PRODUCTION AND PROPERTIES OF METAL COATED POWDERS FOR USE IN THE PRODUCTION OF ENGINEERING COMPONENTS

by

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To my wife

For all the love and support
she has given me

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SUMMARY

This research involves a study of some of the methods available for covering material substrates with a metallic coating. The materials studied were metal and ceramic powders, and the research attempted to produce composite powders, which could be used in the powder metallurgy industries as cheaper replacements for existing expensive powders. For example, iron powder particles coated with copper/tin could replace expensive bronze powders in machine bearing production.

The experimental work involved the study of two wet coating techniques for the deposition of metals from aqueous solutions onto various substrate materials. These methods were: chemical displacement plating and electroless (or chemical) plating. A fluidised bed apparatus and stirred baths were used in the handling and plating of the powder particles. The former apparatus has been specifically developed for pilot plant production of plated powders, and can handle between 0.5 and 1 kg of particles with diameters up to 300 μm. The particles are kept apart and can move freely within the powder bed, whilst immersed in a continually flowing aqueous plating solution.

Two types of core materials were used; an atomised iron powder with particle size between 125 and 150 μm; and ceramic powders as follows: alumina (45 μm), silicon carbide (29 and 250 μm), tungsten carbide

(45μm) and tantalum carbide (45 μm). The metal coatings studied for the above powders were copper, tin, cobalt and nickel.

Once the composite powders were produced, chemical analyses were carried out to determine the content of metal (or metals) deposited on each sample of powder. Coatings which were 62.4wt% of the powder composite were made. The iron based composite powders were compacted into cylinders at pressures ranging from 480 to 870 Nmm^{-2} and densities between 6.73 and 8.00 gcm^{-3} were achieved. For the metal coated ceramic powders, the pressures required for compaction ranged from 380-860 Nmm^{-2} and densities ranged between 2.22 and 11.20 gcm^{-3} depending on the powder substrate. The cylindrical compacts were mechanically tested and the following properties determined: density, tensile strength, compressibility and modulus of elasticity, and the results of the coated and uncoated powder compacts were compared.

The surface structure of the coatings on the powder particles were examined using scanning electron microscopy for all the powders and the microstructure of compacts in the unsintered and sintered states were studied.

The results showed that the behaviour and properties of the composite powders were controlled, as expected, by the amount, type and properties of the metal coating. The compressibilities, in particular, of the coated ceramic powders were improved by using a

metallic coating. These powders could find uses in many production areas involving cemented carbides, sintered alloy steels, bearings, electromagnetic dielectrics, wear resistant materials used in brakes and clutches, and any materials which need to be made from composite powders.

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CHAPTER 1

INTRODUCTION TO POWDER METALLURGY

1.1 THE IMPORTANCE OF THE POWDER METALLURGY INDUSTRY
[References: 1, 4, 5, 7, 10, 11 and 18]

Although powder metallurgy is still a small industry, being less than 1% of the world's total metals industry, powder products are found in virtually all areas of life. Without them, many achievements and conveniences of our present civilisation would have been impossible.

The success of the powder metallurgy industry (it has enjoyed an above average growth rate for the past four decades) derives from its ability to mass-produce complex structural parts with savings in labour, material, and energy.

In recent years, an entirely new dimension has been added with the achievement of full density and improved control of purity and microstructure. This has resulted in the fabrication of high performance materials, permitting extended service life and more efficient use. Examples of commercial uses include aerospace superalloys, low alloy steels, dispersion strengthened alloys, tool steels, and cermets, such as cemented carbides.

These developments have added considerable diversification to the conventional "press and sinter" techniques and are opening up new markets beyond the traditional ones of the automotive, agricultural, domestic machines and office equipment industries.

A Brief History of Powder Metallurgy
[References: 3, 4, 5, 7 and 8]
The earliest development of powder metallurgy can be traced back to 5000 years ago in several parts of the world.

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Around 3000 BC, the Egyptians used a "sponge iron" for making tools by the reduction of iron oxide in charcoal furnaces. Around AD 1100, the Arabs were making high quality swords from iron powder. Another example is the Delhi column in India, which dates from about 300 AD. This column is made from approximately 6.5 tonnes of reduced iron powder. In the nineteenth century, this process was used to produce platinum and tungsten wires, which led to the development of chemically precipitated powders and new consolidation routes, which avoided high temperatures. Such activities occurred in both Russia and England, where precipitated and hot worked powders were used to overcome inadequate temperature capabilities.

The first significant use in general manufacturing, however, was in the 1920s; when powder metallurgy was used to produce tungsten carbide cutting tool tips and non-ferrous bushings. In recent years, the process has become highly developed and large quantities of powder metal products are made annually. From 1960 to 1980, the consumption of iron powder has increased tenfold with about 20,000T of powder used in Britain, ten times this amount used in America and an equal amount in Japan, in the beginning of the 1980s. The relatively recent expansion of powder metallurgy processing into areas requiring high quality materials, and unique properties in high productivity areas are expected to continue creating opportunities in the future during the 1990s.

What is Powder Metallurgy?
[References: 1 and 16]
Powder Metallurgy could be defined as the study of the processing of metal powders, including fabrication, characterisation and conversion of metal powders into useful engineering components.

Therefore, it could be considered as the name given to a process, wherein fine metal powders are blended, consolidated into a desired shape (usually by compaction); and then heated (sintered) in a controlled atmosphere. This is done at a temperature below the

melting point of the major constituents, for a sufficient time to bond the contacting surfaces of the particles, and to establish the desired properties.

The process, commonly designated as "PM", readily lends itself to mass production of small, intricate parts of high precision, often without requiring additional machining. It is now a term given to the process of making all types of powders, whether metallic or ceramic, into engineering products. For this reason, the powder metallurgy industry is a major growth industry today.

1.2 WHY POWDER METALLURGY?
[References: 1, 2 and 4]

It is reasonable to ask why bother breaking up pieces of metal into a powder only to re-assemble them again. There are in fact a number of very good reasons, which can be discussed as follows:

1. Refractory Metals
These are usually metals with very high melting points; for example, tungsten (3140°C), tantalum (3000°C), osmium (3045°C), and molybdenum (2800°C) etc. These are refractory metals and are very difficult to produce by melting and casting because they are brittle in the cast state. They are initially extracted in the form of a powder. This lends them readily available to the Powder Metallurgy process.

2. Composite Materials
If two or more materials are normally immiscible in the molten state, then an intimate mixture of these materials can be obtained through Powder Metallurgy. The most common examples of this are as follows:

i) The use of cemented carbides used for cutting tools and wear resistant parts;
ii) Electrical contact materials, such as copper/tungsten, silver/cadmium oxide.

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iii) Friction materials for brake linings and clutch facings in which abrasive non-metallic materials are embedded in a copper or other metallic matrix.

3. Porous Materials

Porous metals are used in items such as oil retaining bearings, filters, flame traps, bubblers, cooling devices etc. The Powder Metallurgy process, since it is concerned with the production of a part having a significant and controlled porosity, lends itself readily to the manufacture of these products.

4. Production of Intricate Engineering Components

Because high dimensional accuracy is possible with Powder Metallurgy, minimal waste of material occurs, and post production machining operations are not necessary. Therefore a Powder Metallurgy part is cheaper to produce than one obtained from a more conventional route. However, there are limitations over shape and overall size of component, if it is made by die compaction processes. Cold isostatic pressing now enables very large components to be made.

5. Special High Duty Alloys

Under this heading, there are a range of hardened alloys with dispersed phases, of which SAP (Sintered Aluminium and Powder) was one of the first. In general the powder is compacted into a blank, or billet, which is then subjected to forging and forming in the normal way. Often, cold isostatic pressure is used to make large billets, which can then be processed further.

These alloys have very precise compositions which would not result were melting and casting processes used. Alloys having a particular coefficient of expansion, or magnetic properties, fall into this category.

In theory, any size or component can be produced, ranging from a few grammes to several hundred kilogrammes. This means that a single process can be employed for part production.

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Significant economic benefits are achieved by this technique. Large numbers of die-compacted small parts can be rapidly produced, at less cost than by normal forging or machining processes. Metal utilisation is almost 100%, less energy is required, and the process is ecologically clean.

The mechanical properties of components are equal to, or even better than, those obtained via traditional metallurgical methods. Cutting tools and wear parts, for example, have an increased life span when produced by powder metallurgy.

Powder metallurgy offers the designer material/property combinations that permit production of engineered structures for optimised mechanical and physical properties.

Powder metallurgy is the only viable manufacturing process for materials with very poor workability, such as ceramics and carbides.

1.3 APPLICATIONS OF POWDER METALLURGY
[References: 1, 2, 4, 5, 7, 12, 16 and 18]

The tremendous advantages of powder metallurgical material has led to a variety of applications, which range from the most exotic and sophisticated innovations, which enable rockets to reach for the stars; to parts in our domestic washing machines and automobiles. The main applications are outlined below:

1. Automotive Applications
Due to the improved material and processing techniques, as well as the increased awareness and knowledge of powder metallurgy techniques by design engineers, plus the recognised reductions in manufacturing costs of mass produced techniques, there has been major redevelopment of powder metallurgy materials for a multitude of automotive uses. This has been achieved by the use of high tonnage compaction presses and high compressibility powders, which allow for higher densities and larger parts to be obtained. The availability of larger sintering

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furnaces and advanced techniques in automation procedures has also spurred this new growth.

Examples of the range of automotive parts being made today by powder metallurgy are shown in Figure 1.

2. Aerospace Applications

In most of these applications powder metallurgical techniques are used because the material used cannot be cast in an economical or practical manner; there is a need for controlled porosity; or there is some unique property which can only be obtained by using this method. See Table 1 for aerospace applications [Reference 4].

3. Consumer Products and Business Machines

Powder metallurgical materials are widely used in the multitude of consumer products, because of their electrical and magnetic properties, and secondary manufacturing operations as plating and surface finishing are possible. See Figure 2 for examples of domestic applications.

4. Atomic Energy Applications

Powder metallurgical techniques have been widely applied to material fabrication problems associated with nuclear power reactors. Powder metallurgical processing of particular importance in this field are: the ability to make alloys of elements with large differences in density or melting point; also the ability to make multi-phase materials, especially metal-ceramic types; the ease of making layered or clad types of materials. Metals of particular importance are: uranium and thorium as fuel materials, and zirconium and its alloys are useful as cladding materials to inhibit corrosion in reactors.

Many conventional materials have also been used for nuclear applications. Various techniques, such as hot pressing, isostatic compaction and slip casting have been successfully applied to nuclear applications.

TABLE 1:   SOME EXAMPLES OF AEROSPACE APPLICATIONS WHICH USE POWDER
           METALLURGY TECHNIQUES

Material	Aerospace Application
Beryllium	Heat shields for space capsules, gyroscope parts, antennas, Telstar space vehicles and some structural parts
Tungsten	Used in plasma jet engines
Tungsten-Silver	For infiltrated composites, rocket nozzles
Molybdenum	To protect silicon semiconductors in rectifiers used in space
Tantalum	Used for capacitors in complex controls and communications of space satellites and vehicles, heat shields of propulsion units of missiles.

