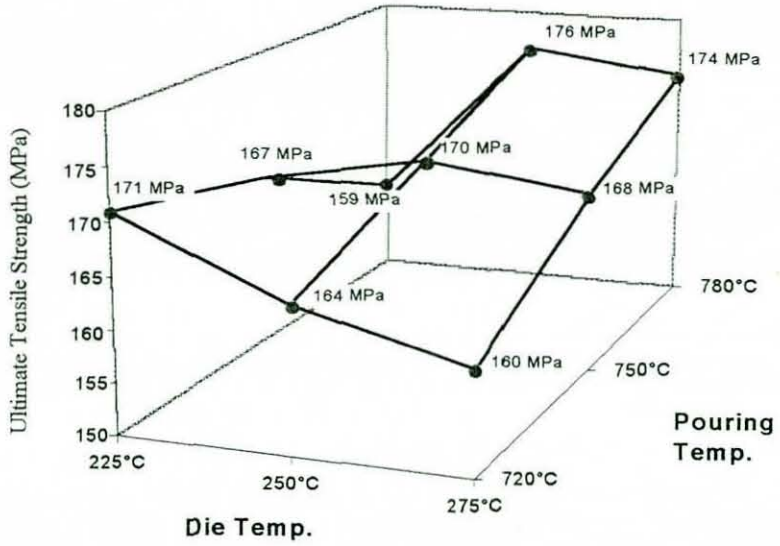
It can be seen, from the figures above, that the pouring and die temperature had no influence on the grain sizes for RZ5-14% vol. Saffil MMC. The micrographs presented in figures 9-26 to 9-28, show the average grain size ranges from 24µm to 25µm.

9.5.2 The Mechanical Properties and Microstructures of the RZ5-14% vol. Saffil MMC Tested at 250°C

The following sections will illustrate the effect of pouring and die temperature on the elevated temperature properties and microstructures of the RZ5-14% vol. Saffil MMC. Detailed results of the mechanical properties of squeeze infiltrated RZ5-14% vol. Saffil MMC specimens, tested at elevated temperature, are presented in table 8 of appendix C.

9.5.2.1 The Elevated Properties of RZ5-14% vol. Saffil MMC Tested at 250°C

The effects of pouring and die temperature on UTS at an elevated temperature of 250°C are shown in figure 9-29. The chart indicates that the combination of 780°C pouring temperature and 250°C die temperature produced the highest value for UTS. The results again indicate that the highest temperature gradient, with the combination of the highest pouring temperature of 780°C and lowest die temperature of 225°C, produced the lowest value for UTS.



Range
159 - 176 MPa
(17 MPa variation)

Highest UTS
Modulus = 46 GPa
0.2% P.S. = 101 MPa

Lowest UTS
Modulus = 42 GPa
0.2% P.S. = 99 MPa

Figure 9-29 The effects of pouring and die temperature on UTS at 250°C (RZ5-14% vol. Saffil MMC)

In essence, the overall effects of pouring and die temperature were UTS values (tested at 250°C) with a range of 17 MPa, i.e. a lowest UTS of 159 MPa and a highest UTS of 176 MPa. At the highest value of 176 MPa, the Young's modulus value was 46 GPa and the 0.2% proof stress was 101 MPa at 250°C.

The effects of pouring temperature on the % elongation and the % area reduction at different levels of die temperature are shown in table 9-8.

	225°C		250°C		275°C	
	% Elongation	% Area Reduction	% Elongation	% Area Reduction	% Elongation	% Area Reduction
720°C	3.5%	3.3%	3.4%	3.3%	3.3%	2.7%
750°C	3.1%	2.2%	3.4%	2.4%	3.5%	2.3%
780°C	3.4%	3.3%	3%	2.2%	3.1%	2.4%

Table 9-8 The effects of pouring and die temperature on % elongation and % area reduction of RZ5-14% vol. Saffil MMC specimens tested at 250°C.

It can be seen, from table 9-8, that % elongation ranges from 3 to 3.5% and the % area reduction from 2.2 to 3.3%. The results indicated a reversed pattern between % elongation and % area reduction and UTS value at an elevated temperature (figure 9-29), where the lowest values of elongation of 3% and area reduction of 2.2% was associated with the highest UTS.

9.5.2.2 The Microstructural Examination of the RZ5-14% vol. Saffil MMC Tested at 250°C

Metallographic examinations were conducted to evaluate the effects of various process parameters (pouring and die temperature) on the squeeze infiltrated RZ5-14% vol. Saffil MMC microstructures following exposure to the 250°C test condition. Microstructural examinations were carried out on specimens, selected from those which gave the highest, intermediate and lowest UTS values. The combinations were: (i) pouring temperature of 780°C and die temperature of 250°C for the highest UTS value (figure 9-30), (ii) pouring temperature of 750°C and die temperature of 275°C for the intermediate UTS value (figure 9-31), and (iii) pouring temperature of 780°C and die temperature of 225°C for the lowest UTS value (figure 9-32). The results of the microstructural examinations are presented below.

Figure 9-30 Optical microstructure of the squeeze infiltrated RZ5-14% vol. Saffil MMC with the highest UTS of 176 MPa at 250°C, providing an average grain size of 24µm.

Figure 9-31 Optical microstructure of the squeeze infiltrated RZ5-14% vol. Saffil MMC with an intermediate UTS of 168 MPa at 250°C, providing an average grain size of 24µm.

Figure 9-32 Optical microstructure of the squeeze infiltrated RZ5-14% vol. Saffil MMC with the lowest UTS of 159 MPa at 250°C, providing an average grain size of 24µm.

Again, it can be seen that the pouring and die temperature had no influence on the grain sizes for RZ5-14% vol. Saffil MMC casting. The micrographs shown in figures 9-30 to 9-32, do not indicate any difference in grain size as all have an average grain size of 24µm.

9.6 The Effects of Pouring and Die Temperature on Casting Dimensions

In line with the analysis of the effects of pouring and die temperatures, it was also possible to include a restricted study of the dimensional repeatability of the squeeze casting process. Of all the different casting processes, squeeze casting is known to offer the best results in terms of the repeatability of

casting dimensions. The capability of repeating a tolerance of ±0.2mm has been reported by Clegg [167]. In the author's opinion, the likely factors which influenced the casting dimensions of squeeze cast components were: (i) control of the pouring and die temperature, (ii) control of the applied pressure and (iii) the consistency of die coating thickness.

At the time of this research, no work was reported on the possible effects of these factors on the dimension accuracy; this, in particular, relates to pouring and die temperatures. Therefore, the effects of pouring and die temperature on the dimensions of squeeze cast RZ5 alloy were studied. Dimensional comparisons were made along the width of the cast ingots. The production of this data allowed the further evaluation of the process capability of squeeze casting, in terms of its ability to produce consistently accurate castings. Dimensions measured from castings produced from the nine process temperature combinations are recorded in table 9-9 and graphically presented in figure 9-33.

	225°C	250°C	275°C
720°C	74.74 mm	74.74 mm	74.72 mm
750°C	74.70 mm	74.73 mm	74.73 mm
780°C	74.71 mm	74.76 mm	74.74 mm

Table 9-9 Width of squeeze cast RZ5 ingot produced with applied pressure of 60 MPa

From the results presented in table 9-9, the overall effects of pouring and die temperature were casting dimensions within ±0.03mm, i.e. a smallest width of 74.70mm and a largest width of 74.76mm. In figure 9-33, the results show no apparent trends between the process (pouring and die) temperatures and the repeatability of casting dimensions.

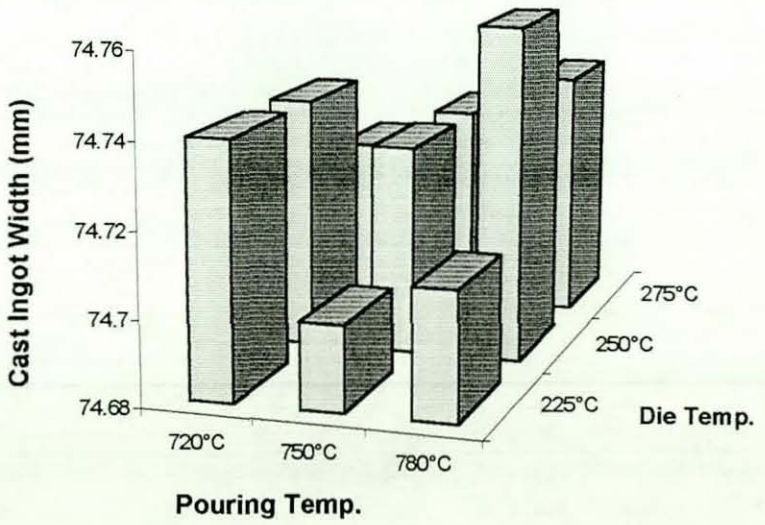


Figure 9-33 The effects of pouring and die temperature on the casting dimension (width)

Of the factors (pouring and die temperature, applied pressure and die coating) mentioned earlier, die coating was seen as most likely to cause dimensional variation in squeeze casting. The process temperatures, graphically presented in figure 9-33, do not exert any apparent influence. In relation to the applied pressure, a value of 60 MPa was carefully maintained for all the castings. It was therefore unlikely that this was the cause of the dimensional variations.

The variations in casting dimensions were most likely caused by the irregularity in die coatings, since it was impossible for the operator to manually provide a precise layer of coating to an accuracy of 10μm to 30μm. One possible approach for the improvement of consistency would be to mechanise the coating process.

9.7 The Selection of Casting Parameters for the Improvement of Material Properties through Heat Treatment

It was possible to establish the best pouring and die temperature combination to obtain the optimum ambient and elevated temperature UTS values for each of the materials studied in this research. The detailed study of the influence of process temperatures on ambient and elevated UTS values was carried out and the results were presented in sections 9.2 to 9.5. However, for many engineering applications, castings are required to operate in both ambient and elevated temperature environments and it was essential that the material had excellent properties at these operating temperatures.

Heat treatment was carried out on selected castings to evaluate the improvement in properties. However, the overall improvement in material properties was not the same for all the different combinations of casting parameters; certain combinations produced greater improvement. It was not an efficient use of time and resources, nor was it necessary for heat treatment tests to be carried out on all the ranges of casting parameters explored in this research. Rather, it was possible to extract the combination of casting parameters which provided the most potential for improvement in material properties through heat treatment.

Therefore, a system for the assignment of numerical weightings was used to determine the set of process parameters that produced castings with the best UTS values at ambient and elevated temperatures. This system of numerical weighting was applied for each of the four research materials. The assignment of a numerical weighting to an individual set of casting parameters was introduced to indicate the potential for improvement of material properties at both elevated and ambient temperatures. The weighting can be used to make comparisons, in order to identify the programme consisting of only the essential heat treatment tests. The system of numerical weighting provided a ready method for the comparison of casting parameters, in a tabular form, as opposed to reading of graphs which may cause confusion.

Therefore, it will be explained how the casting parameters were numerically weighted (on individual basis) and selected on the basis that the combination of casting parameters with the highest weighting will result in the best material properties after heat treatment. The desired parameter values for pouring and die temperatures for heat treatment tests were selected by comparing values of "comparative weighting" calculated from the UTS results from tests carried out at ambient and elevated temperatures. The value for comparative weighting was selected as the highest value from a set of individual comparative weighting values. To obtain the best ambient and elevated tensile properties from the study for each material, the individual weighting values had to be calculated, firstly for the tests at ambient temperatures and secondly for tests at elevated temperatures. The UTS values for the ambient temperature (designated by column "X" in the ensuing tables) and elevated temperatures (designated by column "Y" in the ensuing tables) were tabulated for their respective pouring and die temperatures.

The optimum tensile property of the batch was the maximum value from the set of weighting values. Each weighting value was calculated by subtracting the mean from each individual value of each group (i.e. ambient UTS and elevated UTS). Accordingly:

$$\text{Comparative Weighting} = [\text{UTS}_{(\text{ambient})} - \text{mean UTS}_{(\text{ambient})}] + [\text{UTS}_{(\text{elevated})} - \text{mean UTS}_{(\text{elevated})}]$$

$$= \left[Xi - \overline{X} \right] + \left[Yi - \overline{Y} \right]$$

Using this expression, the comparative weightings for each material studied in the secondary casting programme were calculated and tabulated in tables 9-10 to 9-13.

Pouring Temperature	**Die Temperature**	*Xi* (Ambient Temperature UTS)	*Yi* (Elevated Temperature UTS)	**Comparative Weighting** $\left[Xi - \overline{X} \right] + \left[Yi - \overline{Y} \right]$
780°C	225°C	173	97	7
750°C	225°C	169	90	-4
720°C	225°C	179	85	1
780°C	250°C	198	74	9
750°C	250°C	191	77	5
720°C	250°C	189	78	4
780°C	275°C	184	73	-6
750°C	275°C	184	75	-4
720°C	275°C	181	77	-5
	Mean Value	**183** $(\overline{X})$	**80** $(\overline{Y})$	

Table 9-10 Calculations to obtain the highest 'weighting values'. The parameters indicated by the highest weighting were considered to produce the best ambient and elevated tensile properties of squeeze cast RZ5DF alloy (section 9.2).

It can be seen from table 9-10 that the values for the comparative weighting range from -6 to 9. In general, the highest die temperature of 275°C resulted in the lower UTS at both ambient and elevated temperatures, indicated by a low weighting value of -6 to -4. This can also be seen by comparing the values of UTS plotted in figures 9-1 and 9-5.

The highest weighting value of 9 was obtained at a pouring temperature of 780°C and die temperature of 250°C. Therefore, this set of process parameters was used for heat treatment studies on the test specimens. Furthermore, this was selected as the set of process parameters for obtaining the best results for both ambient and elevated temperature applications on squeeze cast RZ5DF alloy.

Pouring Temperature	**Die Temperature**	*Xi* (Ambient Temperature UTS)	*Yi* (Elevated Temperature UTS)	**Comparative Weighting** $[Xi - \bar{X}] + [Yi - \bar{Y}]$
780°C	225°C	229	133	-17
750°C	225°C	237	143	1
720°C	225°C	231	148	0
780°C	250°C	242	154	17
750°C	250°C	236	143	0
720°C	250°C	236	141	-2
780°C	275°C	247	147	15
750°C	275°C	235	145	1
720°C	275°C	231	137	-11
	Mean Value	**236** $(\bar{X})$	**143** $(\bar{Y})$	

Table 9-11 *Calculation to obtain the highest 'weighting values'. The parameters indicated by the highest weighting were considered to produce the best ambient and elevated tensile properties of squeeze infiltrated RZ5DF-14% vol. Saffil MMC (section 9.3).*

It can be seen from table 9-11 that the values for the comparative weighting range from -17 to 17. In general, the lower pouring temperatures of 750°C and 720°C resulted in the lowest UTS at both ambient and elevated temperatures, indicated by a low weighting value of -11 to 1. This can also be seen by comparing the values of UTS plotted in figures 9-9 and 9-13.

The highest weighting value of 17 was obtained at a pouring temperature of 780°C and die temperature of 250°C. Therefore, this set of process parameters was used for heat treatment studies on the test specimens. Furthermore, this was selected as the set of process parameters for obtaining the best results for both ambient and elevated temperature applications on squeeze infiltrated RZ5DF-14% vol. Saffil MMC casting.

Pouring Temperature	Die Temperature	*Xi* (Ambient Temperature UTS)	*Yi* (Elevated Temperature UTS)	Comparative Weighting $[Xi - \bar{X}] + [Yi - \bar{Y}]$
780°C	225°C	173	98	-1
750°C	225°C	171	98	-3
720°C	225°C	172	99	-1
780°C	250°C	186	88	2
750°C	250°C	195	94	17
720°C	250°C	179	94	1
780°C	275°C	181	90	-1
750°C	275°C	180	90	-2
720°C	275°C	176	90	-6
	Mean Value	**179** $(\bar{X})$	**93** $(\bar{Y})$	

Table 9-12 Calculation to obtain the highest 'weighting values'. The parameters indicated by the highest weighting were considered to produce the best ambient and elevated tensile properties of squeeze cast RZ5 alloy (section 9.4).

It can be seen from table 9-12 that the values for the comparative weighting range from -6 to 17. In general, the highest and lowest die temperature resulted in the low UTS at both ambient and elevated temperatures, indicated by a low weighting value of -6 to -1. This can also be seen by comparing the values of UTS plotted in figures 9-17 and 9-21.

The highest weighting value of 17 was obtained at an intermediate pouring temperature of 750°C and die temperature of 250°C. Therefore, this set of process parameters was used for heat treatment studies on the test specimens. Furthermore, this was selected as the set of process parameters for obtaining the best results for both ambient and elevated temperature applications on squeeze cast RZ5 alloy.

Pouring Temperature	Die Temperature	*Xi* (Ambient Temperature UTS)	*Yi* (Elevated Temperature UTS)	Comparative Weighting $[Xi - \bar{X}] + [Yi - \bar{Y}]$
780°C	225°C	232	159	-13
750°C	225°C	239	167	2
720°C	225°C	241	171	8
780°C	250°C	243	176	15
750°C	250°C	237	170	3

(continues on next page) →

720°C	250°C	234	164	-6
780°C	275°C	243	174	13
750°C	275°C	234	168	-2
720°C	275°C	233	160	-11
	Mean Value	**237** $(\overline{X})$	**167** $(\overline{Y})$	

Table 9-13 Calculation to obtain the highest 'weighting values'. The parameters indicated by the highest weighting were considered to produce the best ambient and elevated tensile properties of squeeze infiltrated RZ5-14% vol. Saffil MMC (section 9.5).

It can be seen from table 9-13 that the values for the comparative weighting range from -13 to 15. However no particular trends were apparent for this material (RZ5-14% vol. Saffil MMC).

The highest weighting value of 15 was obtained at a pouring temperature of 780°C and die temperature of 250°C. Therefore, this set of process parameters was used for heat treatment studies on the test specimens. Furthermore, this was selected as the set of process parameters for obtaining the best results for both ambient and elevated temperature applications on squeeze infiltrated RZ5-14% vol. Saffil MMC casting.

9.8 Results for the Heat Treated Castings produced with the Selected Conditions

Heat treatment was used to further improve the mechanical properties of the four materials on which this research was based, i.e. the RZ5DF alloy, RZ5DF-14% vol. Saffil MMC, RZ5 alloy and RZ5-14% vol. Saffil MMC. It was necessary however, to determine (i) a suitable heat treatment process and (ii) the heat treatment parameters/conditions to be used with the process. The conditions which had to be determined included the duration of heat treatment for solutionising and precipitation ageing hardening.

A number of heat treatment processes were available, these were previously discussed in section 3.8.1. The treatment processes are: solution heat treatment (T4), artificial ageing and stabilising treatment (T5), solution treatment followed by artificial ageing treatment (T6) and solution treatment followed by stabilising treatment (T7). In general, only the T5 treatment was used for magnesium-zinc base alloys and in particular, the artificial age hardening T5 treatment was well established commercially for RZ5. As the RZ5DF alloy was similar in composition to the RZ5, T5 treatment temperatures were selected and the investigation focused on the study of the influence of varying durations for the partial solution treatment and for age hardening to determine the best treatment condition to improve the properties.

The T5 heat treatment process involves two stages of heating; firstly, partial solution treatment involves the heating of the specimen for 2 hours at a temperature of 330°C. Secondly, the material was allowed to cool in air. The third stage was artificial age hardening, which involves reheating the specimen at 180°C for a duration of 10 to 16 hours. The purpose of partial solution treatment was to dissolve any concentrated solute remaining in the structure. The subsequent age hardening was conducted to provide the strengthening effect through the production of controlled second phase precipitates (MgZn). The heat treatment conditions, i.e. the individual temperature and durations for the partial solution treatment and age hardening stages were based on recommendations from the material suppliers for the RZ5 alloy.

However, there was an additional consideration because composites of the RZ5 and RZ5DF alloys were involved. It has been reported that there tends to be an accelerated ageing effect in MMCs because of the presence of fibres, owing to the high dislocation density generated from thermal mismatch between the fibres and the alloys [20][170]. As a consequence, the required duration for age hardening on MMC was shortened. Therefore, the detailed heat treatment conditions for partial solution treatment and age hardening had to be determined for the RZ5 and RZ5DF MMCs, for which there was no available information at the time of this research. To determine the appropriate heat treatment conditions, an analysis of a series of peak age hardness and tensile test results was required and the investigation focused on the study of the influence of varying durations for the partial solution treatment and age hardening stages.

It has been reported that the hardness change resulting from precipitation was relatively small for MMCs [171], and tensile properties changes cannot be reliably deduced from hardness measurements. Consequently, it was necessary to study the effect of the hardening process by the direct measurement of the resulting hardness and tensile properties. To establish peak hardness curves, an average of five measurements were taken from each specimen to ensure a more reliable average. Detailed studies to establish the best heat treatment condition for each of the materials are now presented.

9.8.1 Results for the Heat Treated RZ5DF Alloy Produced with Selected Conditions (Pouring Temperature 780°C and Die Temperature 250°C)

Heat treatment was conducted on squeeze cast RZ5DF alloy to further improve the optimum properties obtained from the best combinations of pouring and die temperatures identified in section 9.2. The RZ5DF alloy was a new alloy introduced in this research, as a result no heat treatment information was available. Hence a series of hardness curves had to be constructed to obtain the ageing response of squeeze cast RZ5DF alloy. The effects of heating at 330°C and ageing at 180°C, with varying durations, on the hardness properties are presented in figure 9-34.

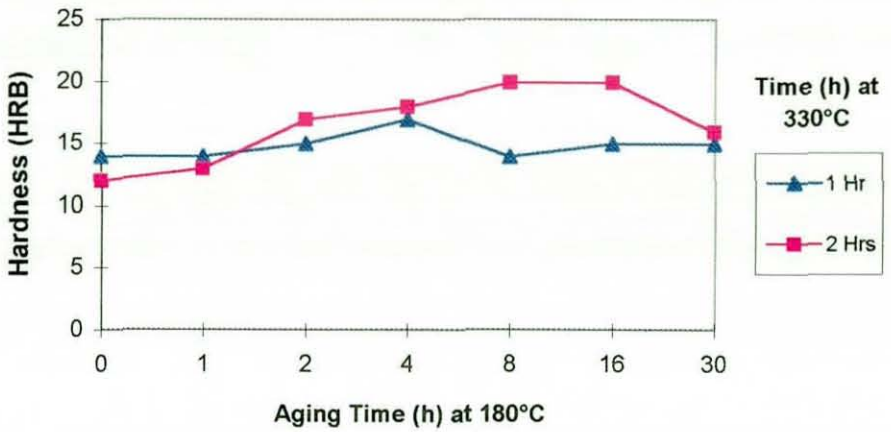


Figure 9-34 Ageing curves for RZ5DF alloy. Specimens were treated at 330°C for 1 or 2 hours, air cooled and aged at 180°C.

It may be seen from figure 9-34, that three heat treatment conditions produced the highest hardness. These were further examined, in order to reliably select the best heat treatment condition, by direct measurement of resulting tensile properties. The three heat treatment conditions were: 2 hours at 330°C for the partial solution treatment, used in combination with 4, 8 and 16 hours respectively at 180°C for the age hardening stage. The tensile properties produced under these conditions are presented in figure 9-35. Detailed results of the tensile properties are presented in table 9 of appendix C.