1.4 PRODUCTS FROM POWDER METALLURGY
[References: 2, 4, 5, 7, 14 and 16]

These products can be generally classed into four main groups as follows:

a)   Complex shaped products - gears, cams, internal shapes, piston rings, etc.

b)   Products from materials which would otherwise be extremely difficult to manufacture, e.g. tungsten carbide tips for tools and titanium carbide tips.

c)   Porous components: self-lubricating bearings;   made of bronze, iron or brass. They are of relatively low density and are easily impregnated or persuaded to absorb lubricants of various types.

d)   Filters manufactured from powder or woven meshes (usually rolled or pressed into shape). Made from bronze, stainless steel, or pure nickel. Used for corrosive fluid filtration, e.g;. liquid oxygen;   pressure limiters;   flame arresters;   fluidised beds; acoustic absorption; liquid aeration and breather vents.

e)   Products where combined properties of two metals or of metal and non-metal are desired: uranium carbides uranium oxides; zirconium borides are sintered for a wide range of nuclear applications. Composites for friction materials with iron or copper base with special additions designed to modify the frictional characteristics.

1.5 DISADVANTAGES OF POWDER METALLURGY
   [References: 1, 4 and 7]

The major disadvantages of the process are as follows:

1. Inferior Strength Properties
In some cases powder metallurgical parts have the mechanical properties which are less than wrought or cast products of the same material. Their use may be limited when high stresses are involved. However the required strength can frequently be obtained by using different and usually more costly materials, or by using secondary processing, such as forging, or hot pressing.

2. Relatively High Die Cost
Because of the high pressures and severe abrasion involved in the compaction process, the punches and dies must be made of expensive materials and be relatively massive. Production volumes of less than 10,000 identical parts are normally not practical, unless the press tool costs are ignored.

3. High Material Cost

On a unit weight basis, powdered metals are considerably more expensive than wrought or cast stock. However, the absence of scrap and elimination of machining often offsets the higher material costs. Powder metallurgy is usually employed for parts, where the material cost is outweighed by the special shape or property of the component.

4. Design Limitations

In compaction processes, the powder route is simply not feasible for some shapes. Parts should have essentially uniform sections along the axis of compression. The overall size must be within the capacity of available presses: and cross-holes or special chamfers are not possible. Some complex shapes are, however, possible with a different form of compaction such as cold isostatic pressing, slip casting or injection moulding, In the latter process very complex shapes are now possible.

1.6 CHARACTERISTICS OF POWDERS
[References: 3, 5, 7, 11, 15 and 16]

A powder particle is defined as the smallest unit of a powder that cannot be subdivided and is characterised by the basic properties of the material from which it has been made. The physical character of the surface of the particle, and the particle shape and size, are the principal factors determining the success or failure of a powder metallurgy process, and their importance cannot be overemphasised.

The main characteristics which are important to the behaviour of the powder are:

FLOW RATE: It is a measure of the ease by which a powder can be fed and distributed into a die, it can significantly influence the cycle time of the pressing operation and hence, the rate of production. The standard flow meter for metal powders is the Hall Flowmeter which is a funnel shaped apparatus having a calibrated orifice. The time required for a given amount of powder to flow unaided through the orifice is

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reported as the flow rate of the powder. For powders that do not flow, a special funnel is used or a zero value is reported.

COMPRESSIBILITY: A given weight of metal powder is compacted at a definite pressure in a test die under accurately controlled conditions. The ratio of the density of the green compact to the theoretical density of the component powders is a measure of the compressibility of the powder.

GREEN STRENGTH: This is used to assess the ability to handle after compacting, but before sintering. The strength of a green compact is ordinarily determined by pressing a given weight of powder to a specified density in the shape of a rectangular bar of standard size. This test bar is supported at both ends as a simple beam and loaded transversely to fracture. The modulus of rupture is reported as the green strength of the powder undergoing evaluation.

MICROSTRUCTURE OF THE POWDER: This influences those characteristics such as plasticity and the ability to be cold pressed. It influences the pressing pressure, flow characteristics and the final product strength. Powder can be fully dense or solid, porous, crystalline, partially crystalline (as with rapidly solidified powders), containing other phases or precipitates etc.

PARTICLE SIZE AND SHAPE: Frequently powders are made up of various sizes of particles, ranging from 0.1 to 1000 μ m. The more irregular the particle shape the greater the difficulty in assigning a unique particle size. This causes a disagreement between the particle size distributions obtained by different techniques and instruments. Shapes can be round, angular, dendritic, rod-like etc.

SURFACE AREA: The surface area is an average measure of the external surface of a large number of particles. It provides insight into the powder behaviour, when dealing with chemical activity, catalysis, friction, adsorption, contamination, pressing and sintering. It is a useful powder characteristic because it often correlates with various

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kinetic and geometric characteristics. Consequently surface area is combined with other parameters as part of an overall description of the powder.

TAP DENSITY: Very often a mass of loose powder will be mechanically vibrated or tapped. The density of the powder mass increases due to this treatment and is always higher than the apparent density.

This comes about by the induced movement of the particles which causes separation and allows the particles to settle to a greater extent and so decreases the porosity between them;   this will lead to an increase in the density.

CHEMICAL COMPOSITION: Chemical analyses are usually run on all powders to determine the amount of the major metal present, such as the total copper content in copper powder;   plus such impurities as might normally be expected in the material and which, if present in excess, could cause unusual and undesirable behaviour in processing. Impurities, such as oxide or nitride films, weaken the final product, and alloying elements control the mechanical behaviour.

APPARENT DENSITY: This is associated with the flow characteristics and it is a measure of the powders' ability to fill available space without the application of external pressure. The apparent density refers to the weight of a unit volume of loose powder and it is one of the most critical and useful characteristics of a powder.   It is important because it determines the actual volume occupied by a mass of powder and this ultimately determines the size of the compaction tooling and the magnitude of the press motions necessary to compact and densify the loose powder.

1.7 THE POWDER METALLURGY PROCESS

The powder metallurgy process normally consists of five basic steps:

1. Production of powders.
2. Blending or mixing.
3. Compacting.
4. Sintering.
5. Post-sintering operations where necessary.

Figure 3 shows a schematic flow chart of the powder metallurgy process.

1. Production of Powders
[References: 3, 5, 12, 13, 16 and 20]

There are several methods for producing metal or ceramic powders where the shape and properties of the powder particles depend on the method used. The most common methods involve atomization of molten material, reduction of metal oxides, electrolytic deposition, decomposition of metallic compounds, and mechanical comminution. These techniques are explained below. Other less commonly used methods are precipitation from a chemical solution, production of fine chips by machining and vapour condensation. The source material is generally bulk metals, ceramics and alloys, ores, salts, and other compounds.

a) Atomization

Atomisation involves forming a liquid material stream by injecting molten metal or ceramic through a small orifice. The stream is then broken up by jets of inert gas, or water, thus producing solid particles. Particle size depends on the temperature of the material, rate of flow, nozzle size, and jet characteristics. In one variation of this method, a consumable electrode is rotated rapidly in a helium filled chamber. The centrifugal force breaks up the molten tip of the electrode, thus producing particles.

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b) Reduction

Reduction of metal oxides is carried out using gases, such as hydrogen and carbon monoxide, as reducing agents. Very fine metallic oxides are thus chemically reduced to the metallic state. This process produces metal powders of uniform size. The powders are spongy and porous and have spherical or angular shapes.

c) Electrolytic Deposition

Metal powders may also be produced by electrolytic deposition, either from aqueous solutions or from fused salts. These powders are among the purest. The size and shape of the particles depend on process parameters.

d) Thermal Decomposition of Carbonyls

Metal carbonyls, such as in carbonyl, $Fe(CO)_5$, and nickel carbonyl, $Ni(CO)_4$, are formed by reacting iron or nickel with carbon monoxide. These products are then thermally decomposed to iron nickel, producing small, dense, uniform spherical particles of high purity.

e) Comminution

Mechanical comminution (pulverisation) involves crushing, or grinding brittle materials into small particles. With brittle materials the powder particles are angular in shape, whereas with ductile metals, they are flaky and not particularly suitable for powder-metallurgy applications.

2. Blending or Mixing
[References: 1, 3, 11, 12, 16 and 18]

Blending (mixing) of powders is the second step in powder metallurgy processing and is carried out for the following purposes:

a) Because the powders made by the various processes described above
    may have different sizes and shapes, it is necessary to mix them
    to obtain uniformity. The ideal mix is one in which particles of
    each material are distributed uniformly.

b) In order to impart special and mechanical properties and certain
    characteristics to a powder metallurgy product, powders of
    different metallic and other materials may be used.

c) In order to improve the flow characteristics of the powder metals,
    and to improve die life and reduce friction between the metal
    particles;   lubricants or waxes may be mixed with the powders.
    Typical lubricants are stearic acid or zinc stearate in amounts of
    0.25 to 5% by weight.

Mixing of powders must be carried out under controlled conditions to
avoid contamination or deterioration of the powders. Excessive mixing
may change the shape of the particles, and may also work-harden them,
thus making the subsequent compacting operation more difficult.
Powders can be mixed in air or other gaseous atmospheres. There are
several types of blending equipment available with various capacities
and mixing characteristics.

3. Compacting
    [References: 1, 2, 5, 9, 10, 11, 12, 13 and 16]
One of the most critical and controlling steps in the powder
metallurgy process is compacting. Here, loose powder is compressed and
defined into a shape known as a "green" compact. Product density and
the uniformity of that density throughout the compact are important
parameters. Generally, uniform high density is desired together with
sufficient green strength for handling and transport to the sintering
furnace.

Most compacting is done with mechanical presses, although hydraulic
and hybrid (combinations of mechanical, hydraulic or pneumatic)
presses are often used. Pressures required for pressing can range up

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to 800 MPa. Press capacities are in the order of 1800 to 2700 kN, although presses with much higher capacities are used for special applications. The choice of the press depends on parameters such as part size and configuration, density requirements, and production rate. An important consideration with regard to pressing speed is the entrapment of air in the die cavity at high speeds, as the presence of air will prevent proper compaction.

Most compacting is performed at room temperature in tungsten carbide or alloy steel dies. The die is filled with the powder and compacted, the effects being:

i) Increased density by reduction of the voids between powder particles;

ii) Cold welding, mechanical locking and adhesion of the particles for green strength;

iii) Increased contact area between the particles by plastically deforming them.

Compaction processes can also be summarised as follows:

a) **Die Compaction**

Die compaction represents the most widely used method and is considered as the "conventional" technique. It is discontinuous, using both upper and lower punches. The die provides the cavity into which powder is pressed and gives lateral constraints to the powder. Both punches are loaded to generate stress within the powder mass. Ease of powder flow is important for proper feeding of the powder into the die cavity.

The apparatus is usually designed to provide a SINGLE ACTION or DOUBLE ACTION pressing.

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The SINGLE ACTION powder compaction involves a downward movement of the top punch within the die barrel, while the bottom punch remains stationary.

The DOUBLE ACTION powder compaction is mainly used for thicker and more complex parts; where both the upper and lower punches move against the powder mass for compaction.

Since density is a function of thickness, it is difficult to produce products where more than one thickness is involved. Where non-uniform thicknesses are desired, more complicated presses or methods are used.

Most common die materials are air- or oil-hardening tool steels. Tungsten carbide dies are used for more severe applications. Punches are generally made of similar materials. Die and punch surfaces must be lapped or polished (in the direction of tool movements) for improved die life and overall performance.

b) **Isostatic Pressing**
When extremely complex shapes are desired, the powder is generally encapsulated in a flexible mould and subjected to uniform high pressure by a liquid or gas medium, this method is known as Isostatic Compaction.

There are two types of isostatic pressing as shown below:

i) **Cold Isostatic Pressing (CIP):** In Cold Isostatic Pressing, the powder is placed in a flexible rubber mould and then pressurised hydrostatically in a chamber, commonly using water. The main advantage of CIP is the uniformity of pressure in all directions and the absence of the die-wall friction; this produces compacts of practically uniform density irrespective of length to diameter ratio.

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Typical applications of this technique are tool steels, carbide tools and dies, filters, many ceramic products and parts made of super-alloys.

ii) **Hot Isostatic Pressing (HIP):** Hot Isostatic Pressing combines compaction and sintering in still another manner. Here powder is hermetically sealed in a flexible, airtight, evacuated can and then subjected to a high temperature, high pressure environment. The container is made of a high melting point sheet metal and the pressurising medium is inert gas or vitreous fluid. The main advantage of HIP is its ability to produce compacts with essentially 100% density, good metallurgical bonding, and very good mechanical properties. The process is relatively expensive and is used for making superalloy components for the aerospace industry.

It is also routinely used as a final densification step for tungsten carbide cutting tools and powder metallurgy tool steels. More recently, HIP has been employed to close internal porosity in superalloy and titanium-alloy castings for the aerospace industry.

c) **Rolling**

In powder rolling, or roll compaction, the powder is fed into the roll gap in a two high rolling mill and is compacted into a continuous strip at speeds up to 0.5 m/s. This strip is then passed through a sintering furnace and re-rolled to finished size.

The characteristics of the powder are very important when compared with those for die compaction. Here flowability, particle shape, particle size, softness and size distribution greatly influence the green density of the strip.

Sheet metals for various electrical and electronic components and for coins can be made by powder rolling.

d)   **Powder Extrusion**

In this technique, the metal powder could be forced through an orifice or a die of an appropriate shape. The powder may either be mixed with a plasticizer or contained in a can, which is extruded with the contained powder at high pressure and elevated or room temperature. Powder extrusion can yield extremely high densities and a product that does not have to be sintered if the powder was heated originally.

Different types of metals and alloys have been treated in this manner particularly in aluminium based alloys.

e)   **Powder Forging**

An important development in powder metallurgy techniques is the use of preformed, sintered alloy-powder compacts which are subsequently cold or hot forged to the desired final shapes in closed dies. These products have good surface finish and dimensional tolerances with uniform and fine grain size and distribution. The superior properties obtained make this technology particularly suitable for making automotive and jet-engine parts that are highly stressed.

f)   **Explosive Compaction**

This is a high energy rate technique, which generally uses explosives. The pressure by this method can be generated either by a punch moving at high velocity, or by an explosive charge. Explosive compacting is not necessarily confined to cylindrical compacts, shapes such as cones and hollow cylinders have also been explosively compacted by this technique.

Several products have been made with the highest relative densities, using materials such as iron, nickel, copper, and aluminium powders.

g) **Injection Moulding**

This method is an extension of the scope of powder metallurgy techniques. It is similar to plastic injection moulding using a plastic binder mixed with metal powder.

Basically this method is different from the conventional powder metallurgy techniques, in that the powder used in moulding must be finer than in conventional powder metallurgy processes, also lower isostatic pressures are required in injection moulding dies.

Products produced by this method can have very complex shapes and are denser and have higher mechanical properties for a given set of sintering conditions. Parts are also more uniform, strong and the production rates are high. However, removal of the plastic binder before sintering is difficult and large shrinkages (up to 40% volume change) occur during sintering.

h) **Other Consolidation Methods**

There are various types of other techniques which can be considered such as:
- slip casting
- vibratory compaction
- spray forming, and
- consolidation by atmospheric pressure.

Generally speaking, the aim of the above methods is to consolidate those materials which are difficult to form by conventional melting and casting; in order to produce the final shapes economically and to control impurities.

These techniques are primarily applicable to refractory powders, such as cemented carbide powders, ceramics etc.

4. Sintering

[References: 1, 2, 3, 5, 9, 12, 13, 14, 15, 16, 17]

Sintering is the process whereby the compacted metal powder is heated in a furnace with a controlled atmosphere to a temperature below its melting point, but sufficiently high to allow bonding of the individual particles. Prior to sintering, the compact is quite brittle and its strength (green strength) is low. In order to facilitate handling, compacts may be pre-sintered by heating them to a temperature lower than the normal temperature for final sintering.

Mechanics of Sintering

The nature and strength of the bond between the particles, and hence of the sintered compact, depend on the mechanisms of:

- Diffusion
- Plastic flow
- Evaporation of volatile materials in the compact
- Recrystallisation
- Grain growth
- Shrinkage.

Sintering mechanisms are complex and depend on the composition of metal particles, as well as processing parameters.

Sintering Parameters

The principal governing variables in sintering are temperature, time and the atmosphere; these are defined as follows:

a) Sintering Temperature

Sintering temperatures are generally within 70 to 90% of the melting point. As temperature increases, two adjacent particles begin to form a bond by a sintering mechanism (solid-state bonding). As a result, the strength, ductility, and thermal and electrical conductivities of the compact increase, as well as its density. This mechanism usually leads to shrinkage of the compact.

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If the two particles are of different materials, alloying can take place at the interface. It is also possible for one of the particles to be of a lower-melting-point material than the other. In that case, the particle may melt and, because of surface tension, the liquid material surrounds the higher-melting-point solid particle. This is known as liquid-phase sintering. An example is cobalt in tungsten carbide to make cemented carbides. In this way stronger and denser parts are obtained.

b)   Time of Sintering
Sintering times at these above mentioned temperatures range from a minimum of about 10 minutes for iron and copper alloys to as much as eight hours for tungsten and tantalum.

Depending on time and processing history, different structures and porosities can be obtained in a sintered compact. This also depends on the extent of diffusion, recrystallisation, and grain growth. Porosity cannot be completely eliminated because of the presence of voids during compaction and gases evolved during sintering. Porosity can be either a network of interconnected pores or cavities, or closed holes.

c)   Sintering Atmospheres
Protective atmospheres are used in the sintering of metallic powder systems and the reasons for controlling the atmosphere during sintering are as follows:

  i)   to control the carburization and decarburization of iron and iron-based compacts;   and
  ii)   to reduce oxides, or to prevent oxidation of the compacts.

An oxygen-free atmosphere is thus essential for the sintering of metallic systems. Although a vacuum is used mainly for refractory metal alloys and stainless steels, the gases most commonly used with a variety of other metals are hydrogen, dissociated or burned ammonia, and exothermic or endothermic-types of

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atmospheres.   Proper control of the atmosphere is essential for sintering and to obtain optimal properties.

d)   Stages of Sintering

Continuous sintering furnaces are used for most production today. These furnaces have three chambers:

i)   Burn-off chamber to volatilise (or "de-wax") the lubricants in the green compact in order to improve bond strength. During this part of the cycle, the component is relatively weak and in the early stages of heating, it is giving off wax or lubricant vapours. De-waxing must be steady and thermal shock must be avoided. Generally, the furnace dimensions and the component loading are controlled to give slow, but progressive heating.

ii)   High temperature chamber for sintering. Once the green part has been raised to the predetermined sintering temperature (usually 60% to 80% of the melting temperature of the lowest melting point constituent), it should be completely free of waxes and will already be partly sintered. The sintering temperature is chosen to balance size changes and density against strength and hardness.

Ordinarily, the density of the compact is increased by sintering and shrinkage will occur. This is associated with increased areas of contact of the particles and reduction of the size of pores in the compact.

iii)   Cooling chamber. This can have a marked effect upon the mechanical properties of the final component. Fast cooling rates generally give harder materials with finer micro structures, whilst slow cooling rates give softer coarser micro structures. Machineability is also affected. Up to 5000 parts/h can be achieved and the time can vary depending on the material composition.

e)   **Sintering Characteristics**

A weighed quantity of a powder is compacted under controlled conditions and the dimensions of the green specimen are recorded. After sintering under controlled conditions dimensional measurements are again taken. The specimen is finally tested for mechanical properties. Sintering characteristics are generally reported in terms of dimensional changes, density changes, final density, and mechanical properties.

5.   Post Sintering Operations

In order to further improve the properties of sintered powder metal products, or to give them the special characteristics, several additional operations may be carried out. Among these are machining, deburring, coining, sizing, forging, impregnation, infiltration; and other processes are possible such as heat treatments, plating and painting.

a)   **Machining and Deburring**

Many components require extra machining processes carried out on them before they are complete. For example, cross holes have to be drilled, holes have be threaded, key-ways cut etc. These features cannot be introduced in the compaction stage. After compaction, sintering and machining; any sharp edges or sharp corners often need removal. This is done by tumbling the components in a large drum filled with ceramic or plastic granules which polish away such sharp edges, this is called "deburring".

b)   **Coining, Sizing and Forging**

Coining and sizing are additional compacting operations carried out in presses. They are performed under high pressure and cause some deformation of parts. The purpose of these operations is to give precise dimensions to the sintered part ("sizing") and to improve its surface finish and strength by additional densification through plastic deformation ("coining").

An important development is the use of preformed, sintered alloy-powder compacts, which are subsequently cold or hot forged to the desired final shapes in closed dies. These products have good surface finish and dimensional tolerances, with uniform and fine grain size and distribution, and a microstructure relatively free of grain boundary segregation and precipitates. The superior properties obtained make this technology particularly suitable for making automotive and jet engine parts that are highly stressed.

c)   Impregnation and Infiltration
The permeability of powder metallurgy products created with controlled porous structures opens up two other possibilities, impregnation and infiltration. Impregnation refers to the forcing of oil or other liquid into the porous network by either immersing the part in a bath and applying pressure or a combination vacuum-pressure process.

When the porous structure of a powder metallurgy product is not desirable, the part may be subjected to metal infiltration. Here a metal of a lower melting point than the major constituent is forced into the product under pressure, or absorbed by capillary action during the sintering stage.

d)   Heat Treatments, Plating and Painting
Powder metallurgy products may also be subjected to more conventional finishing operations such as heat treatment, and surface treatment. If the part is of high density or metal impregnated, conventional techniques are employed. Special precautions, however, must be employed when processing low density products.

Nearly all common methods of surface finishing are applicable, again with some modification of   porous or low density parts. For example, porous components need to be infiltrated with plastic impregnants, so that electroplating techniques can be

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employed. Painting of surfaces of powder metallurgy components is, of course, easily carried out by spraying or dipping. Hot dipping can be carried out as with fully dense components; as long as the process does not change the microstructure in the heating and dipping into the molten metals, zinc (galvanising) or tin (tin plating) etc.

1.8 PROPERTIES OF POWDER METALLURGY PRODUCTS
[References: 1, 5, 6, 7, 9, 10, 12, 15, 16 and 17]

Because the properties of powder metallurgy products depend on so many variables - types and size of powder, amount and type of lubricant, pressing pressure, sintering temperature and time, finishing treatments, and so on - it is difficult to provide generalised information.