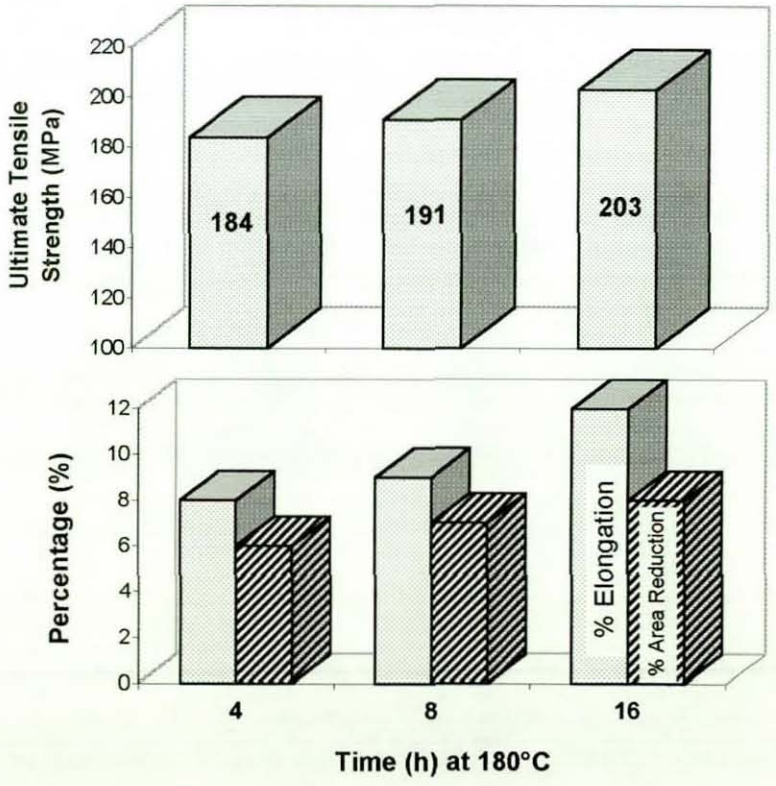


Figure 9-35 Tensile plot of RZ5DF alloy treated at 330°C for 2 hours, air cooled and age hardened for 4, 8 and 16 hours respectively at 180°C.

The results in figure 9-35 show that the heat treated, squeeze cast RZ5DF alloy (330°C for 2 hours, air cooled and aged for 16 hours at 180°C) provided the most promising improvements in strength. Hence, this heat treatment condition (T5 treatment) was selected for further investigations centred around the ambient and elevated properties of the squeeze cast RZ5DF alloy. The resulting properties of the as-cast and T5 treated RZ5DF alloy are presented in table 9-14.

	RZ5DF alloy (as-cast)		**RZ5DF alloy (T5 treated)**	
	Ambient Properties	Elevated Properties	Ambient Properties	Elevated Properties
UTS (MPa)	198 MPa	74 MPa	201 MPa	85 MPa
% Elongation	12.8 %	46 %	13.2 %	46 %
% Area Reduction	7.7 %	65 %	8 %	63 %
Young's Modulus	45 GPa	27 GPa	37 GPa	23 GPa
0.2% Proof Stress	97 MPa	63 MPa	93 MPa	48 MPa

Table 9-14 The as-cast and T5 treated properties of squeeze cast RZ5DF alloy.

Micro-examinations were conducted to obtain qualitative information on the effect of heat treatment on the mechanical properties of the MMC. The micrographs of T5 treated RZ5DF alloy, after tests at ambient and elevated temperature, are shown in figures 9-36 and 9-37 respectively.

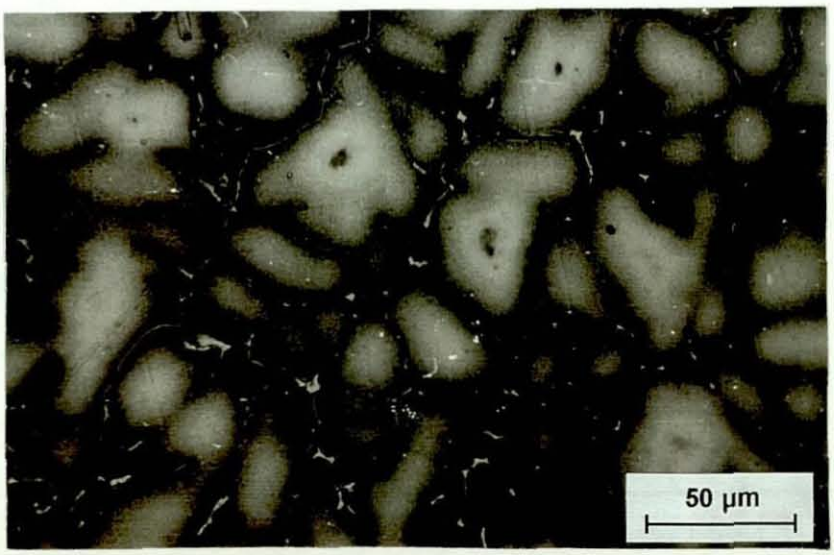


Figure 9-36 Optical microstructure of T5 treated squeeze cast RZ5DF alloy produced with pouring temperature of 780°C and die temperature of 250°C, providing an average grain size of 26µm (tested at ambient temperature).

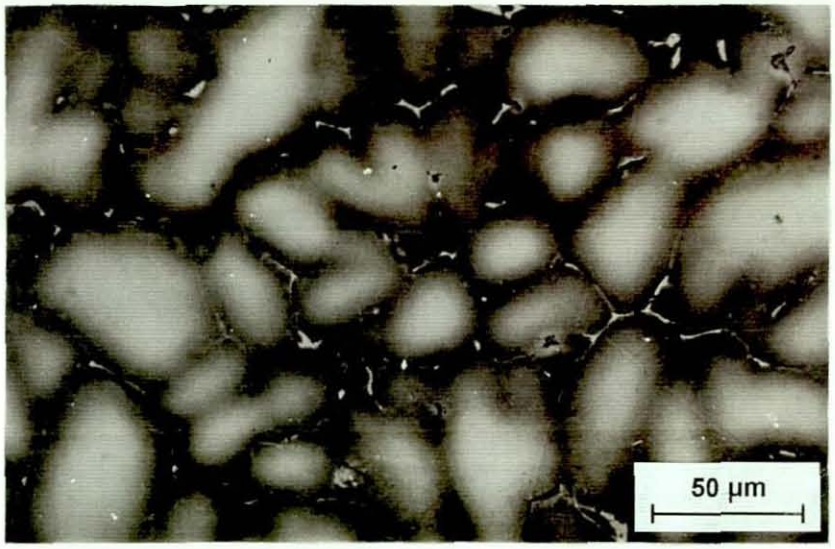


Figure 9-37 Optical microstructure of T5 treated squeeze cast RZ5DF alloy produced with pouring a temperature of 780°C and die temperature of 250°C, producing an average grain size of 28µm after exposure to 250°C (test at elevated temperature).

With reference to the as-cast microstructures in section 9.2.1.2, the microstructures of T5 treated RZ5DF alloy show much of the eutectic compounds dissolved into the grain by the heat treatment. There was also evidence of grain coarsening where grain diameters have increased from 18µm (figure 9-2) to 26µm (figure 9-36).

9.8.2 Results for the Heat Treated RZ5DF MMC Produced with Selected Conditions (Pouring Temperature 780°C and Die Temperature 250°C)

Heat treatment was carried out on the RZ5DF-14% vol. Saffil MMC in an attempt to further improve the optimum properties, obtained from the best combination of pouring and die temperatures identified in section 9.3. Composites that are heat treated undergo an accelerated ageing effect in relation to the parent alloys which constitute the composite. The presence of fibres made it necessary to determine the particular heat treatment durations that were required. This was an essential part of the research because the RZ5DF MMC is a new alloy. The determination of the heat treatment durations to maximise the mechanical properties of the RZ5DF-14% vol. Saffil MMC was carried out using a series of hardness curves which were constructed to display the ageing response of RZ5DF MMC to heat treatment. The effect on the hardness properties of the MMC when partially solution treated at a temperature of 330°C, in combination with age hardening at 180°C with varying durations, is presented in figure 9-38.

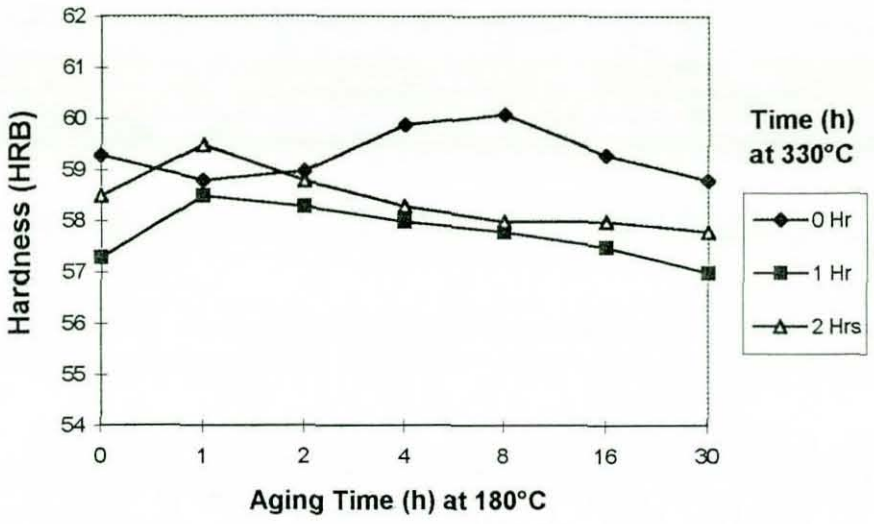


Figure 9-38 Ageing curves of RZ5DF-14% vol. Saffil MMC. Specimens were partially solution treated at 330°C for durations of 1 or 2 hours respectively, air cooled and age hardened at 180°C. Hardness results for specimens without solution treatment (0 hours) are also included.

Based on the evaluation of peak hardness values shown in figure 9-38, no or very minor influence of heat treatment on the hardness of test specimens can be identified. This was mainly due to the presence of fibres within the specimens, which dominates any change in matrix hardness due to heat treatment. It is widely known that the Saffil fibres of the MMC were much harder than the matrix and that the heat treatment process only affects the matrix.

In some instances, improvements in the hardness value of the MMC may be obtained, due to strengthening of the bond between fibres and matrix. However, the increase in the hardness of the matrix due to heat treatment, was generally insufficient to exceed that of the hardness provided by the fibre. The heat treated composites, therefore, do not appear to show any improvement nor response to the heat treatment as illustrated by the hardness measurements, shown in figure 9-38. Hardness tests only provided a partial view of the influence of heat treatment on the MMC and further tests were required. Therefore, tensile tests were conducted on specimens treated by nine different combinations of heat treatment conditions to further understand the influence on the properties of the RZ5DF MMC. Detailed tensile results for RZ5DF-14% vol. Saffil MMC, subjected to these heat treatment conditions, are presented in table 9 of appendix C.

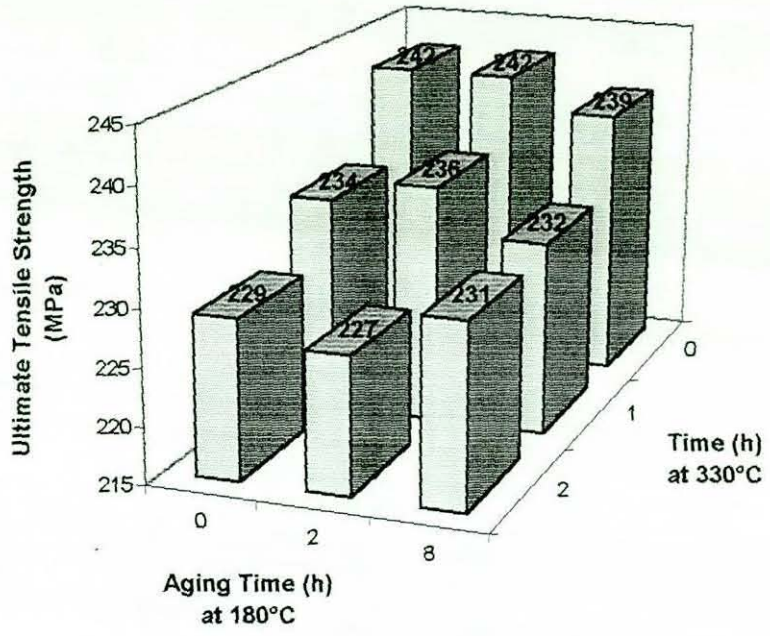


Figure 9-39 Tensile results for RZ5DF-14% vol. Saffil MMC. Specimens were partially solution treated at 330°C for 1 or 2 hours, air cooled and aged at 180°C.

The tensile plot in figure 9-39 shows that the casting exposed for a longer time exhibits a greater decrease in UTS values, i.e. exposing the tensile specimen at 330°C for a duration of 2 hours causes a significant decrease of 13 MPa. Conversely, heating the tensile specimen at 180°C for a duration of 8 hours causes a corresponding drop of only 3 MPa. This shows that any heat treatment resulted in a decrease in values for UTS of the MMC. The results also indicate the operating temperature (180°C) to which the MMC can be exposed to before a drop in UTS value was expected.

Microstructural evaluations on the test specimens were required to determine the cause(s) for the decrease in UTS values following heat treatment. The microstructures before and after heat treatment are shown in the following figures.

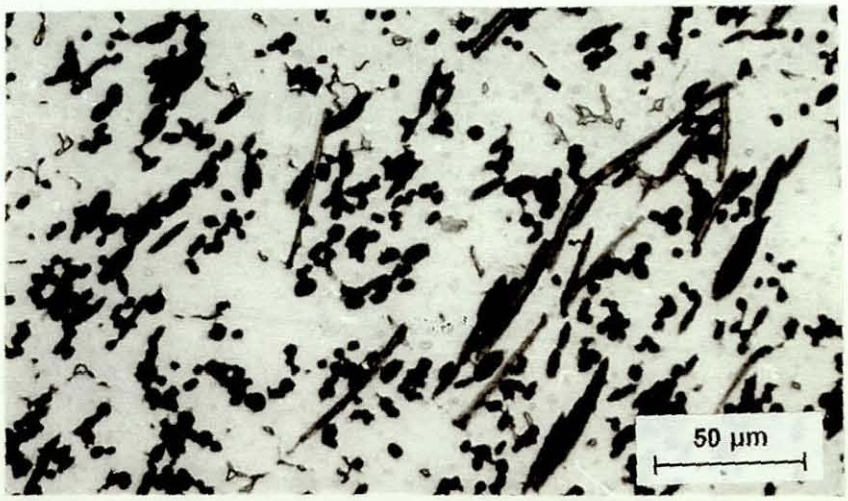


Figure 9-40 Optical microstructure of as-cast squeeze infiltrated RZ5DF-14% vol. Saffil MMC produced with pouring temperature of 780°C and die temperature of 250°C.

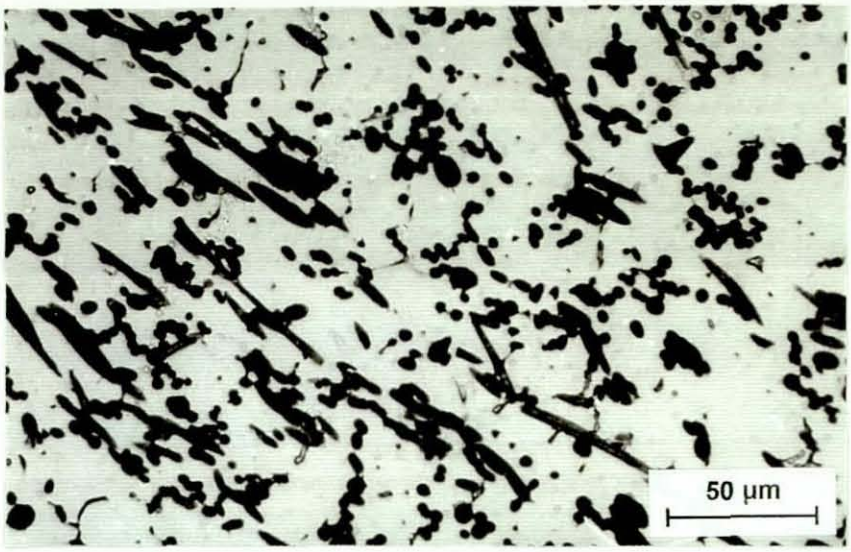


Figure 9-41 Optical microstructure of squeeze infiltrated RZ5DF-14% vol. Saffil MMC produced with pouring temperature of 780°C and die temperature of 250°C, after 8 hours of exposure at 180°C.

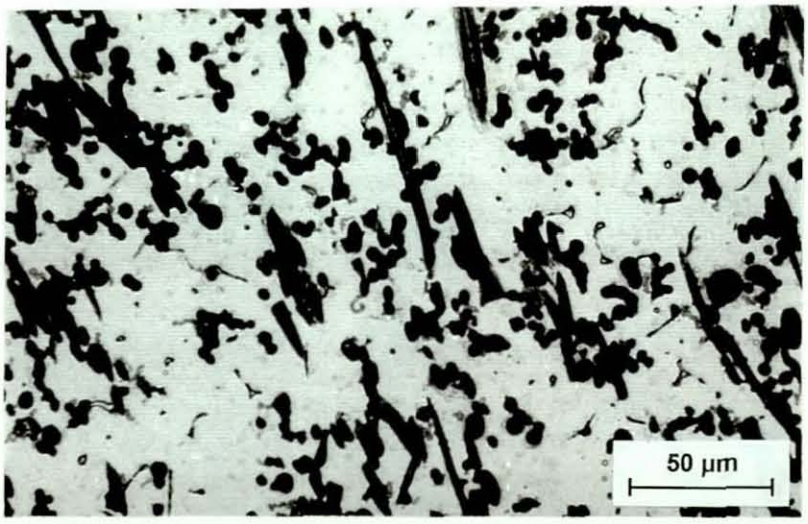


Figure 9-42 Optical microstructure of squeeze infiltrated RZ5DF-14% vol. Saffil MMC produced with pouring temperature of 780°C and die temperature of 250°C, after 2 hours of exposure at 330°C.

Figure 9-40 shows the as-cast microstructure in which there is a clean interface between the fibres and the matrix. Figures 9-41 and 9-42 show the effects of heat treatment involving temperatures of 180°C for 8 hours and 330°C for 2 hours respectively. A gradual deterioration in the quality of the bond at the interface can be seen, with the worst effect seen in figure 9-42.

9.8.3 Results for the Heat Treated RZ5 Alloy Produced with Selected Conditions (Pouring Temperature 750°C and Die Temperature 250°C)

Based on the recommendations of Magnesium Elektron Limited (MEL) [101] and the justifications which were presented in section 3.8.1, the precipitation treated (T5 heat treatment) condition was

selected to achieve optimum properties for the RZ5 alloy. The influence of T5 treatment on squeeze cast RZ5 alloy and the comparison between the properties of as-cast and T5 treated RZ5 alloy are presented in table 9-15. Detailed tensile properties of the heat treated RZ5 alloy are displayed in table 10 of appendix C.

	RZ5 alloy (as-cast)		**RZ5 alloy (T5 treated)**	
	Ambient Properties	Elevated Properties	Ambient Properties	Elevated Properties
UTS (MPa)	195 MPa	94 MPa	206 MPa	105 MPa
% Elongation	11 %	44 %	12.1 %	41 %
% Area Reduction	7.2 %	66 %	7.6 %	58 %
Young's Modulus	47 GPa	39 GPa	32 GPa	29 GPa
0.2% Proof Stress	90 MPa	43 MPa	105 MPa	70 MPa

Table 9-15 As-cast and T5 treated properties of squeeze cast RZ5 alloy.

Micro-examination had, therefore, to be conducted to obtain qualitative information focusing on the effects of heat treatment on the microstructure which in turn affects the mechanical properties. The micrographs for the T5 heat treated RZ5 alloy, subjected to tests at both ambient and elevated temperatures, are shown in figures 9-43 and 9-44 respectively.

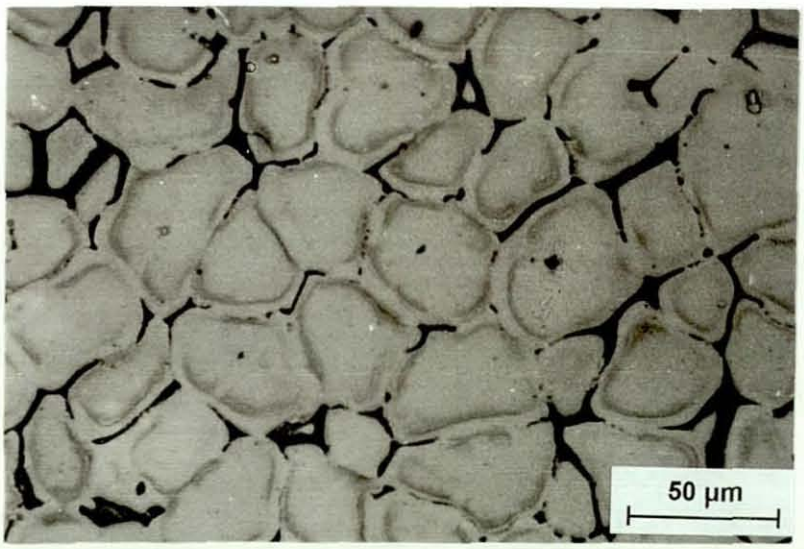


Figure 9-43 Optical microstructure of T5 treated squeeze cast RZ5 Mg alloy produced with pouring temperature of 750°C and die temperature of 250°C, providing an average grain size of 26µm (tested at ambient temperature).

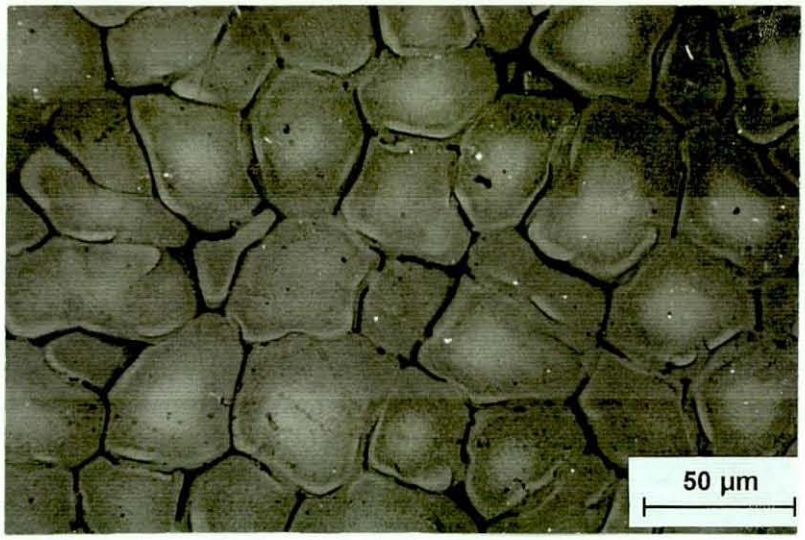


Figure 9-44 Optical microstructure of T5 treated squeeze cast RZ5 Mg alloy produced with pouring temperature of 750°C and die temperature of 250°C, producing an average grain size of 28µm after exposure to 250°C (test at elevated temperature).

With reference to the as-cast microstructures in sections 9.4.1.2 and 9.4.2.2, the microstructures of T5 treated RZ5 alloy show that much of the eutectic compounds were dissolved in the grain by the heat treatment. There was also evidence of grain coarsening, with diameters increasing from 21µm (figure 9-18) to 26µm (figure 9-43), and from 23µm (figure 9-22) to 28µm (figure 9-44) following exposure to 250°C.

9.8.4 Results for the Heat Treated RZ5 MMC Produced with Selected Conditions (Pouring Temperature 780°C and Die Temperature 250°C)

Similar to the RZ5DF MMC, the heat treatment durations to maximise the mechanical properties for the RZ5-14% vol. Saffil MMC using the best combination of pouring and die temperatures identified in section 9.4, were determined. The effect on the hardness properties of the MMC when partially solution treated at a temperature of 330°C, in combination with age hardening at 180°C with varying durations, is presented in figure 9-45.