Properties can range all the way from low-density, high-porosity parts with a tensile strength as low as 70 MPa; to high-density, minimal-porosity pieces with tensile strengths of 1250 MPa or more, strengths of 275 to 350 MPa being most common.

In general, however, all mechanical properties show a strong dependence on density, with fracture related properties such as toughness, ductility, and fatigue life showing a stronger dependence than strength and hardness. The strength properties of the weaker metals and alloys are often equivalent to the same wrought metals. As alloying elements are added to produce higher strength powders, the resultant properties tend to fall below those of wrought products by varying, but usually substantial, amounts. As larger presses and processes, such as powder metallurgy forging, are employed to provide greater density, the strength properties of powder metallurgy products will more nearly equal those of wrought materials.

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Porosity can also affect physical properties. Electrical, thermal, and magnetic properties all vary with density. On the positive, front, the porosity does promote sound and vibration damping and many powder metallurgy parts are designed to take advantage of this feature.

1.9 SUMMARY: OBSERVATIONS OF THE POWDER METALLURGY PROCESS

1. Selection of Metal Powders

There are two main factors which should be considered when selecting a metal powder:

 i) the alloy required in the finished part; and
ii) the physical characteristics needed in the powder.

2. Classification of Metal Powders

Metal powders are distinguished from one another and classified under the following headings:

1. Particle size and distribution
2. Particle shape
3. Surface profile
4. Particle microstructure, whether solid, porous etc.

3. Successful Production by Powder Metallurgy

This depends on proper selection and control of the following process variables:

a) Powder characteristics
b) Powder preparation
c) Type of compacting process
d) Design of compacting tools and dies
e) Type of sintering furnace
f) Composition of sintering atmosphere
g) Choice of production cycle
f) Post sintering operations required.

1.10 AIM OF RESEARCH

Research activities in the field of powder metallurgy are steadily increasing using traditional powders and powder mixes. There is now a growing need for new types of powders with a variety of properties. Hence there is an interest in composite powders which can combine the characteristics and properties of a number of materials in one powder.

The ability to make coated powders by hydrometallurgical methods has led to the possibilities of a new generation of powders designed to have specific physical and mechanical properties.

The aim of this research was to examine the methods available for coating powder particles, both metallic and ceramic particles, with one or more metallic coatings. The nature and character of these composite powders would be examined and studied fully. After such examination, the properties of compacts made from these composite powders in the sintered and unsintered states would be determined. This would be followed by a microstructural examination of the sections of the compacts and the coated powders. Hence it was hoped to understand the effect of these coatings on the properties of these composite powders, so that some estimate of the alloying or other effects, could be made.

The possible applications of these composite powders would also be considered, such as the possibility of iron particles coated with a copper/tin composite coating, which could be used as a cheap replacement for bronze powders in filters or bearings.

Metal coated ceramics, particularly carbides and oxides, have some applications in the cermet manufacturing industries. Metal matrix composites can also be made from such metal coated ceramics, as well as many porous cermets. These metal coated ceramics could also have uses in flame spraying, plasma spraying and other coating processes which use powders.

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In this research the powders studied were limited to: iron, silicon carbide, tungsten carbide, tantalum carbide and aluminium oxide. The coatings applied used metals which were copper, nickel, cobalt and tin. Only certain combinations were chosen which could have an industrial application, such as for example, nickel coatings on silicon carbide and cobalt coatings on tungsten carbide.

The research also aimed to discover the best operating parameters needed to coat a particular powder successfully, so that this knowledge could be applied to coat any type of powder particle with any type of metal or combination of metals.

CHAPTER 2
LITERATURE SURVEY ON COATED POWDERS

2.1 COMPOSITE POWDERS

1. What are composite powders?

The term "composite" usually designates a combination of materials which form neither alloys nor compounds. This includes both metal-metal and metal-non-metal combinations.

Composite powders usually consist of "coated" powder particles. Each individual particle consists of a core of one particular material surrounded by a shell of another material. The two materials may not necessarily combine to form an alloy or a compound.[5]

2. History of composite powders

The first attempt to produce composite powders was recorded in the patent of Viertel and Egly[21] issued shortly after 1900. The procedures used were either similar to the one developed for the hard metals[22], or called for introduction of the binder in liquid form by dipping, infiltration etc. Gebauer developed such a procedure in 1916[23] and the process was carried further by Baumhauer[24] and Gillette[25] in 1924. Pfanstiehl[26] in 1919 obtained patent protection for a heavy metal, consisting of tungsten and a binder which contained copper and nickel. Around 1937[27], the bearing production was considered to be the first commercial application. In the period from 1945 to 1955, a large number of publications were listed by Kieffer and Jangg[28], in their review on the new developments in powder metallurgy. Protective coatings on uranium dioxide particles for the nuclear fuel industry have also contributed to this field of technology[29].

Between 1960 and 1970 much work was done on the production of coated powders using two different techniques known as vapour deposition and

hydrogen precipitation from aqueous solutions. These two techniques were developed in the USA and Canada by Powell, Oxley and Bloche who described the vapour deposition technique[30], while Meddings, Kunda and Mackin[31] and Evans[32], among numerous others, described the hydrogen reduction method. Sherritt Gordon Mines Limited[33] published a patent specification in 1981, which outlined significant inventions in progress for producing metal-coated composite powder particles. Core materials, which may be utilised with this invention, included metals and non-metals which can be coated with a layer of metal, also known as the hydrometallurgical method.

Meddings[34] mentioned in 1984 that there were more than fifty types of composite powders, consisting of a core and coating, which have been produced commercially. Foba and Coleman[10] in 1986 studied three wet manufacturing methods employed for the production of the coated powders. These methods include electrolysis, electroless techniques and displacement reactions. They investigated the mechanical properties of coated iron powder compacts, and the properties of the resulting compacts were compared to those of compacts produced from pure iron powder. As a result of this investigation it was found that the iron can be coated with metals, which include Cu, Ni, Pb, Co, Sn etc by any of the coating methods mentioned previously. These coatings can affect the properties of given composite compacts, and they can be produced in coherent coverings without precipitating the metal coating as free particles within the iron powder. It was also noticed that the composite powders take up properties of the coating, which controls the mechanical behaviour of the compacted composite powders.

2.2 PRODUCTION TECHNIQUES OF COATED POWDERS

1. Immersion/Displacement Coatings (References 10, 35 and 36)
Metal displacement reactions, also known as immersion plating reactions or cementation, have been known and used for hundreds of years primarily to win metal from leach liquors. This process involves

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the precipitation of a material from a solution of one of its salts onto the surface of a less noble material. Therefore, potentials generated by chemical reaction with the bath at a metal surface may produce electrodeposits without an external source of current. If the substrate metal is electrochemically more active than the protecting metal, the surface of the first may act as a catalyst and reducer, and the resulting process is defined as an "immersion coating".

The general reaction between the cation A^{m+} and a metal B giving the metal A and cation B^{n+} is:

$$nA^{m+} + mB(s) \rightarrow nA(s) + mB^{n+} \qquad (1)$$

The electrochemical nature of such a reaction can be seen more clearly if equation 1 is separated into oxidation and reduction half-reactions. At an anodic site on the surface of metal B, oxidation is taking place:

$$mB(s) \rightarrow mB^{n+} + mne \qquad (2)$$

At a cathodic site, the cation of the second metal is being reduced:

$$nA^{m+} + mne \rightarrow nA(s) \qquad (3)$$
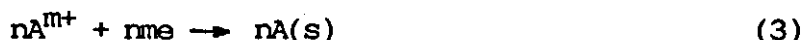
Thus a condition for the displacement reaction to occur is that the electrode (reduction) potential of pure B in the solution of B^{n+} ions is more negative than that of pure A in the solution of A^{m+} ions.

There are a number of metals which are produced by metal displacement from solution, these are: Cu, Ag, Au, Bi, Ge, Cd etc. Certain alloy deposits (e.g. brass) can be produced provided complexants are used to equalise the reduction potentials of the two metals concerned.

The main advantage of this method is that it is cheap and easy to carry out. Limitations with the method are many, such as: non-metallic substrates cannot be coated. Also, the coating thickness is

limited to a maximum of 1 micron because as this process continues, the substrate becomes less accessible to drive the reaction. The risk of corrosion occurs if the solutions are not complex enough due to a tendency of the coating to be noble towards the substrate.

2. Electroless (or Chemical) Plating

Electroless deposition is a chemical reduction process which depends upon the catalytic reduction of a metallic ion in an aqueous solution containing a reducing agent, and the subsequent deposition of the metal without the use of electrical energy[37]. In this respect it differs from electroplating and can be an alternative coating method[38]. Also it differs from other types of chemical plating processes, such as displacement processes, in not involving a chemical reaction with the basis metal [39]. Metal deposition by this technique can be only on a catalytic surface and unfortunately not every metal will act as a catalyst and the choice is therefore limited to those in the eighth group of the periodic table. Unfortunately the successful operation of an electroless process does not end with the selection of the correct ingredients for the bath. The rate of deposition is markedly affected by temperature and pH variables[40]. To overcome this problem, usually complexants are added to the bath in order to maintain the metal ion in solution.

Electroless deposition provides several characteristics superior to electrodeposition processes by producing a more uniform coating, particularly when applied to complex geometrical parts. It may also be applicable to a greater variety of substrates ranging from metals and semiconductors to non-conductors. The use of an auxiliary power supply and the need for electrical contacts is eliminated. The resultant deposits of this technique have unique chemical, mechanical and magnetic properties. Since electroless deposits involve the use of reducing agents and complexants in the bath, it could be considered as an expensive method[40]. This deposition technique can be applied for coating of many metals, metalloids, metal alloys, non-metallic elements, refractory materials and plastics.

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Nickel is a very suitable material for coating, however, under proper conditions, other metals such as cobalt, copper, molybdenum and silver alone or in combinations of two or more metals, can also be applied for coating[41]. All these metals are precipitated from their solutions in the elemental form according to the reaction:

$$Me^{++} + H_2 \rightarrow Me + 2H^+$$
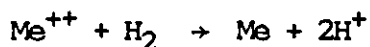
The exception to this general rule is molybdenum, which precipitates as a lower oxide and can be converted to the metallic form by subsequent reduction with hydrogen at about $1000^\circ C$. (See references 36, 41 and 42).

3. Electrolytic Deposition Process

The laws of electrolysis, formulated by Faraday in 1833[39], afforded the first quantitative demonstration of the electrical nature of matter, and have long defined the unit quantity of electricity. Today, unmodified by newer discoveries, they remain among the foundations of physical science.

Electrolysis is carried out in a bath which may consist of fused salts or of solutions of various kinds; in commercial practice it is almost invariably a water solution, where the metals are deposited from solution by electrolysis, which requires electrical contact to be made with the substrate material being coated. In order to deposit coatings upon powders it is essential to maintain the powder in continuous motion.[43]

A fluidised bed with an electrode technique is usually most convenient to use in this respect. The idea of a fluidised bed with an electrode led to much research in the 1970's on the characteristics of the powders produced by this new process. It promised to be an improved method in electrowinning and even electroforming[44], where much greater surfaces, due to use of particles, will make it possible to operate at higher current densities with increased current efficiencies. This

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electrode has so far proved very problematic showing a variation in potential distribution across the bed depth.