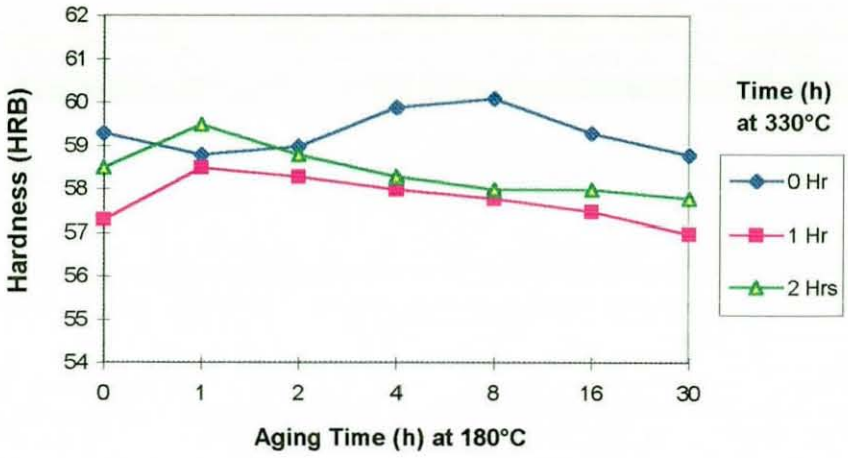


Figure 9-45 Ageing curves of RZ5-14% vol. Saffil MMC. Specimens were partially solution treated at 330°C for durations of 1 or 2 hours respectively, air cooled and age hardened at 180°C. Hardness results for specimens without solution treatment (0 hours) are also included.

Based on the evaluation of peak hardness values shown in figure 9-45, no or very minor influence of heat treatment on the hardness of test specimens can be identified. This again was attributed to the presence of fibres within the specimens, which dominates any change in matrix hardness due to heat treatment.

A similar result for the RZ5DF MMC was found, where the heat treated composites do not appear to show any response to the heat treatment as illustrated by the hardness measurements, shown in figure 9-45. Again, tensile tests were also required because hardness tests only provided a partial view of the influence of heat treatment on the MMC. Therefore, tensile tests were conducted for specimens treated by nine different combinations of heat treatment conditions to further understand the influence on the properties of the RZ5 MMC. Figure 9-46 displays the effects of various heat treatment durations on the UTS of the RZ5 MMC. Detailed tensile properties for RZ5-14% vol. Saffil MMC, subjected to different combinations of heat treatment durations, are presented in table 10 of appendix C.

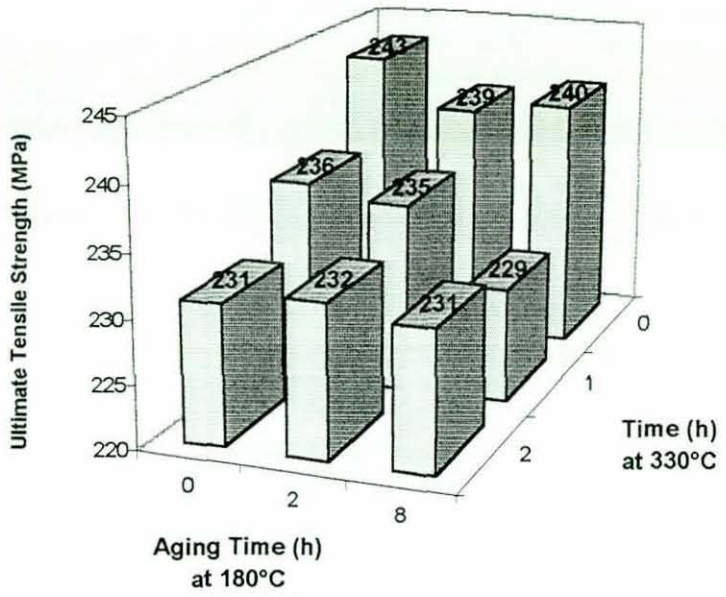


Figure 9-46 Tensile results for RZ5-14% vol. Saffil MMC. Specimens were treated at 330°C for durations of 1 or 2 hours respectively, air cooled and age hardened at 180°C. Tensile results for specimens without solution treatment (0 hours) are also included.

The tensile plot in figure 9-46 shows that the castings exposed to a higher temperature exhibit a greater decrease in UTS values, i.e. exposing the tensile specimen at 330°C for a duration of 2 hours caused a significant decrease of 12 MPa. Conversely, heating the tensile specimen at 180°C for a duration of 8 hours caused a corresponding drop of only 4 MPa. This again shows that any heat treatment resulted in a decrease in values for UTS of the MMC. The results also suggest that an operating temperature of 330°C should not be recommended for Mg-Zn MMC, as a significant decrease in UTS would be expected.

Microstructural evaluations on the test specimens were conducted to determine the cause(s) for the decrease in UTS values following heat treatment. The microstructures of specimens following age hardening at 180°C for a duration of 8 hours and partial solution treatment at 330°C for a duration of 2 hours, are shown in the figures 9-47 and 9-48 respectively.

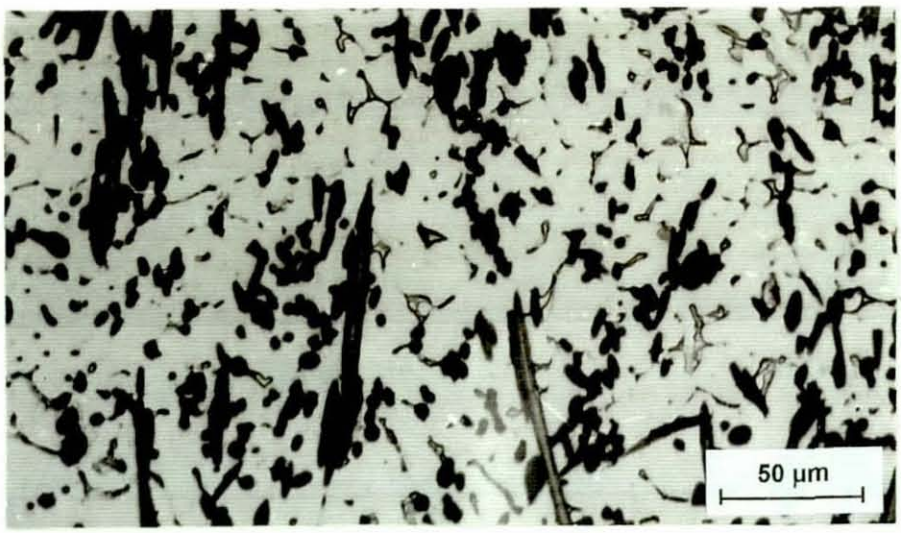


Figure 9-47 Optical microstructure of squeeze infiltrated RZ5-14% vol. Saffil MMC produced with pouring temperature of 780°C and die temperature of 250°C, after 8 hours of exposure at 180°C.

Figure 9-48 Optical microstructure of squeeze infiltrated RZ5-14% vol. Saffil MMC produced with pouring temperature of 780°C and die temperature of 250°C, after 2 hours of exposure at 330°C.

Figures 9-47 and 9-48 show the effects of heat treatment involving temperatures of 180°C for 8 hours and 330°C for 2 hours respectively. A gradual deterioration in the quality of the bond at the interface can be seen, where the worst effect can be seen in figure 9-48.

9.9 Summary

The experimental results obtained from different pouring and die temperatures used in the secondary casting programme have been presented in this chapter. This has included the UTS values, % elongation, % area reduction and metallographical studies for the four different materials (alloys and composites) used in this research. The selection of optimum casting conditions using a system of comparative weighting has also been explained. The results obtained from subsequent heat treatment tests have also been included. The following chapter will now discuss, in detail, the observations made from the experimental work.

Chapter 10

Discussion

10.1 Introduction

This chapter discusses the observations and results presented on the primary and secondary casting programmes. The influence of the different processes on mechanical properties is presented and the investigation of the different preform systems, preform temperatures, applied pressures and the duration of applied pressure is discussed. The effect of fibre addition on tensile properties is highlighted. The distribution of alloying elements in both alloys and composites is analysed. The effects of process parameters, grain refinement additions and heat treatment are discussed. Finally, a summary of the experimental results and a list of the research contributions are presented.

10.2 The Comparison of Three Casting Processes

A comparison was carried out on three casting processes, namely, gravity die casting, squeeze casting and squeeze infiltration. The basis of comparison was the mechanical properties, material hardness and microstructure of RZ5DF alloy and its composite. This comparison was presented in section 8.2.

A comparison involving squeeze casting and gravity die casting was first carried out to evaluate the influence of zinc and zirconium on the strength of material produced by the two processes. A comparison between Mg-2.5%Zn-RE and Mg-4.2%Zn-RE (RZ5DF) shows that a higher zinc content (Mg-4.2%Zn-RE) lowers the UTS values of the gravity die cast material by an average of 3 MPa. A higher level of zinc, in an ideal situation, should lead to a corresponding increase in tensile strength. However, the increased level of zinc was found to both increase the grain size and the tendency for microporosity which lowered the tensile properties (section 8.2). This increase in grain size is known to decrease the tensile properties of magnesium alloys when tested at ambient temperatures [57]. As a result of these two factors, the binary Mg-Zn alloy has been of limited use when produced using the majority of casting processes. An exception may be found in the case of squeeze casting, which has the advantage of eliminating microporosity, due mainly to the effect of solidification under pressure. Therefore, it has been found that optimum mechanical properties can be attained with a good combination of alloying and process selection. Conversely, the absence of pressure assisted solidification may explain why such a large variation in UTS (9 MPa) was produced for the gravity die cast Mg-4.2%Zn-RE (RZ5DF) alloy. This also accounts for the improvement in tensile properties for the squeeze cast specimens and vice versa for the gravity die casting process, as illustrated in figure 8-1.

A comparison was also made between RZ5DF (Mg-4.2%Zn-RE) and RZ5 (Mg-4.2%Zn-RE-Zr) alloy to study the influence of zirconium addition. In general, the results showed an improvement of as-cast UTS with zirconium addition. The most significant was a 13% increase in strength observed for gravity die cast specimens, in comparison to the 2% increase in strength for squeeze cast specimens. The improvement of strength in gravity die casting is attributed to the grain refinement of the casting, which is promoted through precipitation of small zirconium particles. However there is minor improvement in the strength of alloy containing 0.6 to 0.7% zirconium in squeeze cast components. This can be attributed to the presence of zirconium which has reduced the amount of Mg-Zn compound in the grain boundaries, so that more zinc is more usefully employed in the grain by the formation of the Zn-Zr intermetallic phase.

It was found that fibre reinforcement improved the tensile properties by 102% over the values produced by gravity die casting (figure 8-2). This illustrates the significant improvement which can be obtained by introducing high strength fibres as reinforcement elements within the magnesium matrix.

The hardness values of the cast specimens produced from the three casting processes were shown in figures 8-3 and 8-4. The hardness of the MMC specimens (average value of 61 HRB) was superior to the squeeze cast (average value of 19 HRB) and gravity die cast specimens (average value of 15 HRB). It is interesting to note that the hardness values for both the squeeze cast (alloy) and squeeze infiltrated (MMC) specimens were more consistent (variation of only 3 HRB) than those for the gravity die cast specimens (variation of 9 HRB). This was mainly due to the rapid solidification and constant application of pressure throughout solidification in the squeeze casting process which produced the fine, uniform and pore-free structures shown in figure 8-5. Conversely, the slow solidification rate associated with gravity die casting (figure 8-6) produced variable grain sizes and a coarse structure, resulting in the variation in hardness values in the longitudinal direction of the cast specimens.

The hardness values for all the specimens cast using different processes generally showed relatively consistent values in the transverse direction, with a variation of ±1.5 HRB (figure 8-4). This may be explained by the way the material solidifies in the transverse direction, i.e. the distance across the transverse direction is too short to cause any significant difference in the rate of metal solidification.

The surface quality of the squeeze infiltration process was comparable to conventional squeeze casting and it was superior to gravity die casting, as was shown in figure 8-7. The difference in component surface quality was due mainly to the pressurised solidification of the squeeze casting and squeeze infiltration processes, which ensured a particularly faithful reproduction of the surface of the die.

10.3 The Evaluation of a Suitable Preform System for RZ5DF Alloy

In this section the findings relating to the selection of a suitable preform system for RZ5SF alloy will be discussed.

10.3.1 The Quality and Effects of Squeeze Infiltration with Different Preform Systems

The quality of fibre preforms was investigated to ensure sound preforms were used for squeeze infiltration with the RZ5DF alloy. The examination revealed no or very minimal fibre fracture. This eliminated poor quality preforms (due to fibre fracture) as the possible cause for reduction in material strength.

The effects of squeeze infiltration on different preform systems was studied (figures 8-13 to 8-22). The cast surface and sectional view of preforms containing (i) 14% vol. Saffil with silica binder, (ii) 20% vol. Saffil with silica binder and (iii) 12% vol. Saffil + 9% vol. carbon fibres with alumina-silica binder reveal full infiltration. However, preforms containing (iv) 20% vol. Saffil with alumina binder and (v) 20% vol. carbon with alumina silica binder were found to be only partially infiltrated. The incomplete infiltration of the preform system (iv) was attributed to the low permeability and poor rigidity of the preform, which resulted in deformation during infiltration. For preform system (v), the main cause of incomplete infiltration was poor wetting between the carbon fibres and magnesium alloy. This may be attributed to the fact that carbon and magnesium are thermodynamically stable [18]. Even though pressure assisted infiltration is known to compensate for poor wettability between the reinforcements and the liquid metal, the inert nature of both materials has made it unlikely that sufficient bonds have formed at the fibre-matrix interfaces to enable transfer of load by shear forces.

10.3.2 The Comparison of Tensile Properties of the RZ5DF-MMC Produced with Different Preform Systems

A comparison of tensile properties of RZ5DF MMC composites produced with Saffil fibres was made (section 8.3.3). It was found that a higher UTS value (259 to 284 MPa) was obtained relative to composites containing carbon fibres (181 to 233 MPa). The lower UTS value for the carbon reinforced RZ5DF alloy is again attributed to the weak bond between the carbon fibres and the magnesium matrix.

The wettability of the carbon fibres plays a major role in the strength of the composite. Carbon fibres have poor wetting characteristics with magnesium which results in an increased resistance to infiltration. As a consequence, the high applied pressure of squeeze casting has caused the fracture of carbon fibres (figure 8-29). This has resulted in ineffective fibre reinforcement as the fibre lengths have fallen below the critical value, resulting in lower tensile properties.

10.3.3 The Comparison of the Hardness Properties of the RZ5DF MMC Produced with Different Preform Systems

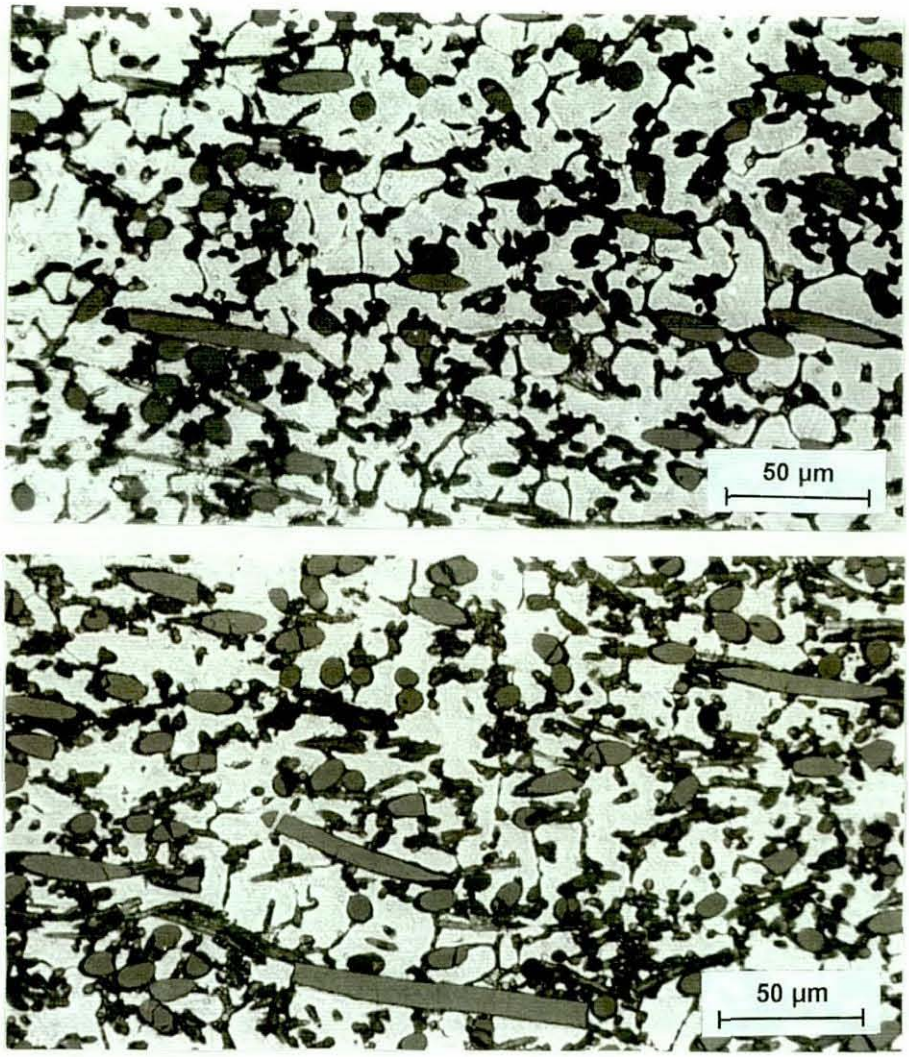
Hardness values for RZ5DF MMC composites produced with different preform systems were compared in section 8.3.4. Large differences were observed in the hardness values of the preform systems. For instance, the RZ5DF alloy reinforced with 20% vol. Saffil + alumina binder provided an average hardness value of 80 HRB, relative to 62 HRB for the RZ5DF alloy reinforced with 20% vol. carbon + alumino silicate binder. The factors that may have caused variations in hardness include:

Compatibility between process parameters and preform system. Incorrect selection of process parameters and/or preform system caused deformation of the fibre preform during infiltration. This deformation increased the fibre content in the reinforced region and this in turn increased the UTS and hardness properties of the cast MMC (by 25 MPa and 13 HRB respectively). The squeeze infiltrated RZ5DF alloy with 20% volume fraction Saffil fibres and a 5% alumina binder is an example of increased hardness caused by deformation of the fibre preform. A macrostructure illustrating this deformation was presented in figure 8-19.

Fibre Hardness. Since alumina is harder than carbon, it is reasonable to expect that castings of magnesium reinforced with alumina fibres will be much harder than those reinforced with carbon fibres. A comparison of the hardness values of RZ5DF alloy castings infiltrated with an equal volume of Saffil or carbon fibres showed that those reinforced with Saffil fibres were, on average, 5 HRB points harder than those reinforced with carbon fibres (section 8.3.4).

Fibre volume fraction. A higher fibre content increases the hardness of the RZ5DF alloy because more stiff and hard fibres are present in the composite. The hardness values of RZ5DF alloy infiltrated with different volumes of Saffil fibres were shown in figures 8-24 and 8-25. The results showed an increase of 6 HRB (on average) with an increase in fibre content from 14 to 20%.

Fibre Segregation. The RZ5DF-12% vol. Saffil and 9% vol. carbon composite had a higher hardness in the middle and top sections of the specimen (readings taken at locations: 3, 6, 9, 12 and 15), as illustrated in figure 8-24. This characteristic may be attributed to a pushing effect on the smaller diameter (e.g Saffil) fibres which forced them towards the bottom of the preform during infiltration. In the author's opinion, it was the combination of Saffil (smaller diameter) and carbon (larger diameter) fibres which allowed this pushing effect to occur. In effect, the smaller diameter Saffil fibres were pushed through the spaces between the larger carbon fibres. Evidence of the segregation of Saffil fibres to the lower section of the composite can be seen in figure 10-1.



(i)

(ii)

Figure 10-1 Micrograph of the RZ5DF 12% vol. Saffil + 9% vol. Carbon composite, from the (i) top half and (ii) bottom half of the composite

From figure 10-1, it can be seen that the micrograph taken from the top half of the composite has a lower concentration of smaller diameter Saffil fibres relative to the bottom half of the composite. In contrast, it can be seen from figure 10-1 (ii) that there is a greater concentration of smaller diameter Saffil fibres between the larger diameter fibres.

10.3.4 The Microstructure of RZ5DF MMC Produced with Different Preform Systems

The micro examinations of the MMCs produced with different preform systems revealed that Saffil preform systems showed sound cast structures, i.e. no shrinkage porosity (section 8.3.5). However evidence of fibre clustering was seen with a higher (20%) volume fraction of fibres, this may be attributed to the fact that denser preforms present greater resistance to full infiltration by the melt. The hybrid composite containing 12% vol. Saffil + 9% vol. carbon exhibited significant shrinkage porosity within the composite, which can be attributed to the low permeability of the hybrid preform (section 8.3.6). The magnesium-carbon reinforced composite exhibited significant fibre fracture throughout the composite structure (figure 8-29), which led to ineffective fibre reinforcement and the lower tensile

strength of 181 MPa. This is in comparison to the higher UTS value (266 MPa) of the RZ5DF alloy with 20% vol. Saffil + silica binder, a difference of 85 MPa.

10.3.5 The Comparison of Fibre Preform Permeability

The permeability studies, in section 8.3.6, showed that carbon fibre preforms had a higher permeability, mainly due to the larger fibre diameter (8μm) in comparison to Saffil fibres (3μm). This is similar to the permeability of bonded sand moulds, large sand grains have large pores between them thus allowing air to permeate more readily. Conversely, small grains (consider smaller fibres diameter in this investigation) have smaller pores or voids which creates a more difficult and tortuous route through which the air must pass.

Larger diameter fibres provide high permeability, but this is at the expense of resulting in a weaker MMC. There are two reasons for a weaker MMC with larger diameter fibres. Firstly, larger diameter fibres have a greater possibility of containing crystalline imperfections and this resulted in a weaker fibre element for the MMC. The significance of fibre diameter for tensile properties was described in section 4.4.3. Secondly, for a given fibre volume, a smaller diameter fibre has more surface area in contact with the matrix than a large diameter fibre. Based on the mechanics of reinforcement, the MMC strength is dependent on the fibre diameter and its surface area. The use of smaller diameter fibres, therefore, has contributed to the improvement of the MMC strength.

Hybrid preforms that contain two or more sizes of fibres, will have a different permeability value. A mixture of fibre sizes and distribution tends to result in the filling of voids between larger fibres (e.g. carbon) by the smaller fibres (e.g. Saffil), thus reducing the overall permeability. The low permeability number obtained in the study for the 12% Saffil + 9% carbon volume fraction fibre preform supports this argument (section 8.3.6).

10.4 The Evaluation of Suitable Preform Temperature for RZ5DF-14% vol. Saffil MMC

The influence of preform temperature on mechanical properties, material hardness and microstructure of squeeze infiltrated RZ5DF-14% vol. Saffil fibre preform will be discussed in this section. Different combinations of preform temperature and applied pressure caused variations in UTS and hardness values, as reported in section 8.4. The four preform temperatures were: 250°C, 400°C, 600°C and 750°C and the three applied pressures which were considered were: 60 MPa, 80 MPa and 100 MPa.

10.4.1 The Correlation Effects of Preform Temperature and Applied Pressure on Tensile Properties of Castings

Two main factors, namely preform temperature and applied pressure, influence the UTS values for the MMCs. The tensile strength of the castings produced from different combinations of preform and

applied pressure were presented in figure 8-32. The influence of these factors is now closely examined, in conjunction with hardness tests and microstructural examinations, to explain the variations in UTS. The individual cases are examined in the sequence of 750°C, 600°C, 400°C and 250°C as this will enable the UTS results to be contrasted on the basis of the consistency of the fibre distribution.

Preform temperature of 750°C. It was found that the highest preform temperature resulted in more consistent tensile properties (figure 8-32). The preform temperature of 750°C cast with the same alloy but different levels of applied pressure produced the most consistent UTS values, with a variation of only 3 MPa (range from 244 to 247 MPa). The higher preform temperature (above the liquidus temperature of the melt) allows the molten metal to be infiltrated into the preform with minimum restriction. Using a higher preform temperature allows a wider range of applied pressure to be used for infiltrating the melt into the preheated preform with minimum resistance to infiltration. It was also noted that there was less variation in fibre distribution.

Preform temperature of 600°C. This level of preform temperature produced a higher variation of UTS in comparison with 750°C. The highest UTS of 259 MPa was achieved with an applied pressure of 80 MPa. There are two principal reasons for the high UTS associated with this particular combination of preform temperature and applied pressure. Firstly, examination and comparison of microstructures and fracture surfaces of MMC samples taken throughout the casting revealed an even distribution of fibres, as illustrated in figure 8-40. Even distribution of fibres is necessary for better tensile properties, because the chances for physical contact between fibres which leads to stress concentrations and initiation points for failure are reduced. Secondly, the preform temperature of 600°C was approximately 33°C below the liquidus which resulted in the rapid solidification of the infiltrated molten magnesium. This produced a much finer grain structure, shown in figure 8-36, in comparison to the higher preform temperature, which in turn increases the mechanical properties of the MMC. A more detailed discussion of the distribution of the preform fibres in the microstructures is presented in section 10.4.3.

Preform temperature of 400°C. This case exhibits the highest variation in UTS when compared to the other preform temperatures, and clearly shows that too low a preform temperature will cause fibre clustering and preform deformation during squeeze infiltration. The clustering of fibres usually causes physical contact which results in stress concentrations and initiation points for crack propagation in brittle fibres. Examination of the fracture face, for the specimen produced at 80 MPa, (figure 8-39) revealed that fibres were in contact.

Preform deformation increases the fibre concentration in localised areas, and in particular in the central region of the casting. As the increase in fibre concentration was not accompanied by physical contact (or with very minimal contact) of the fibres, the tensile tests revealed a higher level of UTS

values for this particular casting. An increase in UTS due to increase in fibre content was illustrated by the example where too low a preform temperature of 400°C was used in conjunction with 60 MPa applied pressure. The microstructural examinations (figure 8-41) revealed a higher concentration of fibres in the central part of the infiltrated preform. The reasons for this were attributed to a combination of two factors. Firstly, the low preform temperature allowed rapid solidification of metal prior to the application of pressure at both the preform surface and locations near to the die wall. Secondly, the insufficient applied pressure during squeeze infiltration caused intermittent infiltration of the molten metal at the solidification front and further intensified the clustering and overlapping of fibres within the composite. Whilst these effects increased the tensile strength through the increase in fibre content in localised regions of the composite, this uncontrolled production of MMC was highly undesirable because of the inconsistent properties which resulted. The results indicated a difference of up to 23 MPa in the UTS value relative to those produced with 80 MPa and 100 MPa applied pressure respectively (figure 8-32).

Preform temperature of 250°C. The results indicated that the lowest preform temperature of 250°C cast with applied pressure of 80 MPa produced the lowest UTS (201 MPa). The low tensile properties can be attributed to a much greater clustering of fibres compared to the MMCs produced with a preform temperature of 400°C. As mentioned above, the clustering of fibres caused physical contact which resulted in stress concentrations and initiation points for failure, which eventually led to lower tensile properties.

10.4.2 The Correlation Effects of Preform Temperature and Applied Pressure on Hardness Properties of Casting

The comparison of hardness values of RZ5DF MMC produced with different preform temperature and applied pressure was reported in section 8.4.2. It was found that composites produced with the lowest preform temperature of 400°C and lowest applied pressure of 60 MPa produced a significant degree of variation in hardness value, with a range of ± 8 HRB in comparison to the usual ± 3 HRB observed in other combinations of preform temperature and applied pressure. The microstructural evaluation of this specimen revealed fibre concentration in the central region of the casting. This being the last area to solidify, the effects of fibres rejecting to that region and the intermittent infiltration promoted clustering in that region. Such agglomeration increased the measured UTS, as the tensile test specimens used in this research were machined from the central region of the casting. This explains the high UTS value of 253 MPa obtained with the low preform temperature of 400°C. The effects of such agglomeration are illustrated in figure 8-41. Therefore it is necessary to perform hardness tests in conjunction with the tensile tests to verify the distribution of fibres and properties throughout the cast structure.

10.4.3 The Microstructure of RZ5DF MMC Produced with Different Preform Temperature and Applied Pressure

The micrographs of the specimens squeeze infiltrated under 750°C and 600°C preform temperature generally show less densely packed fibres at the surface of the preform (figures 8-35 and 8-36 respectively), as compared to those produced with a lower preform temperature of 400°C and 250°C (figures 8-37 and 8-38 respectively). The lower preform temperature resulted in fibre clustering during infiltration, such clusters are associated with physical contact between fibres which resulted in stress concentrations and initiation points for failure, leading in turn to lower tensile properties. The presence of preform deformation and fibre clustering became less evident as the preform temperature was increased; this is seen through the gradual reduction of fibre clusters at the preform surface for preform temperatures equal to and greater than 600°C, as shown in figures 8-35 and 8-36.

Examination of the fracture faces of the tensile test specimens squeeze infiltrated at the preform temperature of 400°C revealed fibre clustering (figure 8-39). Clustering resulted in reduction of tensile properties by 17 MPa (in comparison to those produced with a preform temperature of 600°C) and this may explain why most MMCs squeeze infiltrated with a 400°C preform temperature had much lower tensile properties (figure 8-32).

10.4.4 The Influence of Zinc on the Selection of Optimum Preform Temperature for Magnesium

A major part of this research was to establish the influence of the addition of zinc as an alloying element to strengthen magnesium alloys. Alloying magnesium with zinc, which is a lower melting point metal, produces a binary alloy (Mg-Zn) which has a low solidus temperature relative to its liquidus temperature. The low solidus temperature is pronounced in the magnesium alloys containing more than 3% zinc. This alloy is classified as a long freezing range (LFR) alloy and exhibits dispersed porosity when processed by most conventional casting processes resulting in undesirable casting quality.

However, the application of pressure and the resultant rapid solidification, characteristic of the squeeze casting process, eliminates dispersed porosity. Furthermore, a LFR alloy may have advantages for squeeze infiltration, as the extended presence of a liquid phase within the composite improves infiltration under pressure. The freezing ranges of the two research alloys: RZ5DF and RZ5 alloys measured from the cooling curves (section 7.5) are 159°C (633°C-474°C) and 127°C (639°C-512°C) respectively.

Zinc is therefore the most appropriate alloying addition for providing the required processing characteristics and its selection was based on the understanding that zinc contributed to the ease of infiltration. In this research, 600°C preform temperature may be seen to be the ideal for Mg-Zn base alloy; this may not necessarily apply for other alloys because the ideal preform temperature for each alloy is a function of its eutectic melting point. In the author's opinion, the zinc addition to magnesium not only provides the significant advantage it has for solidification, as mentioned above, but it also enables a lower preform temperature to be used due to its low eutectic temperature.

This may explain why most researchers working on squeeze infiltration have selected higher preform temperatures, i.e. 800°C to 1000°C [20][172][173]. Such temperatures would prevent solidification occurring whilst the alloy infiltrated the preform and this would avoid deformation of the preform during infiltration. Even though the use of a higher preform temperature may prevent the occurrence of the latter effect, it will not provide the highest mechanical properties. The results achieved in this investigation show that optimum properties were attained with a preform temperature that was slightly below the liquidus temperature of the alloy. The reasons were: the smaller grain attained with lower preform temperature; and the melt temperature that was sufficiently high to allow adequate metal infiltration to occur.

A comparison of the grain size of the magnesium composites, produced from different preform temperatures, revealed that an average grain size of 30μm was attained with 600°C preform temperature (figure 8-36). Grain sizes exceeding 50μm were produced at 250°C, 400°C and 750°C preform temperatures, as seen in figures 8-38, 8-37 and 8-35 respectively. The presence of such large grains in magnesium composites produced by a preform temperature of 750°C may be attributed to the slow solidification rate relative to the preform temperature of 600°C.

For lower preform temperatures of 250°C and 400°C, the metal infiltrating the preform forms a solidified layer around the fibres. This solidified layer consists of the primary phase in the alloy, which is the first to solidify. This reduces the amount of primary phase constituents in the melt as the remaining melt becomes richer in the low melting point eutectic. Although primary phase still forms by nucleation and growth in the inter-fibre regions, the number of grains formed is reduced and their size is greater.

The results showed that if the preform preheat temperatures were below the melt solidus, the occurrence of premature melt solidification during infiltration was inevitable and prevented infiltration, thus impairing the tensile properties of the material. Therefore, based on the research findings, the optimum UTS is achieved with preform temperatures lying between the liquidus (633°C) and solidus temperature (474°C) of the RZ5DF alloy.

10.5 The Evaluation of a Suitable Applied Pressure for the RZ5DF-14% vol. Saffil MMC

The influence of applied pressure between 0.1 MPa and 120 MPa on mechanical properties, material hardness and microstructure of squeeze cast RZ5DF alloy and squeeze infiltrated RZ5DF-14% vol. Saffil preform containing silica binder will be discussed in this section.

10.5.1 The Comparison of Tensile Properties of the RZ5DF Alloy Produced with Different Applied Pressures

The tensile properties of the cast RZ5DF alloy specimens produced at different applied pressures were shown in figure 8-42. The squeeze cast specimens produced with applied pressures of 60 MPa, 80 MPa and 100 MPa provided the highest UTS values of the seven applied pressures considered. Higher % elongation and % area reduction are associated with higher UTS values. The highest tensile properties were achieved with an applied pressure of 100 MPa, which produced an optimum UTS value of 174 MPa, 11.8% elongation and 8.2% area reduction. The lowest tensile properties were produced with the atmospheric pressure of 0.1 MPa applied in the gravity die casting process, these being UTS of 128 MPa, 4% elongation and 2.9% area reduction.

The relationship between applied pressure and cast properties for RZ5DF alloy was shown in figure 8-42 and the results indicated a significant increase in UTS values with increasing applied pressure. However, it can be seen that an increase in applied pressure beyond 60 MPa did not provide much improvement in tensile properties. The significant improvement in tensile properties of castings produced with 60 MPa applied pressure can be attributed to two factors, namely: (i) the reduction of voids and shrinkage porosity and (ii) the progressive reduction in grain size. This may be seen by comparing the UTS values associated with the microstructures produced with different applied pressures shown in figures 8-48 to 8-53. An average grain size of 127μm was attained at an applied pressure of 0.1 MPa (atmospheric pressure) and the grain size was reduced to 21μm when the applied pressure was increased to 60 MPa. This was a significant six fold reduction in grain size with a 60 MPa increase in applied pressure.

In contrast, the improvements in UTS values when the applied pressure was increased from 60 MPa to 100 MPa were marginal, producing only a 1.2% increase in value for UTS, shown in figure 8-42. This low improvement was also reflected in the microstructure of the castings shown in figures 8-51 to 8-53. Average grain sizes of 21μm and 19μm were attained with applied pressures of 60 MPa and 100 MPa respectively. The small reduction in grain size (by 2 μm) explains the insignificant improvement in UTS despite the 40 MPa increase in applied pressure. There was a 6% decrease in UTS at an applied pressure of 120 MPa, relative to the specimens produced with an applied pressure of 100 MPa.

The micro-examinations of the castings produced with an applied pressure of 120 MPa (figure 8-54) revealed significant variation in grain sizes throughout the cast structure, where a mixture of very fine and coarse grains in the range of 10μm and 100μm were seen. This variation in grain size was attributed to the non-homogenous solidification of the RZ5DF magnesium alloy at the high applied pressure. The causes for the occurrence of this phenomenon will be discussed in section 10.5.4. The presence of large and varying grain sizes may explain why a lower UTS was experienced for the squeeze cast RZ5DF alloy produced with an applied pressure of 120 MPa.

The relationship between applied pressure and tensile properties for the squeeze cast RZ5DF alloy is summarised in figure 10-2.

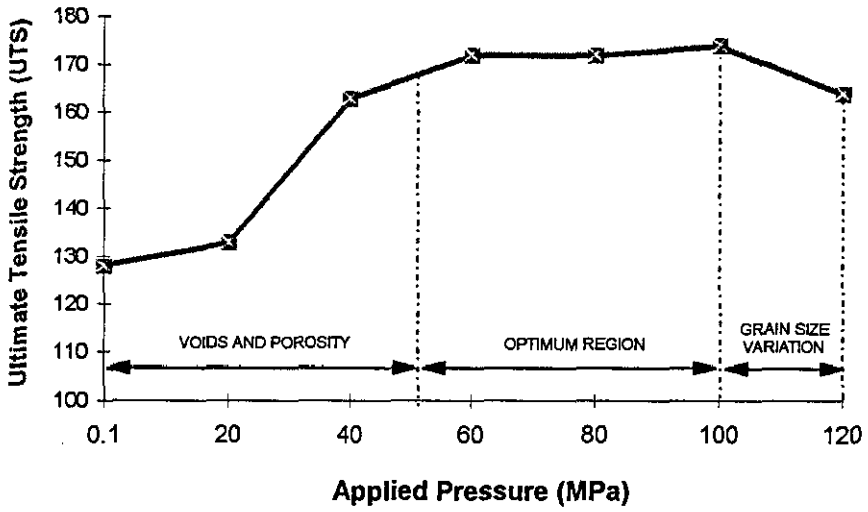


Figure 10-2 Effects of squeeze casting applied pressure on the tensile properties of RZ5DF alloy

With reference to figure 10-2, the low UTS values, at low applied pressure from 0.1 to 50 MPa, are caused by voids and shrinkage porosity present in the casting, due to the inadequate pressure on the molten metal to compensate for the porosity. Aided with the rapid cooling, finer grain sizes were formed with increased applied pressure and as a consequence higher tensile properties were attained. However, after a certain pressure level is reached (in this case 60 MPa), a further increase in applied pressure did not significantly improve the UTS. From the results, it is clear that optimum UTS values are attained with applied pressure greater than 50 MPa. At an applied pressure exceeding 100 MPa, grain size variation in the microstructure resulted in the decrease in UTS values.

10.5.2 The Comparison of Tensile Properties of the RZ5DF-14% vol. Saffil MMC Produced with Different Applied Pressures

The tensile properties of infiltrated RZ5DF-14% vol. Saffil fibre preform MMC produced with different applied pressures were shown in figure 8-43. The highest tensile properties were achieved

with an applied pressure of 80 MPa, and were a UTS of 259 MPa, 2.6% elongation and 2.4% area reduction. The lowest tensile properties were produced in the gravity die casting process with an atmospheric pressure of 0.1 MPa and were a UTS value of 176 MPa, 0.8% elongation and 0.6% area reduction. In this case a gravity die cast MMC was successfully produced with a 14% volume fraction fibre preform because the magnesium readily wets the fibres.

The results illustrated in figure 8-43 showed that the intermediate applied pressures of 60 MPa and 80 MPa provided the higher UTS values amongst the seven applied pressures considered. It was found that too high an applied pressure fractured the fibres within the preform. In contrast, low or insufficient pressure produced common castings defects such as porosity and shrinkage cavities, which invariably lowered the mechanical properties of the material. The effects of higher applied pressures, exceeding that of 80 MPa, increased the tendency for the clustering of fibres and the fracture of fibres (figure 8-60 and 8-61).

The relationship between applied pressure and tensile properties for squeeze infiltrated RZ5DF-14% vol. Saffil preform is summarised in figure 10-3.

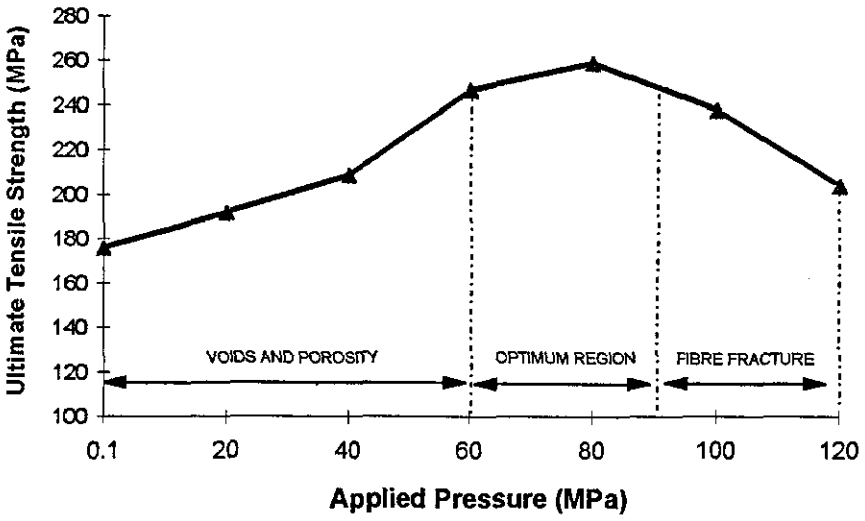


Figure 10-3 The effects of squeeze infiltration applied pressure on the tensile properties of the RZ5DF matrix with 14% volume fraction Saffil fibres

The relationship between applied pressure and UTS for the RZ5DF-14% vol. Saffil MMC casting displays a rather different pattern. This relationship may be separated into three main regions, namely: voids and porosity; optimum; and fibre fracture. The figure clearly shows that a critical pressure is still required to eliminate porosity. In the case of the MMC, an applied pressure of 60 MPa was required, this is 10 MPa more than that which was required for the alloy. The requirement for additional pressure is caused by the network of fibres within the preform that resist metal flow thus increasing the tendency for porosity. Increasing the applied pressure above 90 MPa dramatically

reduces the level of UTS and this deterioration is due mainly to the fracture of fibres through excessive pressure so that their length is below the critical fibre length. The importance of critical fibre length was highlighted in section 4.4.1.

The comparison of the influence of applied pressure on the mechanical properties of the squeeze cast RZ5DF alloy and the squeeze infiltrated RZ5DF alloy into 14% vol. Saffil preform indicated that the applied pressure level was more critical for squeeze infiltration, in comparison to the conventional squeeze casting process. The optimum region is narrowed significantly with the addition of fibres. This reduction in the optimum range can be seen by comparing figures 10-2 and 10-3.

These results may explain why other researchers have had difficulty in the adoption and use of the squeeze infiltration process based solely on the optimum process parameters achieved from the conventional squeeze casting method. It is clear, from this research, that there must be further consideration of additional process parameters such as preform temperature. Indeed, as discussed in section 10.4, the preform temperature has a significant influence on the tensile and hardness properties and also affects the ease of production of the cast components. This research has shown that a separate study for the optimum process parameters is required in the production of squeeze infiltrated MMCs and that they cannot simply be based on parameters obtained from conventional squeeze casting.

Furthermore, it has been shown that LFR alloys, that are a problem for most casting processes, may be suitable for the successful production of components using the squeeze infiltration process. This is due to the extended presence of a liquid phase within the composite, which improves the infiltration of the melt under pressure. The ease of infiltration, without an infiltration front to impede the progress of the liquid metal, results in less resistance to the passage of the metal through the fibre preform. A more even distribution of fibres is thus achieved, without clustering or deformation of the preform during the course of metal infiltration. The full contact of the liquid metal with the die walls causes rapid solidification of the metal, thus producing sound castings.

10.5.3 The Comparison of Hardness Properties of the RZ5DF Alloy and MMC Produced with Different Applied Pressures

Hardness values for the RZ5DF alloy produced with different applied pressures were compared in section 8.5.2.1. Minimal differences were observed in the hardness values resulting from different applied pressures, since the majority of the hardness values for the cast RZ5DF alloy fall within the range of 14 to 20 HRB. However, detailed examination of the castings produced with a lower applied pressure of 20 MPa and 40 MPa show that these have lower hardness values (figures 8-44 and 8-45). Low hardness was expected for these castings as they were specimens produced at a low applied pressure, contained voids and, therefore, have a lower hardness when compared to sound material. The

castings produced by gravity die casting exhibited a significant variation in hardness properties in particular along the longitudinal direction of the specimens. This variation was also noted in the earlier evaluation of the gravity die cast alloy specimens in section 8.2.

Hardness values for RZ5DF-14% vol. Saffil MMC produced with different applied pressures were compared in section 8.5.2.2. The results again suggested that specimens produced at low pressures contain voids and, as a consequence, have a lower hardness than the sound material. An example may be cited of the specimens produced with 0.1 MPa (figure 8-55) and 20 MPa (figure 8-56) applied pressures. The presence of voids and shrinkage was caused by a pressure that was inadequate for the full infiltration of molten magnesium into the porous fibre preform, thus resulting in lower hardness values.

10.5.4 The Microstructure of the RZ5DF Alloy and the RZ5DF MMC Produced with Different Applied Pressures

The micro examinations of the RZ5DF alloy produced with different applied pressures revealed that a critical pressure of 40 MPa was required to suppress microporosity (figure 8-50). The results indicated that grain sizes gradually decreased as the applied pressure increased. This reduction of grain size was attributed to the intimate contact between the melt and die wall which promotes rapid heat transfer, as applied pressure increases. However, as the applied pressure increased above 100 MPa there was an increased tendency for variation in grain sizes. This difference in grain size was observed throughout the cast structure of RZ5DF alloy produced under applied pressure of 120 MPa, where an example of one of the microstructures was shown in figure 8-54. This large variation of grain size was attributed to the non-homogenous solidification of the RZ5DF magnesium alloy at the high applied pressure. A restricted study was carried out to ascertain the causes of the large variation of grain size but, due to time constraints and lack of information in this area, only a few of the plausible causes for the variation of grain size at high applied pressure were identified. These are outlined as follows:

Firstly, according to the Clausius-Clapeyron equation (section 5.4.2.1), the application of pressure during solidification will increase the freezing temperature of magnesium under equilibrium conditions (slow cooling). As in most casting processes, the solidification process in squeeze casting is far from equilibrium. The rapid surface cooling in squeeze casting initially produces fine grains due to the high applied pressure. The remaining melt becomes richer in eutectic through segregation. According to the Clausius-Clapeyron equation, the high applied pressure will cause the melting temperature to rise and the remaining melt, which contains a higher eutectic content, will take longer to solidify and provide a longer period for grain growth. The combined effects of rapid surface layer solidification and an increase in freezing temperature due to higher applied pressure may explain the variation in grain size.

Secondly, the frictional forces between the grain clusters oppose the applied pressure. Therefore, excessive application of pressure diminishes the intended effect and causes an uneven level of pressure and non-homogenous solidification throughout the metal.

Lastly, the extremely high pressure applied throughout the solidification process may cause movement within the partially solidified liquid and physical deformation to the solid metal. This may be likened to a semi-solid forging process and may have resulted in the formation of non-uniform grain structures.

The applied pressure must not be excessive since it has a negative effect on the properties of the cast components. An applied pressure of 60 MPa was sufficient to provide the optimum tensile properties with the minimum of energy. Applied pressures of greater than 60 MPa are impractical for commercial applications as excessive applied pressure will not only be detrimental to the properties of the alloy but will also increase the cost of production. Although it is of academic interest to further investigate the effect of higher applied pressure, such studies are beyond the scope of this research.

The micro-examinations of the RZ5DF-14% vol. Saffil MMC produced with different applied pressure revealed that a critical pressure of 60 MPa was required to suppress any form of microporosity (figure 8-58). The results indicate that if the applied pressure exceeded 80 MPa there was an increased tendency of fibre fracture and clustering of fibres, especially for those specimens produced with an applied pressure of 100 MPa (figure 8-60) and 120 MPa (figure 8-61).

10.6 The Evaluation of the Effects of Applied Pressure Duration for the RZ5DF Alloy and RZ5DF MMC

The influence of the applied pressure duration for squeeze cast and squeeze infiltrated RZ5DF alloy and composite, presented in section 8.6, suggests the existence of a minimum applied pressure duration. The studies were carried out to determine the best applied pressure duration for the squeeze cast RZ5DF alloy and RZ5DF-14% vol. Saffil MMC. Three separate applied pressure durations, namely: 16, 25 and 35 seconds, were studied to determine their influence on the tensile properties.

The selection of an applied pressure duration of 16 seconds was based on a rule of thumb, which states that the maximum duration of pressure application should be 1 second per millimetre of casting section thickness. The casting section thickness used in this experiment was 16mm, therefore, the applied pressure duration of 16 seconds was selected as one of the parameter values. A subsequent comparison of the tensile properties of the specimens indicated that this rule of thumb was inappropriate. Rather, it was found that a minimum applied pressure duration of 25 seconds was necessary for both the RZ5DF alloy and RZ5DF MMC to allow complete solidification and optimum properties through the formation of fine and uniform cast structures.