Many workers[45,46,47,48] have studied the effect of several parameters, such as bed characteristics (height, expansion) and position of feed electrode, on potential distribution. Several composite powders have been obtained and it was also possible to produce composite coatings electrolytically on ceramic powder dispersions[49]. On non-conducting ceramic powders, thin metal coatings are needed (produced say by electroless coating) first before any electrolytic deposition can occur. Composite powders produced by this method include: copper on iron, nickel on cobalt[50], nickel on iron[51] and silver on copper, silver on iron [52], and many other combinations.

4. Mechanical Coating (References 3, 5 and 7)

The procedure for this method involves milling relatively compact particles of a hard metal with a small amount of a finely powdered soft material. It is the procedure generally used for the production of cemented carbide powders[5]. Tungsten carbide and cobalt powders are frequently ball-milled and the product used for preparing cemented tungsten carbide with cobalt as the binder phase. This could be applied for preparing composite powders, especially when quality of coating and obtainable thickness are not important criteria. The equipment used for milling these hard and brittle materials can be of many types, varying with the toughness of the metal. It may consist of one mill, or a combination of several, and may utilise rod mills, ball mills, impact mills, disk mills, stamps and rolls. To minimise contamination of the powder, the wearing parts of such equipment are provided with hard and durable facings. Undue heating and plastic deformation of the powder is sometimes prevented by water-cooling of the grinding surfaces.

5. Chemical Vapour Deposition (CVD)

This is one of the oldest known methods for depositing metals, alloys or compound deposition from the vapour phase onto a solid substrate by some form of chemical reaction[53,54]. This process has been used since 1895 for producing nickel from the thermal decomposition of nickel carbonyls. The nickel carbonyl vapour decomposes into carbon monoxide and metallic nickel, which deposits upon the surface to be coated at high temperatures, usually between 400°C and 800°C. The following reaction can occur:

$$Ni(CO)_4 \rightarrow Ni + 4CO$$
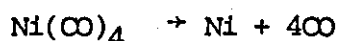
Cline and Wulff in 1954[55] coated various ceramic powders with nickel by this reaction. These workers were involved in the development of cermets for high temperature use and conceived the idea that a complete film of metal around each individual ceramic powder particle might lead to improved physical properties of the sintered cermet body. Powders such as alumina, silica and silicon carbide were coated with complete films of nickel by this technique. Oxides and hydroxides can also be deposited, as in the reduction of oxides with hydrogen, to yield metal or alloy coatings[56]. Chemical vapour deposition is particularly useful in industry today and is an important technique for coating of refractory materials and deposition of refractory coatings, both metallic and ceramic. These coatings find their application in the finishing of cutting tools and drill bits for increased wear and corrosion resistance.

6. Physical Vapour Deposition

This technique is similar to chemical vapour deposition, but the coating usually involves only the deposition of atomic species without any kind of chemical reaction. There are a number of techniques to generate the plating material and the most common of these are either:

Ion sputtering, in which the material is eroded from the cathode when a glow discharge is struck in a partial vacuum; or by evaporating

material off the coating metal or alloy and subsequent deposition on a substrate held at a lower temperature[57,58]. Vapour deposition techniques usually operate under low pressure conditions and careful control of both pressure and temperature gradients between source and substrate are necessary for successful operation[59].

The application and importance of this technique are similar to the chemical vapour deposition. Because of the lower vapour pressures of metal, deposition rates are usually lower than for chemical vapour deposition. Quality and purity of deposits are claimed to be better, and are more easier to control than for chemical vapour deposition.

7. Other Coating Techniques

a) Dip Coating

Among the metal coats obtained by dipping into a liquid, tin and zinc coating are most frequently used in the metal industry; followed in frequency of use by aluminium and low melting alloy coatings. The process involves the substrate being immersed or passed through the molten metal (with low melting point) after suitable pretreatment. This technique is covered in the book on modern electroplating by Lowenheim[39].

b) Conversion Coatings

These techniques involve changes between the surface material and the coating by chemical conversion[60]. The processes which use this treatment include phosphating, carbonitriding, nitriding and carburising. These and others have been described in the book on protective coatings for metals by Burns and Bradley[61].

2.3 THE APPLICATIONS OF COATED POWDERS

Despite the fact that composite powders are relatively new materials, many papers have been published suggesting possible applications in various fields.

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1. Alloys

Coated powders can be used for alloy production by powder metallurgy routes. It is possible to alloy nickel-coated copper powder faster and more efficiently than mixed nickel and copper powders[62]. By conventional methods, adding phosphorus to molten nickel, it is not possible to produce nickel/phosphorus alloys containing more than about 13% phosphorus. By solution deposition methods, it is possible to coat phosphorus powder with a complete coating of pure nickel. The coated powder so produced is quite stable up to 325°C.

In some cases the application of a coated powder is a function of the two components. The sintering of compacts of tungsten or molybdenum powders can be accelerated by the presence of small amounts of nickel. Under normal conditions it is difficult and time consuming to sinter molybdenum powder to nearly full density, but by use of this phenomenon of "activated-sintering" it is possible to achieve this density in much shorter times. Powder mixtures can be used, but it is found that better and more reproducible results can be obtained by depositing the necessary nickel on the molybdenum powder as a coating[63].

2. Porous Metal Strips

Porous metal strips are widely used as filter media or as porous electrodes for fuel cells or nickel-cadmium batteries. These strips are usually made by the compacting and sintering of powders. Composite powders offer an alternative method for porous strip production. Suitable composite materials are metal coated fibres of glass, graphite and polyurethane foam, or graphite powder.

Nickel coated graphite is particularly suitable and unique in this respect. It can be pressure or roll compacted, sintered, hot and cold rolled to any specification and the carbon can then be removed from the fabricated material. This leaves a porous strip, whose final pore size can be controlled by the size of the graphite particles initially employed. Butts[64] describes the application of Ni/Al composite powder

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for preparation of a low density and cellular metallic material, which has good abradability characteristics, is resistant to oxidation and erosion, against the flow of hot and high velocity air. This material will expand during processing, improving contact and bonding within itself and within the substrate. The nickel aluminide product has good bonding with the substrate and does not expand when exposed to high temperatures. The availability of these materials and decreased price has led to a promotion of their usage.

3. Abradable Seals

Nickel/graphite composite powders find an application in a number of aircraft turbine engines as abradable seals. The function of the seal is to preserve the pressure differentials through the various turbine stages and in this way increase the efficiency of the engine. In order to achieve this, the seal is adjacent to the tips of the turbine blades and varies and is located so closely, that the moving blade is permitted to touch the seal and wear a path in it. The seal must be abradable, physically strong to preserve the pressure differential and resistant to oxidation and erosion conditions. These seals may be built up from successive layers of flame or plasma sprayed deposits or by a sintering technique.

Fustukian, Mehta and Evans[65] have studied the application method and properties of nickel/graphite deposits in turbine engines for abradable seals applications. They concluded that the properties of the coatings were sensitive to powder composition and thermal spray distance.

4. Dispersion Strengthened Materials

In a paper published by Kunda and Evans[30,32,66] it mentioned that the submicron particles of various oxides were activated by palladium chloride and coated with nickel and the products were then sintered and consolidated into dense strips. Hardness tests on strips at various temperatures showed that specimens prepared from composite powders containing the oxide phase ($\sim$ 3% by weight) retained its

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hardness at elevated temperatures indicating the possible use of composite powders for preparation of dispersion strengthened materials.

5. Low Friction Materials

Low friction materials are used primarily in engines to reduce the friction of the moving parts. In many instances lubricating oil cannot be applied because of the high temperatures. Composite powders having low friction cores appear to be ideal materials for such applications. Many composite powders having low friction cores, have been prepared and flame or plasma sprayed onto a mild steel substrate without difficulty. The results showed that all of these composite powders could be sprayed using standard flame or plasma spraying equipment[67,68,69]. Powders with particle sizes ranging between 44 and 105 microns were more suitable for flame spraying, while those between 5 and 40 microns were suitable for plasma spraying. Powder particles below 5 microns were undesirable. However, to fully appreciate the use of composite powders as low friction materials, more research work is required.

6. Hard Materials

Extensive investigations on hard facing materials are being carried out in the USA, Japan and Europe, particularly in Austria. These research activities emphasise the role of such materials in modern technology.

Ettmayer and Kieffer[70] and Schwarzchoff and Kieffer[71] have published data on the hardness and melting temperatures of some metal carbides, nitrides and boride compounds. Most of these materials are suitable for applications as hard facing materials and for the manufacture of cutting wheels and cutting tools. Many publications[72] enumerate various methods by which carbides are produced such as: synthesis from elements, reduction of metal oxide by carbon, deposition from gaseous phase, electrolysis of molten salts and chemical precipitation. Similar methods can be employed for the production of nitrides and borides.

2.4 THE PROPERTIES OF COATED POWDERS

The chemistry of the two or more components of a coated powder can obviously vary throughout such a wide range so that it is not possible to generalise easily upon the behaviour of these powders. However, these powders do have some general physical characteristics in common.

Generally, both metallic and non-metallic core powders with particle sizes ranging from less than 1 micron to greater than 100 microns can be coated. Nickel or cobalt, and sometimes copper, is usually used as a metal coating and ranges in thickness from 2 to 3 microns. This thickness range allows adequate particle suspension within the solution. Consequently powders may be produced in which the coating weight comprises 20 to 99 wt%, and core weight represents 1 to 80 wt%.

More than 50 types of composite powders consisting of a core and coating have been produced commercially[34]. Such composite powders are inherently heterogeneous, in that each particle consists of a core of one material and a coating of another. The powder as a whole, however, is homogeneous, because no segregation of the two components is possible during transportation, storage, or handling. The apparent densities of the products are dictated primarily by the specific gravity of the components, the particle shape and size of the core, and the thickness of the coatings, while the screen size distribution of the product is dependent on the quantity of core and the agglomeration of the powder during the deposition of the coatings. The relationship between thickness of coating and the fraction of the total weight represented by the coating is fixed by both the surface area of the core material and the density ratio of the core and coating materials[29,32,33,41].

2.5 THE ADVANTAGES OF COATED POWDERS OVER UNCOATED POWDERS

Coated powders have several distinct advantages over uncoated powders, such as their homogeneity within the powder metallurgy product is increased with coated powders. They have the advantage of joining two diverse materials uniformly by the coating combination which justifies their use in many tribologic applications. These vary from hard and wear resistant materials to low friction bearing materials. Another advantage is the possibility of obtaining high dimensional tolerances on products which can be manufactured at lower cost. An example is the use of less expensive nickel coated glass particles in the fabrication of porous electrodes for air furnaces.

The coating of Cu-Sn alloy on iron particles could lead to the inexpensive production of bronze alloys instead of mixing expensive copper and tin powders to produce the same alloy.

Last, but not least, the main characteristics of coated powders, which could also be considered a great advantage over uncoated powders, is that very thin coatings of metals on high temperature alloy powders are known to activate their sintering and produce high density materials in shorter times and/or at lower temperatures, resulting in better mechanical properties[5,3,17,73].

2.6 CHEMICAL PROCESSES BASED ON POWDERED SYSTEMS

1. Fluidised Bed Processes

The phenomenon of fluidisation of powders has been extensively researched over the last fifty years. It was not until 1952 that research workers at the German firm Knapsack-Griesheim developed this principle as a method of dip coating metallic articles with thermoplastic powders. Within ten years this process had become widely accepted as an industrial coating operation and extended to include the fluidised bed application of thermosetting powder coatings[74].

This technique was also used in powder metallurgy in order to deposit coatings upon powders, where it is essential to maintain the powder in continuous motion, particularly in deposition of metal, non-metal or alloy coatings. Vapour reacting coating deposition represents "gas fluidisation"[46,75,76,77], while the wet method represents the "liquid fluidisation", which includes the electroless and displacement coating methods.

2. Fluidised Bed Electrode

The application of the fluidised bed electrode (FBE) method has been the object of considerable academic[78,79] and some industrial[80,81] interest. As with several other recent advances in electrochemical engineering, FBE resulted from the adaptation of a well established device in chemical engineering to mass transport and scale-up problems in electrochemical reactors. While the physics of its operation at the microscopic level are still a matter of some contention[82,83], there is agreement that its microscopic performance may typically yield (in cross sectional) limiting current densities which are more than two orders of magnitude greater than at a planar electrode operating under comparable electrolyte flow rates[84,85]. This reflects the benefits of a high specific area and high rates of mass transport to the particle surface within the bed.

The main advantage of the FBE over its fixed bed counterpart, where during electrodeposition the bed particles grow as discrete entities, has encouraged its use for electrowinning and for the treatment of metal-bearing industrial effluents. Correlations exist in the literature[86,87] to describe the operation of the FBE, under pure mass transport control.

3. Fluidised Bed Chemical Reactor

There are two types of fluidised bed systems which can be identified as aggregative fluidisation and particulate fluidisation[74,75]. The first tends to be associated with the gas fluidised beds, whilst the second tends to be associated with the liquid fluidised beds. Workers such as Steward and Davidson[88] and Leung[89] agree that the ratios of a

density of solid particles i.e. P_s/P_f reflects on the behaviour of the bed of fluidised particles, whether they act aggregatively or in a particulate manner. Thus a bed of tungsten beads fluidised with water $(P_s/P_f + 19.3)$ shows the formation of bubbles and slugs, while light powders fluidised with high pressure gases can be expanded considerably before bubbling occurs.

Other workers like Richardson and Zaki[90] established an important quantitative relationship that is widely applicable to particulately liquid-solid fluidised beds. This relationship is between the superficial fluid velocity and particle bed voidage, taking into account the wall and interparticle frictional effects.

This relationship is as follows:

$$\frac{U}{U^*} = \varepsilon^n$$

where U = superficial velocity of liquid

ε = voidage

U* = sedimentation velocity in liquid medium under gravity

n = a specific constant.

In theory and practice, fluid viscosity and density, and bed particle size, size distribution, density and shape are all important parameters of fluidisation and fluidisation technology[74,75].

A fluidised bed offers a means of burning coal and other solid fuels efficiently, but using smaller equipment which gives less pollution than more conventional methods of combustion; and so provides an attractive alternative design for power generation purposes. Fluidised bed powder coating[76] is another commercial process, besides fluidised heat treatment furnaces in metallurgical processes[74].

4. Mechanically Agitated Vessels

The mechanical agitation process is used in many chemical processes, where it involves a suspension of solid particles agitated in vessels. This may be achieved either by rolling the vessel or keeping it in motion; or by using impellers or stirrers to disperse or suspend particles in a fluid media[91]. It is desirable that complete suspension conditions prevail both for rapid mass transfer and operational considerations, especially in systems where a reaction occurs between the fluid medium and the particle surface[92].

When a reactive fluid is contained in a vessel and an impeller is used to agitate the particles in that vessel, then the speed at which particles just become fully suspended should be a maximum value with respect to available surface area for mass transfer and power consumption[93].

In work done by Zwietering[94], he proposed an equation for optimum impeller speed from dimensional analysis, and defined the state of complete suspension as that at which no particles remain at rest on the bottom of the vessel for longer than 1 to 2 seconds.

$$V_m = \frac{S D_p^{0.2} \; \mu_L^{0.1} \; (g\Delta\rho)^{0.45} \; B^{0.13}}{\mu_L^{0.55} \; D^{0.85}}$$

where:
- V = optimum impeller speed
- S = constant which is a function of the system's geometry
- D_p = particle diameter
- μ_L = fluid viscosity
- B = solid concentration
- g = acceleration due to gravity
- $\Delta\rho$ = difference between solid density and fluid density
- D = stirrer diameter
- ρ_L = fluid density.

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However, this estimation for V_m was not in agreement with some workers such as Narayan et al[95] and Weisman and Efferding[96], but others such as Neinow[97] and Baldi[98] agreed under specified conditions. The latter indicated that in particular, the ratio of the impeller distance from the tank bottom to the impeller diameter was a more significant parameter than Zwietering had in his work. Baldi's analysis also found another significant conclusion that the average particle size could be substituted straight into the equation without any corrections for particle size distribution.

2.7 COATED POWDERS IN INDUSTRY

The preparation and application of coated powders, sometimes referred to as composite powders, is well known and widely reported in the literature[1-7].

Despite the fact that composite powders are relatively new materials, many papers and patents[33,99-103] have been published suggesting possible applications in various fields. The studies by Lund, Irvine and Mackiw[62] confirmed and showed the advantage in using composite powders in the preparation of alloys. They have studied the sintering and alloying characteristics of nickel coated copper powders, copper coated nickel powders and mixtures of nickel and copper composite powders. The results showed that the composite powders produced a compact of a much higher density. formed alloys at a much faster rate than mixtures of elemental powders and did not segregate during the processing.

The alloying characteristics of Ni-Co-Cu, Ni-Co-Mo compacts prepared from composite nickel coated powders and mixtures of elements were also studied by Lund, Krantz and Mackiw[63].

Another application of such composite powders is in commercial use in a number of fields. For example, nickel coated graphite powders are used in the formation of abradable seals for gas turbine engines. Cobalt coated tungsten carbide powder is flame sprayed onto knife

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blades to form hard, wear resistant cutting edges. Nickel coated aluminium powder[104,68,69] is flame sprayed onto various substrates to provide a strongly adherent bond coat in the preparation for further coating.

A patent by Butts[105] describes the application of Ni/Al composite powder for preparation of low density, cellular metallic material which has good abradability characteristics and is resistant to oxidation and erosion against the flow of hot and high velocity air. This material will expand during processing, improving contact and bonding within itself and with the substrate. The nickel aluminide product has a good bonding with the substrate and does not expand when exposed to high temperature.

Another dimension to composite powder technology is in applications for the activated and liquid phase sintering of refractory metals and ceramics[73].

Ettmayer and Kieffer[70] and Schwarzhoff and Kieffer[71] have published data on the hardness and melting temperatures of some metal carbides, nitrides and boride compounds. Most of these materials require a coating before they are suitable for application as hard facing materials and for the manufacture of cutting wheels and cutting tools.

Similar applications by other publications[72,106] enumerate many methods by which carbides are produced: synthesis from elements; reduction of metal oxide by carbon; deposition from gaseous phase; electrolysis of molten salts; and chemical precipitation. Production and availability of these materials and decreased prices has led to a promotion of their usage.

Other potentially useful applications of coated powders will be in the use of safe metals for coating toxic metals like beryllium, may also be advantageous.

Protective coatings on uranium dioxide particles can be of great benefit for the use in the nuclear fuels industry[29,13].

The bearing and filter manufacturers may also consider Cu/Sn coated iron powder which could have some advantages as a replacement for expensive bronze powders.

Probably the main use of coated powders will be in the field of metal matrix composites and in cermet manufacture. The addition of SiC to aluminium alloys is already a possibility to make a stronger product, is just one example. The use of WC and Co in cemented carbides may be another field where coated WC particles may be useful in the production of WC/Co cermets and similar hard materials.

This research therefore is an attempt to examine some of the possibilities in this area of industrial powders and hopefully to show the usefulness of composite powders.

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CHAPTER 3

EXPERIMENTAL DETAILS

3.1 PARTICLES AND COATING PREPARED AND EXAMINED

This chapter describes the experimental work carried out on coating powder particles with metallic coatings and the various mechanical properties achieved by compacts made from them. This chapter is split into two parts as shown below.

a) Deposition of copper and copper-tin alloy on iron particles with a size range of -150 +125 μm, using a chemical displacement method in fluidised bed apparatus, was carried out producing the following compositions:

1. Fe - 1.5 Cu wt% (stirred bath)
2. Fe - 7.1 Cu wt% (Fluidised bath)
3. Fe - 8.6 Cu wt% (Fluidised bath)
4. Fe - 9.8 Cu wt% (Fluidised bath)
5. Fe - 0.7 Cu - 0.2 Sn wt% (stirred bath)
6. Fe - 3.5 Cu - 1.3 Sn wt% (stirred bath)
7. Fe - 4.3 Cu - 2.1 Sn wt% (stirred bath)

b) Electroless deposition of nickel, copper and cobalt on alumina (45 μm); silicon carbide (29-250 μm, and 400 μm): tungsten carbide (45-150 μm) and tantalum carbide (45 μm) particles was carried out producing the following compositions:

Copper coatings:

1. SiC   (29 μm) - 0.7, 8.1, 35, 62.4 wt%
2. SiC   (250 μm) - 4.3, 14.3 wt%
3. SiC   (400 μm) - 5.3 wt%
4. Al_2O_3 (45 μm) - 3.4, 7.1, 11.7 wt%
5. TaC   (45 μm) - 12.8 wt%
6. WC (45-150 μm) - 8.74, 35.0 wt%.

Nickel coating:
1. Al_2O_3 (45 μm) - 1.9, 3.1, 6.34 wt%
2. SiC   (29 μm) - 1.3, 8.33 wt%
3. SiC   (250 μm) - 3.5 wt%
4. SiC   (400 μm) - 7.4 wt%
5. WC (45-150 μm) - 5.71 wt%
6. TaC   (45 μm) - 4.8 wt%.

Cobalt coating:
1. WC (45-150 μm) - 12.71 wt%
2. SiC   (29 μm) - 0.74 wt%.

Most of the coated powders mentioned above were pressed, and their properties examined to determine the effect of the coatings on the powders during pressing and ejecting.

Properties measured were compaction pressure, density and density changes, hardness of the compacts, and strength and elastic modulus of pressed compacts.

The surfaces of the coated powders and the microstructures of the components were also examined.

These data were collected to assess the effects of the metal coatings on the behaviour of the substrate particles when compacted into a component.

3.2 EXPERIMENTAL METHODS

1. Coating of Iron Particles
A West German iron powder (Mannesmann WPL 200) made by water atomisation of size fraction -150 +125 μm, was used for this research, the specifications of which are given in Table 2.   Details of the Mannesmann process can be found in a review by Jones[107]. This particle

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size distribution was obtained using the standard sieve analysis method. A sintered PVC micro-porous sheet material (Vyon F) was used as the fluidised bed support for the powder bed in most of the fluidised bed electrolytic plating experiments. Appendix 1 contains information on the manufacturers and some technical details on the type of Vyon material used.