However, the results presented in section 8.6 indicated that any further increase in pressure duration did not lead to further improvement in tensile properties. In fact, the tensile properties for RZ5DF alloy were marginally reduced. The RZ5DF alloy produced with 25 seconds of applied pressure produced a UTS value of 172 MPa, 11% elongation and area reduction of 7.2%. In contrast, the UTS was reduced to 170 MPa, elongation to 10.1% and area reduction to 6.7% when the applied pressure duration was increased to 35 seconds (figures 8-64 and 8-65). Despite the reduction of the tensile properties, the UTS variation between the specimens produced with 25 and 35 seconds applied pressure duration was marginal with a difference of only 2 MPa (approximately 1%).

In relation to the RZ5DF composite, the RZ5DF-14% vol. Saffil MMC produced with 25 seconds of applied pressure produced a UTS value of 259 MPa, elongation of 2.6% and area reduction of 2.4%. These values were reduced to 250 MPa, 2.4% and 2.1% respectively when the applied pressure duration was increased to 35 seconds (figures 8-66 and 8-67). Again the difference in tensile properties between the specimens produced with 25 and 35 seconds applied pressure duration was marginal, in particular the UTS values only experience a difference of only 9 MPa (approximately 3.6%).

The reason for the reduction in tensile properties for the alloy and MMC is not clear and may simply reflect experimental variations, especially as the difference in UTS values for both materials was minimal. From the point of view of economic production, longer time delays (beyond the minimum necessary duration), have little benefit and can, in fact, cause wall cracking and difficulty in punch retraction due to thermal contraction of the casting onto the rigid punch. Therefore a minimum applied pressure duration of 25 seconds, that ensured complete solidification, was selected.

10.7 The Evaluation of Alloying Distribution at Fibre-Matrix Interfaces and Grain Boundaries

The results of the analysis of the cast microstructure of RZ5DF and RZ5 alloys and their composites will be discussed, based on the SEM mapping analysis of the distribution of the alloying elements.

Zinc mapping analysis was conducted to assess the distribution of zinc in the cast RZ5DF alloy. The zinc mapping analysis of the gravity die cast microstructure showed the presence of a significant amount of zinc at the grain boundaries with a small amount of zinc within the grains (figure 8-68). In comparison, the zinc mapping analysis of the squeeze cast structures revealed that a greater amount of zinc was present within the grains (figure 8-71). Solution heat treatment was, therefore, not required for magnesium-zinc base alloys, since the zinc was well distributed within the grains. The distribution of zinc within the grains provides the strengthening mechanism for the alloy/grain. In particular, the zinc mapping analysis of the squeeze cast RZ5DF alloy indicated that a greater amount of zinc was present within the grains in comparison to the gravity die cast structures (figure 8-69).

Mapping analyses for both zinc and zirconium were also conducted for the squeeze cast RZ5 alloy, to assess the distribution of these elements in the cast material. The zinc mapping again showed a greater amount of zinc at the grain boundary in relation to the amount present within the grains (figure 8-76). However, there was a constant distribution of zinc within the grains which enhanced tensile properties. The zirconium mapping analysis indicated that the distribution of zirconium was primarily within the grain, with very little or no sign of zirconium present at the grain boundaries (figure 8-77). This indicated that the zirconium remained in solid solution following solidification. The distribution of zirconium had two main strengthening effects, namely: (i) it promoted the formation of nuclei and fine grains in the cast structure during solidification and (ii) the presence of high melting temperature solid (zirconium) within the grain provided a certain level of strengthening to the alloy.

Zinc mapping analysis was also conducted to assess the distribution of zinc in the squeeze infiltrated RZ5DF-14% vol. Saffil composite. This analysis showed that the majority of the zinc was present at the grain boundaries and around the fibres, with a certain amount of zinc present within the grains (figure 8-78).

10.8 The Evaluation of Squeeze Casting Consistency

The consistency of squeeze cast RZ5DF alloy and composite tensile properties and metallographic structures is discussed in this section. This includes the hardness test results taken along the longitudinal and transverse directions for all the castings produced during the primary casting programme.

10.8.1 The Consistency of Tensile Properties along the Transverse Direction of the Casting

Evaluation of the consistency of tensile properties along the transverse direction of the castings was conducted on: (i) the squeeze cast RZ5DF alloy, (ii) the squeeze infiltrated RZ5DF-14% vol. Saffil MMC and (iii) the squeeze infiltrated RZ5DF-20% vol. Saffil MMC. The tensile properties of the castings were summarised in figure 8-80.

The variations in % elongation and % area reduction for MMC specimens are within ±0.4% and are too small to suggest any correlation with UTS. Therefore, only the variations between UTS values will be discussed. The presence of fibres (14% and 20% vol. Saffil) significantly reduced the % elongation and % area reduction on RZ5DF alloy. The original 11.1% elongation and 7.8% area reduction obtained for the alloy was reduced by a factor of approximately six and four times to 2% and 2.2% respectively. However, the UTS values were improved from an average of 172 MPa for the RZ5DF alloy to 244 MPa and 266 MPa for the MMCs containing 14% and 20% vol. Saffil fibres respectively.

10.8.2 The Evaluation on the Summary of Hardness Values Measured during the Primary Casting Programme

The plots of hardness values determined along the longitudinal (figure 8-81) and transverse (figure 8-82) directions show two distinct zones, i.e. those for the RZ5DF alloy and those for the fibre reinforced material. Those with fibre reinforcement had significantly higher hardness values, with increases in the range of 280 to 600% relative to the alloy system. The average hardness values for RZ5DF alloy and its composite were 16 HRB and 60 HRB respectively. The results of all hardness values, and in particular those for the composites, show that a high fibre content provided the highest hardness value.

Specimens A and B were the two with the highest hardness values, as seen in figures 8-81 and 8-82. Specimen A, cast with 12% vol. Saffil + 9% vol. carbon preform system, contained the highest volume fraction of fibres among all the preforms investigated in the research. In relation to the preforms produced with 20% vol Saffil with alumina binder (specimen B), the increased hardness was due to the presence of a higher volume of fibres within the reinforced region, which was a result of preform deformation during squeeze infiltration process (figure 8-14). Conversely, low hardness values for the composite were achieved when a lower applied pressure, such as 0.1 MPa and 20 MPa, was selected as observed in specimens C and D, which were both cast with the 14% vol. Saffil preform system (section 8.8.2).

10.8.3 The Evaluation of Cast Microstructure Consistency

Microstructural examination of the squeeze cast RZ5DF alloy revealed a uniform grain structure throughout the cast specimen (figure 8-84). The applied pressure caused rapid solidification which in turn produced a uniform equiaxe cast structure. The importance of uniform fibre distribution on tensile properties was discussed in section 10.4.1. The microstructural examination of the squeeze infiltrated RZ5DF-14% vol. Saffil composite (figure 8-85), showed the fibre distribution throughout the casting to be relatively uniform. Hence, both microstructural studies of the cast structure confirmed the capability of the squeeze casting process to produce a fine, uniform grain structure and fibre distribution.

10.9 The Evaluation of Squeeze Cast RZ5DF Alloy

The results obtained from the squeeze cast RZ5DF alloy were consistent (section 9.2), where typical values for ambient temperature UTS were in the range of 169 to 198 MPa for various process settings. The values for % elongation and % area reduction were in the ranges of 8.3 to 12.8% and 5 to 7.7% respectively. The effects of pouring and die temperature on the ambient and elevated temperature properties for squeeze cast RZ5DF alloy are discussed in the following sections.

10.9.1 The Effects of Pouring Temperature for Squeeze Cast RZ5DF Alloy

The influence of pouring temperatures, at various die temperatures, on the UTS value are shown in figure 9-1. At the die temperature of 275°C, the higher pouring temperatures of 780°C and 750°C both produced UTS values of 184 MPa and the tensile specimens produced with 720°C provided a UTS value of 181 MPa. The variation produced by the three pouring temperatures was within a range of 3 MPa which showed that the pouring temperature had minimal effect on the UTS values for this die temperature.

At a die temperature of 250°C, a similar pattern of high and consistent UTS values was observed for the pouring temperatures of 780°C, 750°C and 720°C; these were 198 MPa, 191 MPa and 189 MPa respectively, i.e. a gradual decrease of UTS values with pouring temperature. Although the variation in UTS values was greater at 250°C die temperature, the difference was relatively small at 10 MPa between the highest and lowest UTS values.

However, the influence of pouring temperature on the level of UTS of the specimens produced was not as regular at the lower die temperature of 225°C. The lowest UTS value of 169 MPa was produced with the intermediate pouring temperature of 750°C, while UTS values of 171 MPa and 179 MPa were achieved with pouring temperatures of 780°C and 720°C respectively. Although the variations in UTS values were inconsistent, they were, again, within 10 MPa.

It was clear from the results, that the pouring temperature had a small influence on the UTS value. Although there was a variation of up to 10 MPa, this was minimal in comparison to that encountered when die temperature was changed. In order to explain the smaller effect of pouring temperature, it was necessary to consider the experimental results obtained when the die temperature was varied. The effects of the die temperature on the mechanical properties will be discussed in the next section.

10.9.2 The Effects of Die Temperature for Squeeze Cast RZ5DF Alloy

A steep temperature gradient, which is the consequence of combining a high pouring with a low die temperature, will yield a fine microstructure and result in higher mechanical properties [174]. Conversely, castings produced with a shallow temperature gradient are likely to have a large uniform grain structure, which will generally lead to lower mechanical properties. It is confirmed, from the literature, that a steep temperature gradient produces fine microstructures in castings [174][175][176]. In squeeze casting, pressurised solidification leads to high heat transfer due to the intimate contact between the die and solidifying metal. Therefore, as a consequence of this intimate contact between the large mass of die steel and solidifying metal, the dominating factor which controls the temperature gradient becomes the die temperature. A lower die temperature results in fine

microstructure producing higher mechanical properties and conversely, a high die temperature should result in coarse microstructures and lower tensile properties.

However, this combination of both fine microstructures and higher tensile properties was not observed for the lower die temperature of 225°C. The explanation for this was similar to the mechanism described in section 10.4.4, i.e. when infiltrating metal into preforms at the lower temperatures of 250°C and 400°C. In the case of lower die temperature, the molten metal was rapidly chilled and solidified nearest the die wall, forming a solidified layer of fine microstructure at the edge of the casting. Therefore, a low die temperature led to a non-uniform microstructure which was fine grained at the outer layer and coarse grained internally.

The micrograph of the squeeze cast RZ5DF alloy produced with pouring temperature of 780°C and die temperature of 225°C (i.e. highest pouring temperature and lowest die temperature which provided the extreme temperature gradient) taken at the edge of the casting is shown in figure 10-4. The micrograph illustrates the presence of a fine grain structure near the die wall and coarse grains which were found 0.1mm away from the edge of the casting. For comparison, a micrograph which was taken at the edge of the squeeze cast RZ5DF alloy produced with pouring temperature of 750°C and die temperature of 250°C (moderate temperature gradient) is shown in figure 10-5. The latter micrograph shows a minimal presence of fine grains at the edge of the casting.

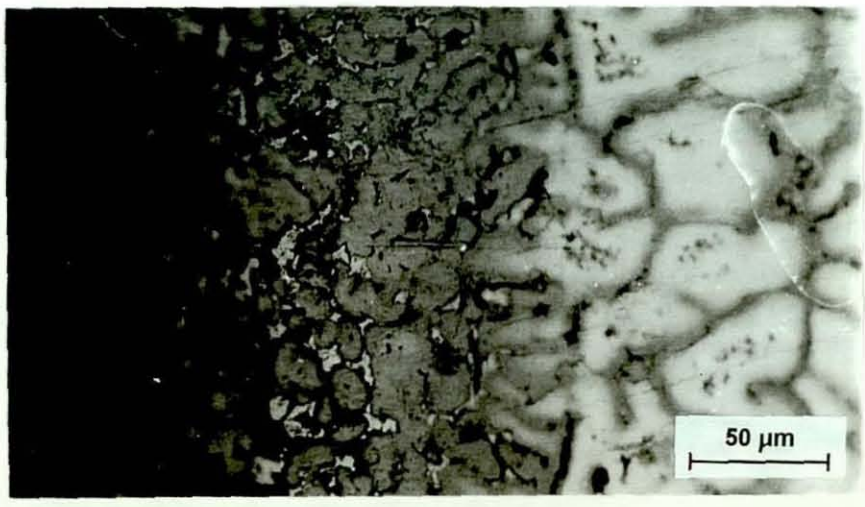


Figure 10-4 Microstructure of the squeeze cast RZ5DF alloy produced with pouring temperature of 780°C and die temperature of 225°C taken at the edge of the casting. The UTS value was 171 MPa.

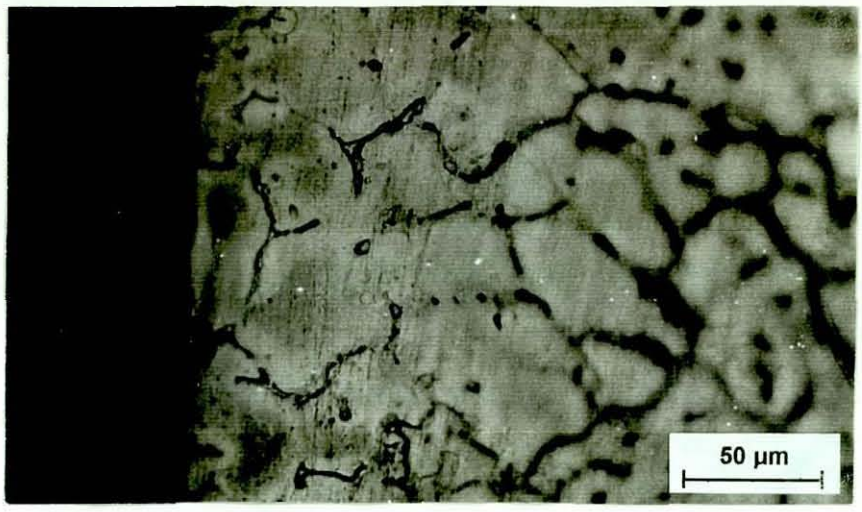


Figure 10-5 Microstructure of the squeeze cast RZ5DF alloy produced with pouring temperature of 750°C and die temperature of 250°C taken at the edge of the casting. The UTS value was 198 MPa.

At the higher die temperature of 250°C, the layer of chill grains was not present, as shown in figure 10-5. This seems to be the minimum (threshold) die temperature to achieve uniform solidification in the casting.

In relation to the die temperatures of 250°C and 275°C, castings produced with a die temperature of 250°C had the greater temperature gradient resulting in fine microstructure which gave the high tensile properties. The UTS values for the intermediate die temperature of 250°C with various pouring temperatures were found to be in the range of 189 to 198 MPa. The results, which were presented in figure 9-1, indicated that the highest temperature gradient for an intermediate die temperature of 250°C with the highest pouring temperature of 780°C produced the highest UTS value of 198 MPa.

The results presented in section 9.2.1, indicated that a high die temperature (275°C) reduced the rate of solidification which led to coarse microstructures. The UTS values for the highest die temperature of 275°C with various pouring temperatures were found to be within the range of 181 to 184 MPa. This will be particularly significant if constant mechanical properties are highly desirable in components, because variations in pouring temperature will not cause significant changes in the strength of the component.

10.9.3 The Effects of Pouring and Die Temperature on the Elevated Temperature Properties of RZ5DF Alloy

The elevated temperature UTS values obtained from the squeeze cast RZ5DF alloy ranged from 73 to 97 MPa and the values for % elongation and % area reduction were in the ranges of 36 to 50% and 36

to 65 % respectively (section 9.2.2). The UTS values of the RZ5DF alloy at elevated temperatures had decreased from a range of 169 - 198 MPa (figure 9-1) to 73 - 97 MPa (figure 9-5), i.e. the UTS values were approximately half of those at ambient temperature. This significant decrease in material strength at elevated temperature (250°C) is believed to be due to the increased occurrence of slip planes in the hexagonal crystal (HCP) structure of magnesium. It has been well documented that the deformation of magnesium above 225°C will cause additional slip planes $\{10\bar{1}1\}$ to become operative [B-3][B-23]. This marked increase in plasticity of magnesium, due to an increased number of slip planes, has limited the use of magnesium for higher temperature applications.

In contrast to the low UTS values achieved at ambient temperature, the lowest die temperature of 225°C for the RZ5DF alloy, produced with different pouring temperatures, provided the highest elevated temperature tensile properties (figure 9-5). The results, presented in section 9.2.2, show that the UTS values ranged from 85 to 97 MPa.

A comparison of specimen grain structures and their associated UTS values revealed that a difference of 2μm in grain sizes (figures 9-6 to 9-8) occurred between the high, intermediate and low UTS values. This difference was so insignificant that it could not be correlated with the UTS values. Nevertheless, it was anticipated that a finer grain size would result in lower tensile properties at elevated temperature [178].

10.10 The Evaluation of Squeeze Infiltrated RZ5DF MMC

In general, the results obtained for the squeeze infiltrated RZ5DF-14% vol. Saffil MMC cast with various combinations of die and pouring temperatures showed variations which fell within a narrow range of values for both the tensile properties and grain size (section 9.3). The effects of pouring and die temperature on the ambient and elevated tensile properties for squeeze infiltrated RZ5DF MMC are discussed in detail in the following sections.

10.10.1 The Effects of Pouring and Die Temperature on the Ambient Temperature Properties of RZ5DF MMC

The ambient UTS values of RZ5DF MMC, ranged between 229 MPa and 247 MPa. The effects of pouring and die temperatures on the tensile properties were found to be minimal, as the ambient UTS values only showed a difference of 18 MPa. The most consistent UTS values, which fell within the range of 235 to 237 MPa, were observed for castings produced with a pouring temperature of 750°C. This is of particular significance if consistent mechanical properties are desirable, since variations in die temperature will not cause significant changes to the strength of the component.

The highest UTS value of 247 MPa was achieved with the highest pouring and die temperatures of 780°C and 275°C respectively. This UTS value was attributed to the higher processing temperature which promoted wetting between the fibres and matrix. Furthermore, the higher temperature enabled greater ease of infiltration and minimised the occurrence of premature solidification. The improved infiltration resulted in less clustering of fibres and improved the tensile properties of the MMC.

Conversely, the highest temperature gradient, which was caused by the combination of the highest pouring temperature of 780°C and lowest die temperature of 225°C, produced the lowest UTS value of 229 MPa. This contrast between the pouring and die temperature produced the greatest temperature gradient and rapid solidification which induced stresses in the fibres.

The comparison of tensile properties between the RZ5DF alloy (section 9.2) and the RZ5DF MMC (section 9.3) showed a significant improvement in the Young's modulus associated with the addition of fibres. The increase was from an average of 43 GPa for the RZ5DF alloy (section 9.2.1) to 55 GPa for the MMC (section 9.3.1). This increase in modulus was attributed to the high modulus fibres present in the matrix. However, the higher UTS values for the MMC were accompanied by a corresponding reduction in values for the % elongation and % area reduction. The UTS value increased from 198 MPa for the RZ5DF alloy to 247 MPa for the MMC. However, the % elongation was reduced from an average of 10.5%, for the alloy, to 2% for the MMC and the corresponding % area reduction was decreased from an average of 6.4%, for the alloy, to 2.1% for the MMC. The reduction in ductility of the material was the result of stiff fibres being present in the MMC.

The microstructural examinations of the RZ5DF MMC specimens showed that UTS values were not a function of grain size. The observed differences in grain sizes associated with the highest, intermediate and lowest UTS values were minimal; the grain sizes measured for the specimens produced with the different combination of process temperatures fell within the range of 24 to 27μm (figures 9-10 to 9-12). The difference of 3μm is very small. The presence of fibres in the structure of the MMC made it difficult to ascertain the grain size accurately, as individual grain boundaries could not be easily identified from the micrographs.

It was found, however, that an even distribution of fibres influenced the material properties. For example, the RZ5DF MMC cast with a pouring temperature of 780°C and a die temperature of 275°C had the highest UTS value of 247 MPa. The corresponding microstructural examination, shown in figure 9-10, revealed an even distribution of fibres. In contrast, the specimen with a low UTS value of 229 MPa showed signs of fibre clustering and this gave rise to an uneven distribution of fibres in the cast structure, seen in figure 9-12. Again the clustering of fibres caused physical contact, which resulted in stress concentrations and initiation points for failure, reducing the tensile properties of the MMC.

10.10.2 The Effects of Pouring and Die Temperature on the Elevated Temperature Properties of RZ5DF MMC

The variations in pouring and die temperature resulted in similar trends in the elevated temperature properties for the RZ5DF MMC to those observed for tests at ambient temperature. It was found that an intermediate pouring temperature of 750°C in combination with various die temperatures produced consistent UTS values in the range of 143 to 145 MPa.

The higher UTS values of 147 MPa and 154 MPa were attained with a combination of the highest pouring temperature of 780°C, in conjunction with die temperatures of 250°C and 275°C respectively. The achievement of high UTS values was again attributed to the higher processing temperature which improved infiltration and promoted wetting between the fibres and matrix.

The stresses induced in the MMC due to a high temperature gradient reduce the properties of the material at both elevated and at ambient temperatures. Again, the highest temperature gradient, which was caused by the combination of the highest pouring temperature of 780°C and lowest die temperature of 225°C, produced the lowest UTS value of 133 MPa.

The evaluation of tensile properties at elevated temperatures carried out for the RZ5DF alloy (section 9.2) and RZ5DF MMC (section 9.3), showed that the addition of fibres provided the best improvements in properties. A 76% improvement in the UTS value was observed, where an average UTS value of 85 MPa for the RZ5DF alloy (section 9.2.2) was increased to 150 MPa for the MMC (section 9.3.2). This increase in UTS value was again attributed to the presence of fibres within the grains and at grain boundaries which impeded the progress of slip. This was particularly significant at the elevated temperature of 250°C since the deformation of magnesium above 225°C will cause additional slip planes to become operative. The presence of high strength fibres in the magnesium matrix will introduce obstructions that prevent the propagation of slip throughout the cast structure and thus increase the overall strength of the alloy at the elevated test temperature.

10.11 The Evaluation of Squeeze Cast RZ5 Alloy

The results obtained from the squeeze cast RZ5 alloy were consistent (section 9.4). Typical values for ambient temperature UTS were in the range of 171 to 195 MPa with the different process settings (as seen in figure 9-17). The values for % elongation and % area reduction were in the ranges of 8.5 to 11% and 5 to 7.2 % respectively. It can be seen that the tensile properties of squeeze cast RZ5 alloy were more consistent when compared to those of the squeeze cast RZ5DF alloy, in which the UTS ranged from 169 to 198 MPa (figure 9-1). The difference between the two materials was the presence of zirconium grain refiner in the RZ5 alloy.

The effects of the process variables (die and pouring temperature) and grain refinement on the mechanical properties for squeeze cast RZ5 alloy are discussed in the following sections.

10.11.1 The Effects of Pouring Temperature for Squeeze Cast RZ5 Alloy

No particular trends for the effect of pouring temperature were observed for these experiments. The reasons for the small effect of pouring temperature are similar to those discussed in section 10.9.2, i.e. the pressurised solidification of squeeze casting caused the effect of die temperature to be of significant influence to the temperature gradient, hence overshadowing the effect of pouring temperature. The only difference between the effects of pouring temperature for squeeze cast RZ5DF and RZ5 alloy was that the combination of a middle range pouring temperature of 750°C and a die temperature of 250°C produced the highest UTS value of 195 MPa. The die temperature was, again, identified to have the main effect on the increase in UTS and this will be discussed in the following section.

10.11.2 The Effects of Die Temperature for Squeeze Cast RZ5 Alloy

The effects of die temperature for squeeze cast RZ5 alloy were similar to those observed for RZ5DF alloy, indicating that a lower die temperature of 225°C produced the lowest UTS values which ranged between 171 MPa and 173 MPa. This relatively low strength was due to the non-uniform solidification which produced a coarse grain structure. A typical example, in which the non-uniform solidification resulted in the formation of fine grain structures at the outer layer and coarse grain structures at the inner layer of the casting, can be seen in figure 10-4.

The uniform solidification in the castings produced with die temperatures of 250°C and 275°C may again explain why higher UTS values in the range of 176 to 195 MPa were obtained, in relation to those tensile specimens produced with a die temperature of 225°C (figure 9-17). In relation to the die temperature of 250°C and 275°C, castings produced with a die temperature of 250°C had the greater temperature gradient resulting in fine microstructure which produced higher tensile properties. The UTS values for the intermediate die temperature of 250°C, with various pouring temperatures, were found to be in the range of 189 to 198 MPa (figure 9-17). The higher tensile properties were the result of a uniform solidification rate which leads to a uniformly fine microstructure.

The die temperature of 275°C produced lower UTS values as a result of the shallow temperature gradient during solidification that led to coarse microstructures, as seen in figure 9-19. The UTS values were in the range of 176 to 181 MPa.

10.11.3 The Effects of Pouring and Die Temperature on the Elevated Temperature Properties of RZ5 Alloy

The elevated temperature UTS values obtained from the squeeze cast RZ5 alloy ranged from 88 to 99 MPa, the values for % elongation and % area reduction were in the ranges of 34 to 46% and 41 to 66% respectively (section 9.4.2).

The UTS values of RZ5 alloy at elevated temperature decreased from a range of 171 - 195 MPa (figure 9-17) to 88 - 99 MPa (figure 9-21), i.e. the UTS values were approximately half of those at ambient temperature. The trend in these results is similar to that observed for the RZ5DF alloy and was also due to the increased occurrence of slip planes.

The results presented in section 9.4.2 indicated that the die temperature had the greatest influence on both the ambient and elevated temperature properties of the RZ5 alloy. In contrast to the properties achieved at ambient temperature, the lowest die temperature of 225°C used in combination with different pouring temperature provided the highest UTS values in a range of 98 to 99 MPa as shown in figure 9-21. The grain size resulting from the three die temperatures demonstrated a difference of only 1μm (figures 9-22 to 9-24). This difference was insignificant and could not be correlated with the UTS values.

10.11.4 The Effects of Grain Refinement (Zirconium) Addition

It was expected that the addition of zirconium would improve the mechanical properties through grain refinement. It has been reported that grain refinement is probably due to the precipitation of small zirconium particles which aid the formation of grains. When the number of nuclei is large, crystallisation proceeds from a large number of points. The presence of zirconium in magnesium produces a fine equiaxed grain structure with typical grain sizes of 30 to 50 μm in sand castings and this generally leads to higher mechanical properties in [70].

However, the values of the grain size obtained in this investigation were in the range of 18 to 32 μm, which is almost half that reported above. This may be due to the speed of solidification for a squeeze casting which is faster in comparison to that for sand castings. No grain size values were commercially available for gravity die castings, hence studies on the microstructures were carried out. Typical microstructures of squeeze cast and gravity die cast specimens can be seen in figures 8-48 and 8-51 respectively, where it may be seen that squeeze casting results in comparatively smaller grain sizes of 21μm compared with those of 127μm for gravity die casting. The larger grain sizes for gravity die castings may be attributed to two factors, namely: (i) the preheated die prevents the rapid solidification of the melt; (ii) the insulating die coating retards heat transfer from the die; and (iii) the formation of an air gap between the solidifying metal and casting slows the rate of solidification.

Differences in the grain shapes were observed between castings produced with and without the addition of grain refinement (zirconium). These observations were shown in figures 9-18 to 9-20 (with zirconium i.e. RZ5 alloy) and figures 9-2 to 9-4 (without zirconium i.e. RZ5DF alloy). It can be seen from the figures that the zirconium addition caused the individual grains to assume a more regular and rounded form.

Contrary to the anticipated effects of grain refinement addition reported in the literature [54] [55][57][70], the metallographic examinations did not show a significant difference in grain size. It was found that castings produced with the addition of grain refinement (RZ5 alloy) contained grain sizes which ranged from 21 to 26μm, while the castings without the addition of grain refinement (RZ5DF alloy) contained grain sizes ranging from 18 to 32μm. For the squeeze casting process, zirconium addition to the RZ5 alloy had no discernible effect (a difference of only 1μm) because the grain structure was refined by the process itself. Therefore, it can be concluded that the use of grain refinement in squeeze casting is unnecessary. This research has shown that the grain structure can be manipulated by process parameters such as applied pressure (section 8.5.3), pouring and die temperature (section 9.4.1.2) .

From the two evaluations (RZ5DF and RZ5 alloy), the grain refinement addition was shown to have minimum influence on the tensile properties, where the ambient UTS values for RZ5DF and RZ5 alloy were in the ranges of 169 to 198 MPa (figure 9-1) and 171 to 195 MPa (figure 9-17) respectively. Despite its minimal effect on grain size, the presence of zirconium produced improvements in elevated temperature properties. The elevated UTS values for RZ5DF and RZ5 alloy were in the range of 73 to 97 MPa (figure 9-5) and 88 to 99 MPa (figure 9-21). This was due to the presence of zirconium (a high melting point element) within the grains (seen in figure 8-77) which impeded the progress of slip at elevated temperatures.

10.12 The Evaluation of Squeeze Infiltrated RZ5 MMC

The results obtained from the squeeze infiltrated RZ5-14% vol. Saffil MMC cast with various combinations of die and pouring temperatures resulted in variations which fell within a narrow range of values for both the tensile properties and grain size (section 9.5). The effects of the process variables (die and pouring temperature) and addition of grain refinement on the ambient and elevated tensile properties for squeeze infiltrated RZ5 MMC are discussed in the following sections.

10.12.1 The Effects of Pouring and Die Temperature on the Ambient Temperature Properties of RZ5 MMC

The presence of fibres reduced the influence of pouring and die temperature on the tensile properties of RZ5 MMC, this is similar to the trend found for the RZ5DF MMC discussed in section 10.10. The

results obtained for the squeeze infiltrated RZ5-14% vol. Saffil MMC were consistent, where typical values for ambient temperature UTS obtained with the different process settings were in the range of 232 to 243 MPa (as seen in figure 9-25). The values for % elongation and % area reduction were in the range of 2 to 3% and 1.7 to 2.2% respectively. The most consistent UTS values for the RZ5 MMC, which fell within the range of 234 to 239 MPa, were observed for the casting produced with a pouring temperature of 750°C. Therefore, the trends for UTS, % elongation and % area reduction are similar to those found for the RZ5DF MMC.

The highest UTS value of 243 MPa was achieved with highest pouring temperature of 780°C and high die temperatures of 275°C and 250°C. This high UTS value was attributed to the higher processing temperature which promoted wetting between the fibres and matrix, and enabled greater ease of infiltration, the explanation for this phenomenon is similar to that observed for the RZ5DF MMC as discussed in section 10.10.1. Conversely, the lowest UTS value of 232 MPa was achieved with the highest pouring temperature of 750°C and the lowest die temperature of 225°C. This may again be attributed to the induced stresses in the fibres as a result of high temperature gradient.

A comparison made between the tensile properties of RZ5 alloy (section 9.4) and RZ5 MMC (section 9.5) revealed significant improvement in the UTS values and the Young's modulus associated with the addition of 14% vol. Saffil fibres. The increases were from an average of 183 MPa (RZ5 alloy) to 238 MPa for the UTS values and 40 GPa (RZ5 alloy) to 54 GPa (RZ5 MMC) for the modulus. This increase in both the UTS and modulus values was attributed to the introduction of high modulus fibres in the matrix. The effect of fibre addition on the ductility of the RZ5 alloy was similar to that for the RZ5DF alloy (as discussed in section 10.10.1), where the % elongation was reduced from an average of 9.8%, for the RZ5 alloy, to 2.5% for the RZ5 MMC and the corresponding % area reduction decreased from an average of 6.1%, for the alloy, to 2% for the MMC. The reduction in ductility of the RZ5 alloy was the result of the presence of stiff fibres in the MMC.

The microstructural examination of the RZ5 MMC specimens showed that the UTS values were not a function of grain size, rather the even distribution of fibres had greater influence over the material properties. For example, the highest UTS value of 243 MPa was associated with an average grain size of 24μm (figure 9-26), in comparison to an average grain size of 25μm observed for the RZ5 MMC specimens with the lowest corresponding UTS value of 232 MPa (figure 9-28). There was only a difference of 1μm in grain size. The microstructure shown in figure 9-26 (the highest UTS value), revealed an even distribution of fibres in comparison with that for the lower UTS value of 232 MPa, which showed signs of fibre clustering, seen in figure 9-28. The difference was that the specimens were cast using different die temperatures. This shows the importance of selecting the right process temperature in order to attain the optimum properties for the RZ5 MMC.

10.12.2 The Effects of Pouring and Die Temperature on the Elevated Temperature Properties of RZ5 MMC

The influence of pouring and die temperature on the tensile and microstructural properties at elevated temperatures were identical in trend to the ambient temperature properties reported in the previous section (10.12.1).

With reference to figure 9-29, it was found that the higher UTS values of 176 MPa and 174 MPa were attained with a combination of the highest pouring temperature of 780°C, in conjunction with die temperatures of 250°C and 275°C respectively. Again, the achievement of high UTS values was attributed to the higher processing temperature which improved ease of infiltration. This, in turn, reduced the possibility of fibre clustering, which was detrimental to the overall properties of the RZ5 MMC. Again, the intermediate pouring temperature of 750°C with various die temperature combinations produced consistent UTS values in the range of 167 to 170 MPa. The lowest UTS values of 159 MPa was again achieved with highest pouring temperature of 780°C and lowest die temperature of 225°C. This high temperature gradient induced stress in the MMC and reduced the properties of RZ5 MMC at both elevated and ambient temperatures.

A comparison made between the ambient and elevated tensile properties of RZ5 MMC, showed that the tensile properties of the MMC tested at ambient temperature had the least variation of 11 MPa (figure 9-25), in comparison to 17 MPa for the specimens examined at elevated temperatures (figure 9-29). This broader UTS range at elevated temperature was attributed to the thermal mismatch strains [87] arising when the MMCs were heated to a higher temperature, as there is large difference in the coefficient of thermal expansion between the matrix and the reinforcement (indicated in section 6.3.1 (iii)). The presence of fibre clustering (figure 9-32) for those MMCs produced with process parameters that were unsuitable, coupled with the additional problem of different thermal expansion, inevitability contributed to greater localised stresses in the casting which led to lower UTS values. This pronounced reduction in the UTS values for those MMC specimens with a certain degree of fibre clustering, explained why a broader UTS range was experienced for both RZ5 and RZ5DF MMC when tested at elevated temperatures.

A comparison made between the elevated tensile properties of RZ5 alloy (section 9.4.2) and RZ5 MMC (section 9.5.2), showed that the addition of fibres provided the best improvements in properties. A 79% improvement in the UTS value was observed, where an average UTS value of 84 MPa for the RZ5 alloy was increased to 168 MPa for the MMC. Again, this increase in UTS value was attributed to the presence of fibres within the grain and at the grain boundaries, which impeded the progress of slip. As discussed in section 10.9.3, the impeded process of slip was particularly significant at an elevated temperature of 250°C, as the deformation of magnesium above 225°C caused additional slip planes to become operative.

10.12.3 The Effects of Grain Refinement (Zirconium) Addition on Magnesium-Saffil Fibre Composite

Contrary to the anticipated effects of grain refinement addition reported in the literature [54] [55][57][70], the metallographic examinations did not show a significant difference in grain size. It was found that castings produced with the addition of grain refinement (RZ5 MMC) produced grain sizes which ranged from 24μm to 25μm (figures 9-26 to 9-28), while the castings without the addition of grain refinement (RZ5DF MMC) produced grain sizes ranging from 24μm to 27μm (figures 9-10 to 9-12). The relative insensitivity of grain size to the presence or absence of grain refining addition suggests that the higher UTS values of the grain refined material may be more a function of the role of zirconium as an alloying element rather than as a grain refiner.

Nevertheless, the studies on the effects of pouring and die temperature on the ambient and elevated temperature properties of RZ5 MMC (presence of zirconium) revealed that greater consistency in UTS was achieved in comparison to specimens of RZ5DF MMC that were free of zirconium. It was found that the UTS values of RZ5DF MMC, tested at ambient temperature, ranged from 229 to 247 MPa, while the RZ5 MMC had a smaller variation of UTS values that ranged from 232 to 243 MPa. Similar trends were observed for the elevated temperature properties of the RZ5DF and RZ5 MMC. The ranges of observed UTS values with different combinations of pouring and die temperatures were found to be minimal for RZ5 MMC, as the elevated UTS values only showed a difference of 17 MPa (figure 9-29), in comparison to 21 MPa for RZ5DF MMC (figure 9-13). The greater consistency in UTS that was achieved for the RZ5 MMC, may be attributed to the zirconium addition which caused the individual grains to assume a more regular and rounded form, a similar explanation was given in the earlier discussion made in section 10.11.4. The uniform grain sizes and form present in the ambient and elevated temperature specimens resulted in more consistent UTS values.

The most significant contribution of zirconium was the increase in elevated temperature properties for magnesium MMC. The explanation for this phenomenon is similar to that associated with the increase in UTS observed for the RZ5 alloy and discussed in section 10.11.4. This was due to the presence of zirconium within the grains (seen in figure 8-70) which impeded the progress of slip at elevated temperature.

10.13 The Evaluation of the Effects of Heat Treatment for RZ5DF Alloy, RZ5 Alloy and Its Composites

Prior to the heat treatment studies, the best casting parameters were determined for each of the four materials researched. The influence of heat treatment on the hardness and tensile properties of the RZ5DF alloy, the RZ5 alloy and its composites will be discussed in this section.

10.13.1 The Selection of Casting Parameters for the Improvement of Material Properties through Heat Treatment

Prior to the investigation of suitable heat treatment, the best pouring and die temperature combinations were established for each of the four research materials (section 9.7). It was necessary to achieve the optimum UTS values at ambient and elevated temperature first through the selection of an appropriate set of process parameters. When this was achieved, heat treatment was used to further improve the UTS values to maximise the potential of the material. A series of numerical calculations was used to determine the set of process parameters that produced the best UTS values for castings at ambient and elevated temperatures.

The comparative weightings for each material studied in the secondary casting programme were calculated and tabulated in tables 9-10 to 9-13 and revealed that:

(i) A pouring temperature of 780°C and die temperature of 250°C produced the best ambient and elevated tensile properties for the squeeze cast RZ5DF alloy (table 9-10).

(ii) A pouring temperature of 780°C and die temperature of 250°C produced the best ambient and elevated tensile properties for the squeeze infiltrated RZ5DF-14% vol. Saffil MMC (table 9-11).

(iii) A pouring temperature of 750°C and die temperature of 250°C produced the best ambient and elevated tensile properties for the squeeze cast RZ5 alloy (table 9-12).

(iv) A pouring temperature of 780°C and die temperature of 250°C produced the best ambient and elevated tensile properties for the squeeze infiltrated RZ5-14% vol. Saffil MMC (table 9-13).

Therefore, these sets of process parameters were subsequently used for heat treatment studies on each material involved in the secondary casting programme. From these tabulated results, castings produced with high pouring temperatures and intermediate die temperatures achieved the best combination of ambient and elevated tensile properties. Hence, a set of process parameters involving high pouring temperatures and intermediate die temperatures was used for obtaining the best results for both ambient and elevated temperatures for the four research materials.

10.13.2 Heat Treatment of Squeeze Cast RZ5DF and RZ5 Alloys

The results from the evaluation of the T5 treatment on squeeze cast RZ5DF and RZ5 alloy were presented in sections 9.8.1 and 9.8.3 respectively.

The RZ5DF alloy was a new alloy introduced in this research and no heat treatment information was available. Therefore a series of hardness curves and tensile plots was constructed to obtain the

influence of the partial solution treatment and the ageing response, in order to establish the best treatment condition(s) for improving the mechanical properties of the squeeze cast RZ5DF alloy.

From the establishment of the peak hardness curves (figure 9-34) and the tensile plots (figure 9-35) for the RZ5DF alloy, it was found that the T5 treatment provided the most promising improvements in the hardness value and strength. Ambient UTS values of 203 MPa, % elongation of 12% and % area reduction of 8% were reported (figure 9-35). Hence, T5 treatment was chosen for the evaluation of heat treatment on the ambient and elevated properties of the squeeze cast RZ5DF alloy. However, the results shown in table 9-14 only displayed a marginal improvement of the tensile properties of the squeeze cast RZ5DF alloy after T5 treatment. The ambient UTS values were only improved from 198 to 201 MPa, and the elevated UTS values were improved from 74 to 85 MPa. This minimal improvement of the tensile properties resulting from heat treatment may be attributed to the fact that the squeeze casting process had already maximised the properties of the RZ5DF alloy by refining the grain structure.

The artificial age hardening T5 treatment is well established commercially for the RZ5 alloy. Hence the T5 treatment was selected to evaluate the influence of its effect on the mechanical properties of the squeeze cast RZ5 alloy. Similar to the RZ5DF alloy, the improvement provided by the T5 treatment to the squeeze cast RZ5 alloy was marginal. The results from table 9-15, indicate that the ambient UTS value was improved from an average of 195 to 206 MPa, and the elevated UTS value was improved from 94 to 105 MPa. The explanation for this is similar to that given for the RZ5DF alloy, where the properties of the RZ5 alloy were already maximised by the rapid solidification associated with the squeeze casting process.

Therefore, as there is only minimal improvement provided by the T5 treatment on squeeze cast RZ5DF and RZ5 alloy, it will not be justifiable in terms of cost and time to introduce this treatment for the two cast alloys.

10.13.3 Heat Treatment of Squeeze Infiltrated Magnesium-Saffil Composite

The results from the evaluation of T5 treatment on squeeze infiltrated RZ5DF and RZ5 alloy were presented in sections 9.8.2 and 9.8.4 respectively.

The phenomenon of accelerated aging of MMCs, primarily due to the high dislocation density generated from thermal mismatch between the Al_2O_3 reinforcements and the matrix, has been reported [20][179]. The accelerated aging in the MMCs is due mainly to the presence of fibres which increase the dislocation density. A series of hardness curves (figures 9-38 and 9-45) and tensile plots (figures 9-39 and 9-46) were produced to evaluate the response of the RZ5DF and RZ5 MMC to the partial solution treatment and age hardening.

From the evaluation of peak hardness values shown in figure 9-38 and 9-45, no or very minor influence of heat treatment on the hardness of test specimens can be identified. It can be seen that the hardness for both the RZ5DF and RZ5 MMCs, following various durations of heat treatment, exhibited a difference of only ±1.5 HRB. This insignificant difference may be attributed to the presence of fibres (hard elements) within the specimens, which dominates any change in matrix hardness caused by heat treatment. As a result, the heat treated composites do not appear to show any improvement or response to heat treatment. Hence, tensile plots were constructed based on nine different combinations of heat treatment durations, as this would facilitate more direct studies of the influence of treatment on properties of the MMCs. The tensile plots, displayed in figures 9-39 and 9-46 for the RZ5DF and RZ5 MMCs, indicated that the heat treatment reduced strength. The greatest decrease in strength of 13 MPa was found when the tensile specimens were treated at a higher temperature of 330°C, and for a duration of 2 hours for both MMCs. A smaller decrease of 4 MPa was found when the tensile specimens were exposed at a lower temperature of 180°C, even at longer durations of 8 hours.

To determine the cause(s) for the decrease in UTS values following the heat treatment, microstructural evaluations were conducted. The detailed microstructural examination revealed the reasons for the deterioration of the mechanical properties of the MMC. As a consequence of the heat treatment, some reaction products (consisting of a brittle phase) were found at the fibre-matrix interface. These reaction products (shown in the figure as the white areas) can be seen clearly on the back scanner micrograph (figure 10-6). EDX analysis, presented in figure 10-7, revealed that the reaction products consisted of Mg, Si, Al, La, Ce and Zn. The main constituents were Mg and Si, which were likely to have originated from the Mg_2Si phase formed by a chemical reaction between the magnesium alloy and the silica (present in binder and fibres). This finding confirms that reported by Inem and Pollard [129].

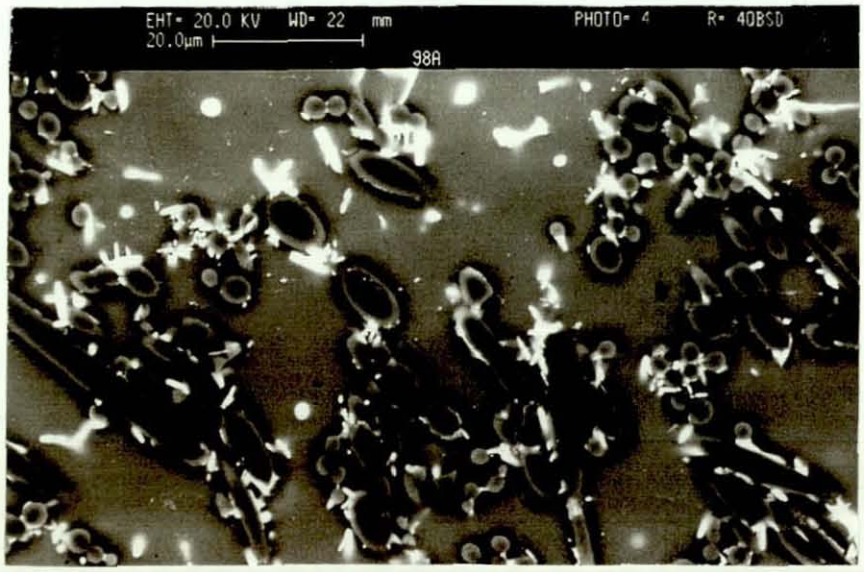


Figure 10-6 An example of a back scanner micrograph revealing reaction products at the fibre matrix interface after 2 hours of heat treatment at 330°C.

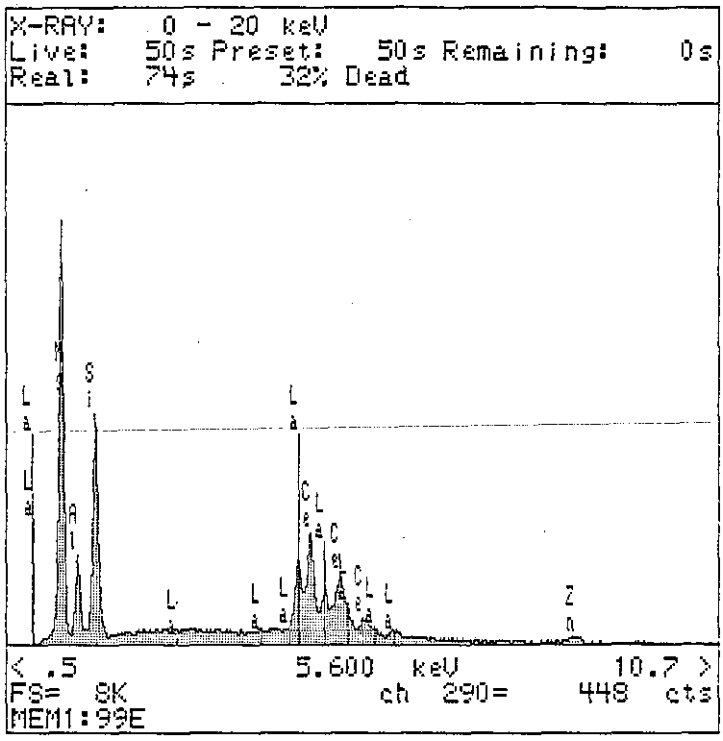


Figure 10-7 EDX analysis taken at the reaction product

This brittle phase was most evident following 2 hours of exposure at 330°C during the partial solution treatment. Magnesium is a highly reactive material at elevated temperatures and forms very strong bonds when used with Saffil fibres in a composite. However, the brittle phase caused by the heat treatment degraded the inherently strong bonds and weakened the interface between the matrix and fibres. In general, such weakening of the bonds at the interface will inevitably decrease the overall properties of the MMCs. Nevertheless the tensile plots (figures 9-39 and 9-46) indicated the potential for using as-cast RZ5DF and RZ5 MMCs at 180°C without causing significant decrease in the tensile properties.