2. Plating of Powders and Handling Techniques
a) Pretreatment of powders
The powders were washed by deionized water and alcohol successively, then the particles were oven dried at 50°C for 20 minutes. This hopefully removed any dirt and oils or greases from the powder samples.

b)   Fluidised bed technique
The fluidised bed vessel consisted of a glass tube with flanges at both ends with an internal diameter of 10 cm and the outer diameter of the flange was around 15 cm. The porous polyethylene disc (Vyon F material) was held between two flanges and used to produce and ensure uniform fluidization. Two semi-circular wooden blocks were designed to fit on the top side of the flanges to join them neatly.

Figure 4 illustrates the cell configuration of the fluidised bed circuit with bath reservoir, pump and flow pipes.

The joints of the flange were held together and rested on a flat wooden base, in which six screws and pins were used for bolting the flanges together, three on each semi-circular block. The flexible set up was made in this way for easy assembly and dismantling.   An additional O-ring was fitted between the flat wooden base and the bottom flange to reduce the risk of the bottom flange being damaged. In order to avoid leakage of the fluidising medium and powders being entrapped between the bed support and the glass flanges, silicon rubber was used and filled the ends of the flange joint neatly. Although this arrangement was very well sealed, the whole assembly had

to be taken apart and thoroughly cleaned after each operation, which was a disadvantage. This was always done after each run to prevent subsequent contamination of the baths and also scratching of the glass flanges by powders.

The characteristics of this fluidised vessel were studied using iron powder of particle size -150 +125 μm with water as the fluidising medium. Some observations and results were obtained during operation of the fluidising bed are shown in Figure 6 and Table 4.

Only one type of iron particle was used as mentioned above. Due to the difference in particle sizes, it was observed that there was a loss of the finer particles into the flow circuit of the fluidising vessel. These fine particles were carried through the pump and collected in the chamber below the bed support. This means that in future studies, a filtering system needs to be used in this fluidised bed apparatus. All experiments on powder plating in this fluidised bed were carried out at room temperature.

c)   Mechanically stirred vessels
Two techniques were used to stir the particles in the plating bath. The first involved a steel rod and a stirrer positioned in the plating bath and driven by a variable speed motor. The rod consisted of a three bladed stirrer; both the rod and the stirrer were made inert to plating solutions and could withstand temperatures up to 90°C. This was done by using PTFE tape wrapped around the cleaned rod and stirrer, in order to make sure that no surface was exposed. A satisfactory performance was achieved for all pH levels used (from 4.5 to 13), at room temperature.

Pretreated powders were added to the stirred solutions with continuous stirring. The solutions and the powder were then held in a Pyrex glass vessel at room temperature.

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The second method was by manual stirring using a glass stirrer. The same procedure mentioned above was carried out and a satisfactory coating was achieved.

d) Bath compositions

The composition of solutions used for copper and copper-tin alloys are shown in Table 5. These solutions were used for coating by the displacement method, using the fluidised bed technique or the vessel bath technique.

e) Displacement plating in the fluidized bed

The iron powder particles of size fraction +125 -150 μm were plated and carried out in an acid copper sulphate solution, see Table 5a and Figure 5. Three different concentrations of copper sulphate solution were used. The pH was 3 with a bed weight of 100g of iron particles giving a packed bed height of about 0.5 cm. It was observed that the deposition of copper ions was confined to the lower half of the powder bed when the flow velocity was below 0.53 cm/s, and before the plating solution reached the top of the bed. This was only noticed with more dilute solutions containing lower copper concentrations. However using higher flow rates gave more uniform results. Maximum flow velocities used were about 2.3 cm/s; with this flow velocity it was noticed that the particle bed expanded over 100%. Some characteristics of iron particle movements in the fluidized bed system are shown in Table 4 and Figure 6, as previously mentioned. A problem was observed only when the microporous bed support had blockages of the pores after prolonged usage. This was due to deposition of copper coating on fine powder particles trapped in the pores. Only displacement copper coating was carried out in this fluidised bed system. The procedure for the deposition of the copper coating on iron powder particles by this method is shown in Figure 5.

f)   Displacement plating process in a mechanically stirred vessel

Coating deposition by the displacement method for a copper-tin alloy was carried out in a mechanically stirred vessel. The solution composition for this method of plating in bath is shown in Table 5b. The basic operation and the apparatus have already been described in Section (c) above. Any quantity of powder up to 300g could be coated in quick batches. Agitation was achieved successfully using up to 3 litres of coating solution. Generally this apparatus is very simple and was used for preparing many samples of coated powders for subsequent examination and testing. Different percentages of coating on iron particles were obtained by this method. The procedure for the deposition of the copper-tin alloy coating is given in Figure 7.

3.3   COATING OF NON-METALLIC (CERAMIC) PARTICLES

1.   Powders Used

Four different ceramic powder particles were used. These were: alumina powder of 45 μm average particle size; tantalum carbide powder of 4 μm average particle size; tungsten carbide of particle sizes between -150 and 45 μm; and silicon carbide of three size fractions; 29 μm, 250 μm and 400 μm. TaC, WC and SiC powders containing free carbon impurities were used as received from Goodfellow Metals Ltd[152]. No attempt was made to analyse metal impurities for these ceramic powders.

2.   Bath Composition

Three kinds of plating solution were used: copper, nickel and cobalt. The composition of solutions for electroless plating for these metallic coatings are shown in Table 6.

3.   Pretreatment of Powders for Electroless Plating Process

All powders were thoroughly washed before introduction into the plating baths. The sequence of operations consisted of sensitizing and activating. The compositions of solutions for pretreatment are shown

in Table 7. After each stage the particles were washed with distilled water. These treatments are important especially for the activation of non-catalytic substrates so that they can be coated readily. The pickling with HCl for all these ceramic powders was around 3 minutes before washing with stannous chloride solution for 3 minutes; followed by treatment with palladium chloride for 2 minutes. During these treatments, palladium chloride is adsorbed on the surface and is reduced in the bath by hydrogen to the metallic form, thus forming catalytically active sites on which the metal precipitates or plates preferentially.

4. **Electroless Copper, Nickel and Cobalt Plating Process for Ceramic Particles**

After the previous pretreatments, the particles were washed with distilled water. The glass container was then changed to a new one before plating with the coating solutions in order to prevent the metallic deposition on the glass wall. The desired temperature was maintained by a water bath. All metallic plating baths were agitated manually or mechanically. The pH of the plating solutions were measured at room temperature. Electroless Cu and Ni coatings were deposited on Al_2O_3, TaC, SiC and WC powder particles, whereas the cobalt was only deposited on WC and SiC particles. The procedures for the deposition of these three plating solutions by electroless techniques are shown in Figures 8, 9 and 10.

3.4 CHEMICAL ANALYSES OF COATINGS

As explained above the coated metal powders were washed with distilled water several times, followed by filtration, then an alcohol wash, and finally drying in an oven at $50^{\circ}C$. The composition of the composite powders was then chemically analysed. This analysis was carried out on suitably diluted solutions on an Instrumental Laboratory Ltd 151 Atomic Absorption Spectrophotometer following the relevant instructions set out in the working manual. Around 0.02g of coated powder was weighed from each batch and then dissolved in 5M nitric

acid. This acid solution was used for dissolving iron powders coated with copper and copper-tin alloy compositions. Similar acid solutions were used for dissolving metal coatings of nickel, copper, cobalt on: alumina, tungsten carbide, silicon carbide and tantalum carbide.

The electroless nickel deposition is usually accompanied with phosphorus as a result of using the hypophosphate as a reducing agent; but only very small percentages of P were found within the electroless nickel deposits, which in effect made no difference to the assumption that the deposition was pure Ni.

3.5 COMPACTION OF COMPOSITE POWDERS

1. Lubricant Characteristics
Zinc stearate was used as the lubricant for all powders, the properties of which are given in Table 3. This lubricant in all cases was applied as a dilute solution of zinc stearate in diethyl ether, and was sprayed into the die cavity. For good lubrication the punches and die walls had to be cleaned after each compaction to remove loose powders, and the punches and die lubricated with fresh lubricant for each compaction. Before the die was filled with powder, the lubricant was allowed to dry on the die walls, for one minute.

2. Powder Compaction
Compaction of powder was carried out on the Instron Universal Testing Machine in an 8 mm diameter tool steel die. This apparatus and complete set up is illustrated in Figure 11a. Figure 12 shows the section of this punch and die set. To compact the powders, the die was filled with a certain amount of powder. The mass levels were estimated from practice samples produced to determine maximum load levels to be used for the compaction process. The die was then placed on the load cell, the top punch was inserted into the die, which was then pressed using a crosshead speed of 2 cm/min. At this stage, the die was held

by the wooden blank which is indicated in Figure 11b. The top punch was then preloaded to 4.5 kg, while the maximum full scale on the Instron was set at 10,000 kg.

After preloading, the blank wood was removed, and the powder was then loaded gradually by the top punch. Once the load was reached, it was held for 3 seconds, and then released. The compact was ejected with the aid of a hollow cylinder placed on the die. The maximum compression load value and the crosshead movement were automatically recorded on an X-Y chart recorder and the compaction stress calculated.

3. Comparison Between Lubricated and Unlubricated Compaction
Samples of iron powder and iron with copper coatings were taken in the size range -150 +125 μm. These were pressed at identical loads, with and without a zinc stearate lubricant so that a comparison could be made of the difference in compaction behaviour between coated and uncoated powders. This would give an indication whether the metal coatings aided compaction or not. This was only done for iron powders and iron powders coated with 1.5, 7.1, 8.6 and 9.8 wt% Cu coatings (see Tables 25-29).

A similar test was carried out for the Cu-Sn coated iron powders, but only for the 3.5 wt% Cu - 3.1 wt% Sn and 4.3 wt% Cu - 2.1 wt% Sn coated powders (see Tables 30 and 31).

3.6 SINTERING OF POWDER COMPACTS

The sintering process for all green compacts was achieved in a commercial furnace which was made available by GKN Ltd, Boundbrook, Lichfield. The type of furnace used was a continuous belt one. For the copper and copper-tin alloys, coated iron powder compacts, these were sintered at 800°C for 9 minutes. Whereas for the ceramic powders coated with nickel and copper the sintering temperatures were 1000°C, for 10 minutes. The sintering atmosphere at 800°C was carried out in

dissociated ammonia, while sintering at 1000°C was carried out in an atmosphere of 25% H_2 - 75% N_2.

All these compacts were weighed on a chemical balance to an accuracy of 0.0001g and all dimension measurements were taken using a micrometer of 0.01 mm accuracy; to obtain the compact densities before and after sintering.

Sweating was observed and non-uniform densification occurred and recorded as a result of sintering. This was observed particularly for all compacts containing copper-tin alloy, while cracks and roughened surfaces occurred on some ceramic compacts containing nickel deposition on alumina. For nickel coated SiC compacts, the dimensional changes observed were considerable particularly for compacts produced at low compaction pressures.