10.14 A Summary of The Experimental Results

A summary of the results generated from the primary and secondary casting programmes is presented in table 10-1. Comparative evaluations were made of specimens produced by the different casting processes, in different materials and of their responses to heat treatment. The bases of these comparisons were the mechanical properties, material hardness and microstructure of the RZ5DF alloy, RZ5 alloy and their composites.

	Heat Treatment	UTS (Ambient)	% Elongation	% Area Reduction	Modulus	0.2% PS	Hardness (HRB)	Grain Size μm)
Gravity Die Casting RZ5DF alloy	as-cast	128 MPa	4 %	2.9 %	36 GPa	55 MPa	10 HRB	127 μm
	T5	140 MPa	4.7 %	3.2 %	29 GPa	57 MPa	8 HRB	131 μm
Gravity Die Casting RZ5 alloy	as-cast	145 MPa	5.3 %	3.8 %	30 GPa	56 MPa	11 HRB	93 μm
	T5	158 MPa	6 %	4.1 %	28 GPa	61 MPa	7 HRB	118 μm
Squeeze Cast RZ5DF alloy	as-cast	198 MPa	12.8 %	7.7 %	45 GPa	97 MPa	19 HRB	18 μm
	T5	201 MPa	13 %	8 %	37 GPa	93 MPa	16 HRB	26 μm
Squeeze Cast RZ5 alloy	as-cast	195 MPa	11 %	7.2 %	47 GPa	90 MPa	18 HRB	21 μm
	T5	206 MPa	12.1 %	7.6 %	32 GPa	105 MPa	14 HRB	26 μm
Sand Cast RZ5 [101]	T5	200 MPa	3 %	-	44 GPa	135 MPa	55-70 Brinell	50 μm
Squeeze Infiltrated RZ5DF-14% Saffil	as-cast	242 MPa	1.5 %	1.7 %	60 GPa	124 MPa	56 HRB	24 μm
	T5	231 MPa	2.6 %	2.8 %	-	-	54 HRB	-
Squeeze Infiltrated RZ5-14% Saffil	as-cast	243 MPa	3 %	2.2 %	60 GPa	130 MPa	59 HRB	24 μm
	T5	231 MPa	2 %	1.6 %	-	-	58 HRB	-

Table 10-1 The effect of different processes on the mechanical properties, hardness and microstructure of the materials

With reference to table 10-1, the UTS values were particularly low for the castings produced by the gravity die casting process, and this was attributed to the inherently low rate of solidification for the process. The lower rate of solidification was a result of the melt solidifying in a preheated die (250°C), which allowed the growth of grains. It was found that the average grain size for the gravity die cast RZ5DF alloy was 127μm, which is seven times larger than those produced by squeeze casting (18μm). The larger grain size resulted in a low UTS value of 128 MPa for the gravity die cast RZ5DF alloy.

In comparison to the results published for sand cast properties, the squeeze casting process was not able to provide much improvement in the UTS values. Nevertheless, one significant improvement for squeeze cast specimens was the increase in ductility (% elongation and % area reduction). These properties will be significant for components requiring a ductile failure mode in service, e.g. steering wheels and road wheels where high ductility values are essential to ensure safety during impact [180].

The tensile properties of MMCs revealed that the decrease in ductility, as discussed in section 10.12.1, was the result of the presence of stiff fibres in the matrix. However, this decrease in ductility may be viewed as a trade off with the increase of elevated temperature properties which enables the use of

magnesium for high temperature applications. Furthermore, it is expected that the wear resistance of the magnesium MMCs may increase their use in such applications as engine pistons, engine blocks and cylinder liners.

In general, it was found that squeeze casting properties did not improve with the T5 treatment in the same way that gravity die cast and sand cast specimens would. This may again be attributed to the fact that the squeeze casting process maximised the properties of the magnesium alloy by refining the grain structure, and any further improvement through heat treatment was not possible. Although squeeze castings generally cost more than gravity or sand castings, squeeze castings do not require the cost and time associated with the additional heat treatment process required to achieve improved properties. Furthermore, squeeze castings have better integrity, higher accuracy and dimensional consistency which provide opportunities to reduce inspection costs. Also, as a near net shape process, squeeze casting provides an excellent material yield due to the absence of a conventional runner and feeder system.

Finally, the results presented in section 9.6, relating to the evaluation of pouring and die temperature on casting dimension, indicated a variation of only ±0.03mm. This indicated that squeeze casting was able to provide repeatable, accurate casting dimensions. Nevertheless, the minimal variations found in the casting dimensions were most likely caused by the irregularity in the application of die coatings. Therefore the results showed that pouring and die temperatures had minimal effect on the casting dimensions and the variations found were most likely caused by the irregularity in the application of die coatings.

10.15 Contributions and Limitations of the Research

In general, the contribution of this research was the establishment of a clear understanding of a number of key parameters for the squeeze casting process, which enabled the production of high strength castings of magnesium alloys and composites. With reference to the objectives presented in section 2.6, the following list of contributions is presented:

(i) It was established that a long freezing range (LFR) alloy may be more suitable for the infiltration of a fibre preform in the production of an MMC. It was found that the infiltration of magnesium-zinc-rare earths alloy into the 14% vol. Saffil fibre preform was possible even under an atmospheric pressure of 0.1 MPa.

(ii) A preform system which promotes infiltration and maximum material properties, with the least fibre content and least cost of the magnesium MMC, has been established for the RZ5DF and RZ5 alloys.

(iii) An understanding has been gained about the key parameters influencing the mechanical properties of squeeze cast magnesium alloys and composites and this will be of significant benefit to the industry. For the commercial production of squeeze cast components, it may be confirmed that there exists an optimum applied pressure, and preform, pouring and die temperature ranges for maximum properties for the magnesium alloys and its composites to be achieved.

(iv) An opportunity to achieve a significant saving of material cost in squeeze casting has been found, as it has been shown that the addition of zirconium (grain refinement element) to the cast alloy does not achieve significant improvement in material properties.

(v) A better understanding of the behaviour of the squeeze cast material tested at both ambient and elevated temperature has been achieved. A methodology, which allows the identification of optimum squeeze casting conditions, has been developed. This has been successfully used in the identification of casting conditions which result in the best tensile properties operating at both test temperatures and there is potential for the greater uptake of this methodology.

(vi) The possibility for significant savings in cost and time, for commercial production, has been found as it has been shown that heat treatment was not required for the squeeze cast alloys and composites. Heat treatment does not result in much improvement to the tensile properties of the RZ5DF and RZ5 alloys. More importantly, it must be realised that the heat treatment process results in a deterioration of properties for the RZ5DF and RZ5 composites.

(viii) The fibre preform permeability test was justified as one mechanism to identify the ease of infiltrating the metal into the preform. Quality assessment of the preform prior to the actual casting has been found to be important, in particular, it is necessary to determine consistency of preform permeability for commercial production. Although this was not listed as an objective of this research, it has been found that there was the necessity for this, and for future research, to conduct preform permeability tests. This may be used as a simple quality control test in industry for the evaluation of preform consistency provided by the supplier(s).

(ix) It has been found that it is important, for commercial applications, to establish some control over the consistency with which die coatings are applied. Otherwise, it should be realised that the inconsistency of die coating thickness is a cause for dimensional variations.

This research has met the stated objectives and mainly focused on determining the immediate properties like tensile and hardness values. Nevertheless, studies on properties such as fatigue and creep behaviour will be of great interest in order to fully establish the performance of this material and this is a suggestion for further work which is presented in chapter 12.

Chapter 11

Conclusions

Based on the studies of process optimisation of squeeze cast magnesium-zinc-rare earth (Mg-Zn-RE) alloys and short fibre composites in this research, the following conclusions are drawn from the primary and secondary casting programmes.

11.1 The Primary Casting Programme

1. The tensile and hardness properties of the Mg-Zn-RE [2] alloys were significantly improved by the incorporation of fibres in the matrix. The tensile and hardness values were increased by a factor of two and three respectively, when compared with the gravity die cast alloy. The incorporation of fibres into the magnesium alloy, however, resulted in a significant decrease in the ductility (% elongation and % area reduction).

2. The as-cast tensile strength, ductility and Young's Modulus of the RZ5DF alloy were substantially superior for squeeze castings. At a squeeze pressure of 60 MPa, the values of UTS, ductility (% elongation) and Young's modulus are 55%, 220% and 25% higher than those for the gravity die alloy.

3. Mg-Zn-RE [2] alloys, which are long freezing range (LFR) alloys, were found to be suitable for the infiltration of the preform. The good wetting between the magnesium and the Saffil fibres resulted in the greater ease of metal infiltration into the preform.

4. Fibre preforms containing carbon fibres were found to be only partially infiltrated by the RZ5DF alloy. The incomplete infiltration of the preform was attributed to the poor wetting between the carbon fibres and magnesium, since both elements are known to be thermodynamically inert to each other. The ineffective fibre reinforcement resulted in lower tensile properties.

[2] **Mg-Zn-RE alloys** refers to both the RZ5DF and RZ5 alloys, both contain the basic element of magnesium, zinc and rare earths. The only different between the two is the presence of grain refinement element (zirconium) in the RZ5 alloy.

5. The preform system consisting of 14% volume fraction Saffil with silica binder was found to have the best value to strength ratio. The low volume fraction of fibres allowed greater ease of infiltration and the good wetting between the magnesium and the Saffil fibres reduced the risk of fibre clustering during squeeze infiltration.

6. A fibre preform permeability test was used to assess the suitability of preforms for infiltration. However, although there was a correlation between permeability and infiltration capability, this was not the only factor of importance. Of equal importance was the wetting characteristics of the preform and matrix combination.

7. A minimum applied pressure of 50 MPa is required to suppress microporosity in the RZ5DF alloy. In the case of the RZ5DF MMC, an applied pressure of 60 MPa was required. This was 10 MPa more than that required for the alloy. The requirement for additional pressure was caused by the network of fibres within the preform that resisted metal flow during infiltration.

8. Increasing the applied pressure beyond 60 MPa did not provide much improvement in the tensile properties for the squeeze cast RZ5DF alloy. The applied pressure of 60 MPa was sufficient to eliminate all traces of shrinkage and gas porosity within the casting. The micro-examinations of the castings with application of pressure greater than 60 MPa did not reveal significant decrease in grain size. A minimal decrease from 21μm to 19μm in grain size was found when the applied pressure of 60 MPa was increased to 100 MPa.

9. High applied pressure, of 100 MPa and above, causes the fracture of fibres during the casting stage for the squeeze infiltrated RZ5DF MMC. The fibre length is shortened by the fractures and this reduces the efficiency of load-transfer from the matrix to the fibres.

10. A moderate preform temperature of 600°C and applied pressure of 80 MPa was required to provide the best tensile properties for squeeze infiltrated RZ5DF-14% vol. Saffil MMC. Smaller grains were attained when the preform temperature was slightly below the liquidus temperature of the alloy and when the melt temperature was sufficiently high to ensure melt infiltration. This in turn, increases the mechanical properties of the MMC under a moderate applied pressure that is sufficient to eliminate microporosity in the casting.

11. A minimum applied pressure duration of 25 seconds was necessary for both the RZ5DF alloy and RZ5DF MMC to allow complete solidification and attain optimum properties through the formation of fine uniform cast structures.

11.2 The Secondary Casting Programme

1. A higher pouring temperature of 780°C and intermediate die temperature of 250°C provided the best combination of ambient and elevated tensile properties for RZ5DF alloy, RZ5DF MMC and RZ5 MMC. A moderate temperature gradient with intermediate pouring temperature of 750°C and die temperature of 250°C provided the best combination of ambient and elevated tensile properties for RZ5 alloy.

2. The highest pouring temperature of 780°C and lowest die temperature of 225°C, consistently produced the lowest UTS values tested at ambient temperature from amongst the RZ5DF alloy, RZ5 alloy, RZ5DF MMC and RZ5 MMC. The non-uniform microstructure of the alloys and the rapid solidification caused by the high cast temperature gradient induces stresses in the fibres present within the MMC.

3. The pressure applied in squeeze casting promoted rapid solidification and a refined grain structure. It is possible to achieve comparable tensile properties in the zirconium-free RZ5DF alloy to those of RZ5 alloy grain refined with a zirconium addition with appropriate processing parameters (pouring and die temperatures).

4. A zirconium addition improved the elevated temperature properties of Mg-Zn-RE [2] alloys. SEM zirconium mapping revealed the presence of the high melting point element within the grains and it is possible that this presence impeded the progress of slip at elevated temperatures.

5. The presence of fibres and zirconium in the Mg-Zn-RE [2] alloys reduced the variation of UTS values caused by different pouring and die temperatures. The variation of UTS values for RZ5DF and RZ5 alloy tested at ambient temperature were 29 MPa and 24 MPa respectively. In comparison to the RZ5DF and RZ5 MMCs variation of UTS values were in the range of 18 MPa and 11 MPa respectively.

6. The RZ5DF-14% vol. Saffil MMC tested at elevated temperature displayed a 76% improvement in the UTS value relative to its alloy. The presence of fibres within the grains and at grain boundaries is believed to impede the progress of slip at the elevated temperature of 250°C. The improvement in UTS may provide an opportunity for the light weight magnesium MMC to be used at elevated temperatures.

7. A T5 heat treatment has minimal effect on the tensile properties of squeeze cast Mg-Zn-RE [2] alloys. The squeeze casting process already maximises the properties by grain refinement through the application of direct pressure. Heat treatment was found to impair the properties of

Mg-Zn-RE [2] MMCs. The occurrence of brittle Mg_2Si phase (reaction product) at the fibre matrix interface during heat treatment degraded the inherently strong bonds and weakened the interface between the matrix and fibres.

8. SEM zinc mapping of the microstructure of squeeze cast RZ5DF alloy revealed that a greater amount of zinc is present within the grains compared to the gravity die cast structure. The solution treatment is not required for the squeeze cast alloy, as the zinc element used for strengthening the structure is well distributed within the grains.

9. The distribution of fibres in an MMC has the greatest effect on the tensile properties, where an even distribution of fibres in the MMC provides the highest UTS. The clustering of fibres increased the point of contact between fibres, which acted as stress concentration and initiation sites for crack propagation in fibres. The selection of the appropriate process parameters (i.e applied pressure and preform temperature) is essential to ensure that an even distribution of fibres is attained.

Chapter 12

Recommendations for Further Work

In completing this research, the author has identified a number of areas for further investigation.

1. In order to realise the full potential of squeeze cast magnesium matrix composites, it will be necessary to develop a stable binder system (i.e. a colloidal zirconia or magnesia binder) to reduce the formation of reaction products during heat treatment with magnesium matrix composites. A development in this direction will provide opportunities for improving the mechanical properties of magnesium MMCs through heat treatment. However, the binder which is developed must also enable the preform to retain its integrity and shape under the high infiltration pressure applied during the squeeze casting process.

2. This research showed that infiltration of the magnesium-zinc-based alloy under an atmospheric pressure of 0.1 MPa was possible. It would be interesting to evaluate the use of a low pressure casting process for the production of magnesium alloys and composites. Examples of low pressure casting processes are low pressure die casting and low pressure sand casting (i.e. the Cosworth process). The potential of using an enclosed environment, with a small positive pressure, will be appealing for the casting of magnesium, as it will reduce the formation of magnesium oxide which is detrimental to the overall properties of the casting. Furthermore the lack of turbulence during filling will avoid oxide formation and gas entrapment, leading to sound castings.

3. Until now, fibre reinforced materials have been studied with the main aim to improve strength. Detailed data on fracture toughness is not sufficient for the design of products for many practical applications. A series of studies on fatigue and creep properties are required because many components are subjected to fatigue and creep failure.

4. For greater acceptance of high performance MMCs as attractive materials for product designers, practical aspects other than those related to the production of the material, need to be researched. Secondary working, joining, recycling and cost are also subjects requiring further investigation.

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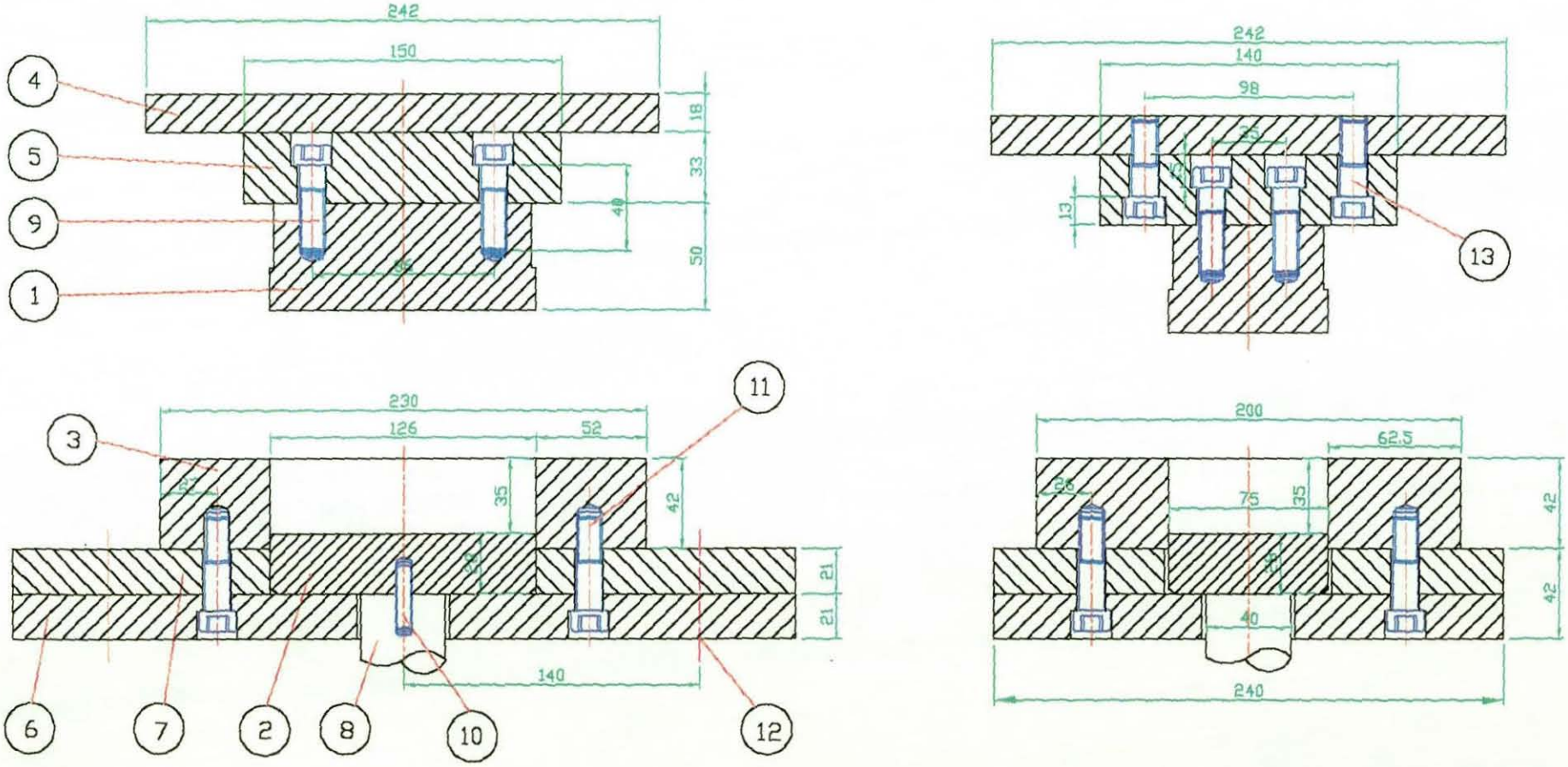
Appendix A

Assembly Drawing and Part List of the Squeeze Casting Die



LOUGHBOROUGH UNIVERSITY DEPARTMENT OF MANUFACTURING ENGINEERING MODULE : PhD Research Project	TOLERANCES UNLESS OTHERWISE SPECIFIED X.X = ± 0.1 X.XX = ± 0.01 X.XXX = ± 0.005	FINISHING SYMBOL	MATL : H13 Die Steel TREATMENT : - HARDNESS : - QUANTITY : 1	PART NAME : Squeeze Casting Die TOOL NO. : - DRAWN BY: MING SHYAN YONG CHECK BY: MING SHYAN YONG	DRG NO: PhD-SQ-Die ANGLE OF PROJ : 1st angle

16.	Dowel Pin	Silver Steel	∅10 x 36	2
15.	Cartridge Heater	Stainless Steel	∅15.8 x 60	2
14.	Cartridge Heater	Stainless Steel	∅15.8 x 76	6
13.	Bolt & Spring washer	High Tensile	M 12 x 35	4
12.	Bolt & washer	High Tensile	M 16 x 45	4
11.	Bolt & Spring washer	High Tensile	M 12 x 45	6
10.	Stud	High Tensile	M10 x 40	1
9.	Bolt & Spring washer	High Tensile	M12 x 40	4
8.	Ejector Bar	Mild Steel	∅ 40 x 40	1
7.	Back Clamping Plate (2)	Mild Steel	370 x 240 x 21	1
6.	Back Clamping Plate (1)	Mild Steel	370 x 240 x 21	1
5.	Punch Backing Plate	Mild Steel	150 x 140 x 34	1
4.	Top Clamping Plate	Mild Steel	242 x 242 x 18	1
3.	Die Cavity	H13	230 x 200 x 42	1
2.	Ejector Plate	H13	126 x 75 x 28	1
1.	Punch	H13	126 x 75 x 50	1
No.	**Description**	**Material**	**Dimension** (mm)	**QTY**
Part List				

Table 1 Part List for the Assembly Drawing of the Squeeze Casting Die

Appendix B : Mechanical Properties from the Evaluation of Process Variables in the Primary Casting Programme

Table	Subject	Specimens Allocation
1	Tensile Properties of Specimens Produced from the Squeeze Infiltration, Squeeze Casting and Gravity Die Casting Processes	section 7.6.2.1
2	Tensile Properties of Squeeze Infiltrated RZ5DF MMC for the Evaluation of Different Preform Systems	section 7.6.2.2
3	Tensile Properties of Squeeze Infiltrated RZ5DF MMC for Investigating the Correlation Between Preform Temperature and Applied Pressure	section 7.6.2.3
4	Tensile Properties of Squeeze Cast RZ5DF Alloy and Squeeze Infiltrated MMC for the Evaluation of Applied Pressure	section 7.6.2.4
5a	Tensile Properties of Squeeze Cast RZ5DF Alloy for the Evaluation of Applied Pressure Duration	section 7.6.2.5
5b	Tensile Properties of Squeeze Infiltrated RZ5DF-14% vol. Saffil MMC for the Evaluation of Applied Pressure Duration	section 7.6.2.5
6	Tensile Properties of Squeeze Cast RZ5DF Alloy and Squeeze Infiltrated MMC for the Evaluation of Process Consistency	section 7.6.2.6

Casting Sample	Process	Material	U.T.S. (MPa)	% Elongation	% Area reduction
1	Squeeze Infiltration	RZ5DF MMC (as-cast)	264	2.5 %	2.2 %
2			255	2.8 %	2.7 %
3			257	2.5 %	2.3 %
6	Squeeze Infiltration	RZ5 MMC (as-cast)	246	3.1 %	2.5 %
7			240	3 %	2.2 %
8			244	2.8 %	2 %
11	Squeeze Casting	Mg-2.5%Zn-RE Alloy (as-cast)	150	3 %	2 %
12			152	3 %	2 %
13			148	3 %	2 %
16	Squeeze Casting	RZ5DF Alloy (as-cast)	202	14 %	8.3 %
17			197	12.8 %	7.5 %
18			196	11.5 %	7.2 %
21	Squeeze Casting	RZ5DF Alloy (T5)	202	12.7 %	7.9 %
22			208	14%	8.5 %
23			193	13 %	7.5 %
26	Squeeze Casting	RZ5 Alloy (as-cast)	190	10.4 %	6.7 %
27			195	10.9 %	7.2 %
28			201	11.6 %	7.8 %
31	Squeeze Casting	RZ5 Alloy (T5)	205	11 %	7.1 %
32			207	12.4 %	7.6 %
33			207	13 %	8 %
36	Gravity Die Casting	Mg-2.5%Zn-RE Alloy (as-cast)	129	2 %	2 %
37			133	2 %	2 %
38			131	2 %	2 %
41	Gravity Die Casting	RZ5DF Alloy (as-cast)	133	4 %	3 %
42			124	4 %	3 %
43			128	4 %	2.8 %
46	Gravity Die Casting	RZ5DF Alloy (T5)	123	3.4 %	2.5 %
47			144	5.1 %	3 %
48			154	5.6 %	4 %
51	Gravity Die Casting	RZ5 Alloy (as-cast)	151	5.5 %	4 %
52			144	5.5 %	4 %
53			139	5 %	3.5 %
56	Gravity Die Casting	RZ5 Alloy (T5)	149	5.5 %	4.1 %
57			159	6 %	3.6 %
58			165	6.5 %	4.6 %

Table 1 Tensile properties of specimens produced from the squeeze infiltration, squeeze casting and gravity die casting processes

Casting Sample	Preform Systems	U.T.S. (MPa)	% Elongation	% Area reduction
61	20% vol. fraction Saffil fibres with 5% silica binder	265	2.4 %	2.3 %
62		264	2 %	2.1 %
63		267	2.4 %	2.3 %
66	20% vol. fraction Saffil fibres with 5% alumina binder	288	0.9 %	0.7 %
67		285	0.9 %	0.6 %
68		278	0.7 %	0.5 %
71	14% vol. fraction Saffil fibres with 5% silica binder	264	2.5 %	2.2 %
72		255	2.8 %	2.7 %
73		257	2.5 %	2.3 %
76	12% Saffil + 9% carbon fibres with 5% alumino-silicate binder	249	2.5 %	1.5 %
77		216	1.2 %	1 %
78		234	1.7 %	1.2 %
81	20% vol. fraction carbon fibres with 5% alumino-silicate binder	176	0.9 %	0.1 %
82		182	1 %	0.1 %
83		185	1.2 %	0.2 %

Table 2 Tensile properties of squeeze infiltrated RZ5DF MMC for the evaluation of different preform systems, cast with constant process parameters.

Casting Sample	Preform Temperature	Applied Pressure	U.T.S. (MPa)	% Elongation	% Area reduction
86	400°C	60 MPa	254	1.6 %	1 %
87			253	1.6 %	0.9 %
88			251	1 %	1.6 %
91	600°C	60 MPa	246	1.8 %	1.7 %
92			246	1.4 %	1.2 %
93			248	1.6 %	1.3 %
96	750°C	60 MPa	247	1.6 %	1.6 %
97			245	1.8 %	1.6 %
98			242	2.6 %	2.2 %
101	250°C	80 MPa	201	2 %	1.6 %
102			201	2.1 %	1.8 %
103			200	2.4 %	2 %
106	400°C	80 MPa	239	2 %	1.7 %
107			252	2.5 %	2.4 %
108			235	2.1 %	2.2 %
111	600°C	80 MPa	264	2.5 %	2.2 %
112			255	2.8 %	2.7 %
113			257	2.5 %	2.3 %
116	750°C	80 MPa	254	2 %	1.8 %
117			243	1.6 %	1.4 %
118			245	1.9 %	1.7 %
121	400°C	100 MPa	229	1.5 %	2 %
122			232	1.8 %	2.4 %
123			229	1.5 %	1.7 %
126	600°C	100 MPa	236	2 %	1.6 %
127			244	2.6 %	2.3 %
128			235	2 %	2 %
131	750°C	100 MPa	246	2.5 %	2.8 %
132			243	2 %	2.3 %
133			244	2.2 %	2.7 %

Table 3 Tensile properties of Squeeze Infiltrated RZ5DF MMC for investigating the correlation between preform temperature and applied pressure. Cast with constant process parameters (other than preform temperature and applied pressure)

Casting Sample	Applied Pressure	Process/ Material	U.T.S. (MPa)	% Elongation	% Area reduction
136	0.1 MPa	Squeeze cast RZ5DF alloy	133	4 %	3 %
137			124	4 %	3 %
138			128	4 %	2.8 %
141	20 MPa	Squeeze cast RZ5DF alloy	129	8 %	5.6 %
142			137	7.2 %	4.7 %
143			132	7.4 %	4.8 %
146	40 MPa	Squeeze cast RZ5DF alloy	165	7.8 %	4.4 %
147			162	7.4 %	4.3 %
148			162	7.2 %	3.8 %
151	60 MPa	Squeeze cast RZ5DF alloy	172	11.3 %	7.1 %
152			173	11.6 %	7.8 %
153			170	10.2 %	6.7 %
156	80 MPa	Squeeze cast RZ5DF alloy	171	10.6 %	7.1 %
157			172	11.1 %	7.5 %
158			174	11.5 %	8.7 %
161	100 MPa	Squeeze cast RZ5DF alloy	177	12 %	8.4 %
162			165	11.4 %	7.5 %
163			179	11.9 %	8.7 %
166	120 MPa	Squeeze cast RZ5DF alloy	162	8.5 %	6.5 %
167			169	9.9 %	7.2 %
168			164	8.6 %	6.7 %
171	0.1 MPa	Squeeze Infiltrated RZ5DF MMC	180	0.6 %	0.5 %
172			177	0.9 %	0.6 %
173			171	1 %	0.8 %
176	20 MPa	Squeeze Infiltrated RZ5DF MMC	187	2.1 %	1.7 %
177			201	1.9 %	1.7 %
178			188	2.6 %	2 %
181	40 MPa	Squeeze Infiltrated RZ5DF MMC	206	1.7 %	1.5 %
182			205	1.7 %	1.4 %
183			215	2.1 %	1.9 %
186	60 MPa	Squeeze Infiltrated RZ5DF MMC	246	1.8 %	1.7 %
187			246	1.4 %	1.2 %
188			248	1.6 %	1.3 %
191	80 MPa	Squeeze Infiltrated RZ5DF MMC	264	2.5 %	2.2 %
192			255	2.8 %	2.7 %
193			257	2.5 %	2.3 %
196	100 MPa	Squeeze Infiltrated RZ5DF MMC	236	2 %	1.6 %
197			244	2.6 %	2.3 %
198			235	2 %	2 %
201	120 MPa	Squeeze Infiltrated RZ5DF MMC	203	2.6 %	1.9 %
202			205	3.2 %	2.5 %
203			203	2.6 %	2.2 %

Table 4 Tensile properties of squeeze cast RZ5DF alloy and squeeze infiltrated MMC for the evaluation of applied pressure, cast with constant process parameters (other than applied pressure)

Casting Sample	Applied Pressure Duration	U.T.S. (MPa)	% Elongation	% Area reduction
206	16 seconds	155	7 %	4.9 %
207		158	8.2 %	6 %
208		158	7.3 %	5.3 %
211	25 seconds	172	11.3 %	7.1 %
212		173	11.6 %	7.8 %
213		170	10.2 %	6.7 %
216	35seconds	168	9.8 %	6.7 %
217		165	9.5 %	6.4 %
218		177	11 %	6.9 %

Table 5a Tensile properties of squeeze cast RZ5DF alloy for the evaluation of applied pressure duration, cast with constant process parameters (other than applied pressure duration)

Casting Sample	Applied Pressure Duration	U.T.S. (MPa)	% Elongation	% Area reduction
221	16 seconds	215	1.9 %	1.9 %
222		207	1.6 %	1.6 %
223		212	1.8 %	2.1 %
226	25 seconds	264	2.5 %	2.2 %
227		255	2.8 %	2.7 %
228		257	2.5 %	2.3 %
231	35seconds	254	2.6 %	2.3 %
232		247	2.2 %	1.9 %
233		249	2.5 %	2.1 %

Table 5b Tensile properties of squeeze infiltrated RZ5DF-14% vol. Saffil MMC for the evaluation of applied pressure duration, cast with constant process parameters (other than applied pressure duration)

Casting Sample	Materials	U.T.S. (MPa)	% Elongation	% Area reduction
236	Squeeze Cast RZ5DF alloy	172	10.8 %	7.8 %
237		173	12 %	7 %
238		172	9.9 %	6.5 %
239		171	10.6 %	9.2 %
240		172	12 %	7.5 %
241		174	11.5 %	8.7 %
242	Squeeze Infiltrated RZ5DF-14% vol. Saffil fibre with silica binder	252	2.2 %	2.3 %
243		240	1.9 %	2.1 %
244		240	2 %	2.1 %
245		237	1.7 %	1.9 %
246		240	2 %	2.1 %
247		255	2.2 %	2.5 %
248	Squeeze Infiltrated RZ5DF-20% vol. Saffil fibre with silica binder	272	2 %	2.4 %
249		263	1.6 %	1.9 %
250		263	1.6 %	2 %
251		265	2.4 %	2.4 %
252		264	2 %	2.1 %
253		267	2.4 %	2.3 %

Table 6 Tensile properties of squeeze cast RZ5DF alloy and squeeze infiltrated MMC for the evaluation of process consistency, cast with constant process parameters

Appendix C Mechanical Properties from the Evaluation of Process Variables (Pouring and Die temperatures) and Grain Refining (Zirconium) in the Secondary Casting Programme

Table	Subject
1	The correlation of tensile properties with pouring and die temperatures for squeeze cast RZ5DF alloy specimens tested at ambient temperature
2	The correlation of tensile properties with pouring and die temperatures for squeeze cast RZ5DF alloy specimens tested at elevated temperature
3	The correlation of tensile properties with pouring and die temperatures for squeeze infiltrated RZ5DF MMC specimens tested at ambient temperature
4	The correlation of tensile properties with pouring and die temperatures for squeeze infiltrated RZ5DF MMC specimens tested at elevated temperature
5	The correlation of tensile properties with pouring and die temperatures for squeeze cast RZ5 alloy specimens tested at ambient temperature
6	The correlation of tensile properties with pouring and die temperatures for squeeze cast RZ5 alloy specimens tested at elevated temperature
7	The correlation of tensile properties with pouring and die temperatures for squeeze infiltrated RZ5 MMC specimens tested at ambient temperature
8	The correlation of tensile properties with pouring and die temperatures for squeeze infiltrated RZ5 MMC specimens tested at elevated temperature
9	Tensile Properties of Heat Treated Squeeze Cast RZ5DF Alloy and MMC
10	Tensile Properties of Heat Treated Squeeze Cast RZ5 Alloy and MMC

Casting Sample	Pouring Temperature	Die Temperature	U.T.S. (MPa)	% Elongation	% Area reduction
254	720°C	225°C	174	10.3 %	6.2 %
255			184	10.7 %	6.2 %
256			180	10.3 %	6.4 %
260	720°C	250°C	187	11.4 %	6.6 %
261			189	12.1 %	6.9 %
262			192	12.6 %	7.5 %
266	720°C	275°C	181	9.5 %	6 %
267			187	9.5 %	6 %
268			175	9 %	6.4 %
272	750°C	225°C	168	8.1 %	4.5 %
273			165	7.8 %	4.4 %
274			175	9 %	6.1 %
278	750°C	250°C	191	12.3 %	8 %
279			189	11.7 %	7 %
280			193	12 %	7 %
284	750°C	275°C	179	10 %	6.2 %
285			184	9 %	6 %
286			190	11 %	7 %
290	780°C	225°C	166	8 %	5 %
291			174	11 %	7 %
292			172	11 %	6.5 %
296	780°C	250°C	202	14 %	8.3 %
297			197	12.8 %	7.5 %
298			196	11.5 %	7.2 %
302	780°C	275°C	185	11 %	6.5 %
303			180	8 %	5 %
304			187	10 %	6 %

Table 1 The correlation of tensile properties with pouring and die temperatures for squeeze cast RZ5DF alloy specimens tested at ambient temperature

Casting Sample	Pouring Temperature	Die Temperature	U.T.S. (MPa)	% Elongation	% Area reduction
257	720°C	225°C	91	50 %	68 %
258			79	49 %	44 %
259			84	50 %	64 %
263	720°C	250°C	73	44 %	62 %
264			74	46 %	60 %
265			88	50 %	64 %
269	720°C	275°C	83	49 %	52 %
270			74	51 %	62 %
271			75	50 %	61 %
275	750°C	225°C	80	48 %	48 %
276			98	47 %	54 %
277			91	45 %	56 %
281	750°C	250°C	78	44 %	54 %
282			76	49 %	62 %
283			77	49 %	64 %
287	750°C	275°C	73	50 %	58 %
288			82	46 %	60 %
289			70	51 %	68 %
293	780°C	225°C	103	31 %	35 %
294			89	46 %	41 %
295			99	32 %	32 %
299	780°C	250°C	70	46 %	66 %
300			76	46 %	64 %
301			76	47 %	64 %
305	780°C	275°C	73	48 %	63 %
306			67	47 %	66 %
307			78	49 %	66 %

Table 2 The correlation of tensile properties with pouring and die temperatures for squeeze cast RZ5DF alloy specimens tested at elevated temperature

Casting Sample	Pouring Temperature	Die Temperature	U.T.S. (MPa)	% Elongation	% Area reduction
308	720°C	225°C	230	2.1 %	2.2 %
309			229	2.1 %	2.5 %
310			235	1.7 %	2 %
314	720°C	250°C	243	2.4 %	2.5 %
315			231	2.1 %	2.2 %
316			234	2.1 %	1.8 %
320	720°C	275°C	233	2.4 %	2.5 %
321			228	2.1 %	1.9 %
322			231	2.4 %	2.4 %
326	750°C	225°C	236	2.1 %	2.2 %
327			239	1.7 %	2 %
328			236	2.4 %	2.5 %
332	750°C	250°C	233	1.2 %	1.5 %
333			239	2.1 %	2 %
334			235	1.7 %	1.8 %
338	750°C	275°C	236	2.4 %	2.5 %
339			237	2.1 %	1.8 %
340			232	1.9 %	1.7 %
344	780°C	225°C	231	1.7 %	2.2 %
345			227	1.4 %	1.7 %
346			229	1.6 %	1.9 %
350	780°C	250°C	242	1.4 %	1.5 %
351			248	1.4 %	1.8 %
352			236	1.6 %	1.9 %
356	780°C	275°C	248	2.4 %	2.5 %
357			248	2.8 %	3 %
358			246	2.3 %	2.4 %

Table 3 The correlation of tensile properties with pouring and die temperatures for squeeze infiltrated RZ5DF-14% vol. Saffil MMC specimens tested at ambient temperature

Casting Sample	Pouring Temperature	Die Temperature	U.T.S. (MPa)	% Elongation	% Area reduction
311	720°C	225°C	147	3.5 %	3 %
312			146	4.1 %	3.5 %
313			152	4.1 %	3.9 %
317	720°C	250°C	140	4.8 %	3.5 %
318			143	4.1 %	4 %
319			140	4.5 %	3.2 %
323	720°C	275°C	141	3.6 %	3.5 %
324			135	3.8 %	3.2 %
325			134	3.5 %	3 %
329	750°C	225°C	146	3.5 %	3 %
330			142	3.8 %	2.2 %
331			142	3.8 %	3 %
335	750°C	250°C	141	3.5 %	2.2 %
336			139	3.1 %	2.5 %
337			148	3.1 %	2.5 %
341	750°C	275°C	145	4.1 %	3.8 %
342			149	4.5 %	4 %
343			140	4 %	3.5 %
347	780°C	225°C	132	3.1 %	2.2 %
348			130	3.8 %	2.7 %
349			137	3.5 %	2.5 %
353	780°C	250°C	153	3.1 %	2.2 %
354			154	3.5 %	2.5 %
355			155	2.8 %	2 %
359	780°C	275°C	152	3.8 %	2.7 %
360			142	3.5 %	3.2 %
361			148	3.8 %	3 %

Table 4 The correlation of tensile properties with pouring and die temperatures for squeeze infiltrated RZ5DF-14% vol. Saffil MMC specimens tested at elevated temperature

Casting Sample	Pouring Temperature	Die Temperature	U.T.S. (MPa)	% Elongation	% Area reduction
362	720°C	225°C	178	9.8 %	6.2 %
363			164	7 %	5 %
364			174	9 %	5 %
368	720°C	250°C	167	8 %	5 %
369			196	12 %	8 %
370			174	9 %	5.5 %
374	720°C	275°C	167	8 %	4.6 %
375			178	8.7 %	5 %
376			182	9 %	5.4 %
380	750°C	225°C	177	10.7 %	5.7 %
381			170	7.9 %	5.4 %
382			167	7 %	5 %
386	750°C	250°C	190	10.4 %	6.7 %
387			195	10.9 %	7.2 %
388			201	11.6 %	7.8 %
392	750°C	275°C	175	9.5 %	5.7 %
393			182	11.4 %	7.4 %
394			182	10 %	7.1 %
398	780°C	225°C	172	9 %	5 %
399			165	8.3 %	5.2 %
400			183	11.8 %	7.6 %
404	780°C	250°C	184	10 %	6 %
405			175	8.8 %	6.2 %
406			199	13.4 %	7.9 %
410	780°C	275°C	174	9 %	5 %
411			193	11 %	6 %
412			176	8 %	5 %

Table 5 The correlation of tensile properties with pouring and die temperatures for squeeze cast RZ5 alloy specimens tested at ambient temperature

Casting Sample	Pouring Temperature	Die Temperature	U.T.S. (MPa)	% Elongation	% Area reduction
365	720°C	225°C	99	45 %	68 %
366			95	40 %	62 %
367			102	40 %	58 %
371	720°C	250°C	97	45 %	65 %
372			93	50 %	68 %
373			93	40 %	60 %
377	720°C	275°C	91	32 %	38 %
378			87	30 %	35 %
379			91	39 %	50 %
383	750°C	225°C	98	46 %	62 %
384			99	45 %	62 %
385			96	44 %	61 %
389	750°C	250°C	91	44 %	64 %
390			93	45 %	68 %
391			99	44 %	65 %
395	750°C	275°C	90	49 %	64 %
396			87	49 %	64 %
397			93	41 %	68 %
401	780°C	225°C	97	35 %	48 %
402			100	43 %	45 %
403			98	40 %	53 %
407	780°C	250°C	96	46 %	63 %
408			85	48 %	59 %
409			84	38 %	50 %
413	780°C	275°C	97	46 %	64 %
414			87	40 %	52 %
415			86	38 %	48 %

Table 6 The correlation of tensile properties with pouring and die temperatures for squeeze cast RZ5 alloy specimens tested at elevated temperature

Casting Sample	Pouring Temperature	Die Temperature	U.T.S. (MPa)	% Elongation	% Area reduction
416	720°C	225°C	239	2.4 %	1.7 %
417			244	2.8 %	2.3 %
418			240	2.4 %	2 %
422	720°C	250°C	237	2.1 %	1.5 %
423			227	1.7 %	1.5 %
424			239	2.1 %	2 %
428	720°C	275°C	231	2.1 %	1.8 %
429			231	2.1 %	1.9 %
430			236	2.4 %	2.2 %
434	750°C	225°C	239	2 %	2 %
435			241	2.4 %	2 %
436			236	2.4 %	1.7 %
440	750°C	250°C	243	2.1 %	1.5 %
441			237	2.4 %	2 %
442			232	2.4 %	2.5 %
446	750°C	275°C	234	2.7 %	2 %
447			237	2.5 %	1.8 %
448			231	2.1 %	1.7 %
452	780°C	225°C	229	2.1 %	2 %
453			234	2.1 %	1.5 %
454			232	2.4 %	1.8 %
458	780°C	250°C	246	3.1 %	2.5 %
459			240	3 %	2.2 %
460			244	2.8 %	2 %
464	780°C	275°C	236	2.8 %	2 %
465			250	3 %	2.2 %
466			244	2.3 %	2 %

Table 7 The correlation of tensile properties with pouring and die temperatures for squeeze infiltrated RZ5-14% vol. Saffil MMC specimens tested at ambient temperature

Casting Sample	Pouring Temperature	Die Temperature	U.T.S. (MPa)	% Elongation	% Area reduction
419	720°C	225°C	170	3.6 %	3.6 %
420			169	3.5 %	3.1 %
421			173	3.3 %	3.2 %
425	720°C	250°C	163	3.7 %	3.5 %
426			170	3.1 %	3 %
427			158	3.5 %	3.5 %
431	720°C	275°C	157	3.5 %	2.7 %
432			164	3.1 %	2 %
433			159	3.3 %	3.5 %
437	750°C	225°C	167	2.8 %	2 %
438			172	3.3 %	2.2 %
439			161	3.1 %	2.3 %
443	750°C	250°C	171	2.8 %	2 %
444			167	3.8 %	2.7 %
445			172	3.5 %	2.5 %
449	750°C	275°C	166	3.1 %	2.5 %
450			170	3.8 %	2.5 %
451			169	3.5 %	2 %
455	780°C	225°C	155	3.8 %	4.7 %
456			167	2.8 %	2.5 %
457			156	3.5 %	2.7 %
461	780°C	250°C	180	3.5 %	2.2 %
462			174	2.8 %	2.5 %
463			174	2.7 %	2 %
467	780°C	275°C	171	3.1 %	2 %
468			170	3.5 %	2.5 %
469			180	2.8 %	2.7 %

Table 8 The correlation of tensile properties with pouring and die temperatures for squeeze infiltrated RZ5-14% vol. Saffil MMC specimens tested at elevated temperature

Casting Sample	Material	330°C (hrs)	180°C (hrs)	U.T.S. (MPa)	% Elongation	% Area Reduction
484	RZ5DF Alloy Tested at Ambient Temperature	2	4	184	8 %	6 %
485		2	8	191	9 %	7 %
486		2	16	203	12 %	8 %
487	RZ5DF Alloy Tested at Ambient Temperature	2	16	202	12.7 %	7.9 %
488		2	16	208	14 %	8.5 %
489		2	16	193	13 %	7.5 %
490	RZ5DF Alloy Tested at Elevated Temperature	2	16	92	47 %	65 %
491		2	16	83	46 %	64 %
492		2	16	81	46 %	62 %
513	RZ5DF MMC Tested at Ambient Temperature	0	0	242	1.5 %	1.7 %
514		0	2	242	2.5 %	2.5 %
515		0	8	239	3 %	3 %
516		1	0	234	2.4 %	2.7 %
517		1	2	236	2.1 %	2.5 %
518		1	8	232	2.5 %	2.8 %
519		2	0	229	3 %	3.5 %
520		2	2	227	2.8 %	3 %
521		2	8	231	2.6 %	2.8 %

Table 9 Tensile properties of heat treated squeeze cast RZ5DF alloy and MMC

Casting Sample	Material	330°C (hrs)	180°C (hrs)	U.T.S. (MPa)	% Elongation	% Area Reduction
522	RZ5 Alloy Tested at Ambient Temperature	2	16	205	11 %	7.1 %
523		2	16	207	12.4 %	7.6 %
524		2	16	207	13 %	8 %
525	RZ5 Alloy Tested at Elevated Temperature	2	16	106	41 %	58 %
526		2	16	103	41 %	57 %
527		2	16	105	42 %	60 %
548	RZ5 MMC Tested at Ambient Temperature	0	0	243	3 %	2.2 %
549		0	2	239	2.5 %	2.2 %
550		0	8	240	2 %	1.9 %
551		1	0	236	2.1 %	2 %
552		1	2	235	1.9 %	1.8 %
553		1	8	229	2 %	1.8 %
554		2	0	231	2.2 %	2 %
555		2	2	232	1.8 %	1.7 %
556		2	8	231	2 %	1.6 %

Table 10 Tensile properties of heat treated squeeze cast RZ5 alloy and MMC