3.7 MICROSCOPIC EXAMINATION OF COATED POWDERS AND COMPACTS

1. Preparation of Materials for Examination
a)   Preparation of samples of all metal composite powders
The examination of cross-sections of the coatings on particles under the electron microscope was done by sprinkling some particles of powder in a metal ring with 25 mm internal diameter. This ring was seated on a PTFE sheet which was held down on a small piece of glass to ensure a flat surface for the particles. The metal ring was then filled with resin up to 3/4 of the height of the ring. This arrangement was then transferred to a vacuum container. The first vacuum cycle was carried out in less than one minute. At this stage air bubbles were formed in the bulk of the resin, which tried to escape from the resin up to the surface. This cycle was repeated three times in order to ensure that all these bubbles were removed.

Care had to be taken to make sure that too much hardener did not escape from the resin. After that the whole set up was taken out and

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left for at least 24 hours at room temperature. The specimen was then peeled off the PTFE sheet gently and subjected to standard grinding treatments which were followed by a polishing stage.

Starting on silicon carbide paper with BSS 240 mesh grit, the specimen was ground very lightly and then repeated on 400 mesh and 600 mesh grit papers, and then finished on 1200 mesh grit paper. Water only was used as lubricant during all these grinding operations.

For polishing a cloth with 6 µm diamond particles was used, followed by 1 µm diamond particles as a final polish. Blue and pink lubricant solutions were used for polishing respectively. Care had to be taken to make sure that not too much pressure was applied, both during grinding and during polishing in order to prevent powder particles from being pulled out from the resin matrix.

For pressed and sintered compacts, the only difference was in the time of the vacuum cycle carried out which was applied for 10 minutes longer than loose powders.

At each stage of preparation, the sections being prepared were subjected to ultrasonic cleaning in alcohol, which was carried out for two minutes in each case.

b) Preparation of samples of metal coated ceramic powders
Powder samples for ceramic coated particles were prepared by sprinkling some powder in a one inch plastic mould. The whole set consisted of two pieces, an O-ring and a flat base.

The mould was then three-quarters filled with "Scandiplast" resin mixture, used especially for hard materials (see Appendix 2). The specimens were then left to set in the moulds until they cooled down to room temperature which took about 30-40 minutes.

No further treatment was required, such as a vacuum treatment, for this preparation as no air bubbles were formed.

The plastic mould was then peeled off the base and the whole set matrix gently pushed out from the O-ring plastic mould.

In a similar manner as described earlier under this section, the specimen was then subjected to both grinding and polishing operations. The same method was used for mounting green and sintered compacts for grinding and polishing.

2. Optical Microscopical Examination

All samples prepared were examined and photomicrographs were obtained on both black and white film and coloured slides. This was carried out using a reflected light optical microscope, where the maximum magnification used was about x100.

3. Scanning Electron Microscopy Examination

Examinations using these techniques were carried out on powder surface structures of uncoated and coated iron and ceramic particles. Also the fracture surfaces of uncoated and coated iron green compacts were studied using the Cambridge Stereoscan S2A Electron Microscope.

An attempt was made to plot X-ray maps of nickel coating on both silicon carbide and tungsten carbide particles sectioned in the loose state. This was done to study and detect the amounts of phosphorus present in the nickel coating produced by the electroless method using hypophosphite as a reductant. This was difficult due to formation of free carbon on the surface of the sample during the examination and since the surface of these hard particles were not smooth enough, because they are difficult to polish, the scanning results were difficult to obtain.

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4.   Electron Probe Microanalysis

This was carried out using the Cambridge Microscan 5 machine. Analyses were carried out only on samples prepared from iron coated powder both with copper and copper-tin alloy. These polished samples were already mounted in rings less than 1 cm thick and with the correct diameter. Scan lengths were between 0.5 and 1 mm long. Chart speed was 10 mm per minute, while scan speed was 10m per minute. Examinations were carried out on loose powders, sintered and green compacts.

3.8   MEASUREMENT OF FLOW RATES AND APPARENT DENSITIES OF POWDERS

The flow rates and apparent densities were measured for iron particles and Cu, Cu-Sn coated iron particles only. These were produced by the displacement technique in fluidised bed and bath systems.   A Hall flowmeter was used for measuring these flow rates and apparent densities[108].

3.9   MEASUREMENT OF THE MECHANICAL PROPERTIES OF COATED POWDER COMPACTS

1.   Density Measurement

The calculation of green density was determined by measuring the dimensions and weights of the compacts.   Each compact was weighed on a balance to an accuracy of 0.0001g. A micrometer was used to measure five values of each of the compact height and a diameter to an accuracy of 0.01 mm and an average was taken.   The measured weights of the pressed compact divided by their calculated volumes, produced the green density of these compacts.

For the theoretical density, the rule of mixtures for the composition was used, and through this value the percentage of theoretical density was measured. Therefore, it is assumed that the lattice structure and parameters of deposited metal in particular was similar to that for which the relevant 100% pure metal density was quoted[153].

2. Hardness Tests

This test was carried out on a Vickers Hardness Machine[109] with a pyramidal diamond indenter. The value of the Vickers Hardness Number (VHN) was measured by taking the average of at least four readings obtained for each compact.

Both green and sintered compacts of iron powder coated with copper and those coated with copper-tin were measured using 10 kg load for the particular indentation obtained.

For all compacts made from ceramic powders coated with nickel and copper, the hardness test was impossible. The failure seems to be because these compacts were very weak and could not stand the loads applied. The test proved unreliable even under very small loads. Also visibility of the hardness value of the two ocular readings obtained per indentation was almost impossible.

3. The Indirect Tensile Stress Tests of Green Compacts made from Iron Powder Coated with Copper and Copper-Tin[110]

This was done on an Instron Testing Machine, as shown in Figure 13. The speed of the Instron crosshead used was 0.01 cm/min. To operate the Instron at such speed, the maximum full scale deflection possible had to be set at 500 kg. The movement of both the load and the crosshead were recorded automatically by an X-Y recorder on a chart with speed of 2 cm/min. The compact was rested on a load cell freely while the top platen was set to move down to cause the specimen to fracture across the diameter as seen in the example shown in Figure 13. The test was stopped after the maximum load had been reached. Once the failure cracks appeared this was recorded and taken as the maximum load and the tensile stress was calculated.

4. The Indirect Tensile Stress Tests and the Modulus of Elasticity on Green Compacts made from Ceramic Coated Powders with Copper and Nickel

The tensile strain tests were carried out on an Instron machine in a similar manner to the green compacts made from iron coated powder as described above. The only difference was in using a strain gauge apparatus attached to the electronic plotter, see Figures 14 and 15, which show the layout of the apparatus. The strain gauge was used to measure the modulus of elasticity of these compacts directly from the same load applied for the indirect tensile test (see Figure 13).

The strain gauge extensometer was calibrated and adjusted by means of three known calibration measurements placed between the contact screws. This adjustment was allowed to give linear proportion with constant extension of 0.5 mm for each 50 units obtained directly from the amplifier. Any movement of the contact screws is recorded on the amplifier, this allows measurement of lateral strains within the compact under test. The tested specimen was positioned so that the contact screws touched the compact along its horizontal axis.

All values were obtained and recorded directly from the X-Y axes on the graphs of the electronic plotter. The input to the electronic plotter was in two ways:

i)   Direct load measurement from a transducer on the Instron machine.
ii) Strain as measured by the strain gauges around the specimen by movement of the two pins on either side of the specimen.

CHAPTER 4

PRESENTATION OF RESULTS

4.1 INTRODUCTION

This chapter involves the presentation of results, in tables and graphs, obtained from the experimental work reported in Chapter 3. These results were obtained from various physical and mechanical tests carried out on coated and uncoated powders, and compacts made from them. The formulae used for reducing these results are listed in Appendix III. Details of the microstructures of these powders and compacts are also listed in this chapter.

4.2 PLATING TECHNIQUES

1. Results of Displacement Plating in the Fluidised Bed System

This technique was carried out for deposition of copper coatings on iron particles. Table 2 shows the specifications of the iron powder used. Prior to the coating process taking place, a relationship between the rate and the height of particles in the fluidised bed was produced as shown in Figure 6, of which results are shown in Table 4. The full procedure of coating by this technique is shown in Figure 5.

The coating of iron particles with copper was carried out using three different copper coating solutions, to produce three different percentages of copper coating powders on iron particles, as shown in Table 8 and Figure 16.

2. Displacement Plating in a Bath

This technique was carried out for coating Cu and Cu-Sn alloys on iron particles. Table 5a and b shows the bath composition and the solution used. The coating procedure for Cu-Sn alloys by this technique is shown in Figure 7.

3. Electroless Plating Technique

This technique was carried out on four types of ceramic particles: SiC (29, 250 and 400 µm), Al_2O_3 (45 µm), WC (45-150 µm) and TaC (45 µm) coated with Cu, Ni and Co.

Table 7 shows the sensitising and activating baths used for pretreatment of powders for the electroless plating process. The electroless plating solution used for coating these ceramic particles is shown in Table 6. The procedures of the electroless plating processes for deposition of Ni, Cu and Co on ceramic particles are presented in Figures 8, 9 and 10 respectively.

Figures 17 to 20 show some of the parameters for the electroless nickel plating solution on SiC powder particles. Here Figure 17 shows a graph of the dependence of Ni yield on duration of coating with temperature.

Figure 18 shows a graph of the effect of particle size on Ni yield of the coating.

Figure 19 shows the effect of the quantity of coating solution and method of agitation on the Ni yield of the coating.

Figure 20 shows the effect of time duration on pH level of nickel solution.

4.3 EXAMINATION OF PHYSICAL PROPERTIES OF COATED POWDERS

Table 9 gives the flow rates and apparent densities of pure iron powder and coated iron powders, with different percentages of Cu and Cu-Sn alloy coatings. These were obtained by displacement coating in the fluidised bed system and the bath technique. The flow rates and apparent densities were measured using a Hall Flowmeter.

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4.4   EXAMINATION OF MECHANICAL PROPERTIES OF COATED POWDER COMPACTS

1.   Uncoated and Coated Iron Powder Compacts
a)   Compaction stress, density, VHN and tensile stress measurements
The summary of calculated averages of compaction stress, density, VHN and tensile stress for pure iron powder compacts; and Cu, Cu-Sn iron coated powder compacts, are shown in Tables 10, 11 and 12. From these tables, the following comparative graphs were obtained:

  i) Graphs of compaction stress versus density are shown in Figures 21 and 22.
 ii) Graphs of density versus tensile strength (or green strength) are shown in Figures 23 and 24.
iii) Graphs of VHN versus pressed density are shown in Figures 25 and 26.
 iv) Graphs of VHN versus tensile strength (or green strength) are shown in Figures 27 and 28.

The summary of comparative results for different percentages of Fe-Cu and Fe-Cu-Sn compacts are presented as follows:

  i) Tables 13 and 14 show the calculated pressed densities with different compaction loads.
 ii) Tables 15 and 16 show the calculated results of pressed density and the tensile (or green) strength.
iii) Tables 17 and 18 show the calculated results of pressed density and average VHN.

b)   Lubricated and unlubricated compaction of coated powders
Tables 25 to 31 show comparative data of dimensional parameters, density, compaction and ejection stresses for lubricated and unlubricated compacts made of Fe, Fe-Cu and Fe-Cu-Sn powders with different percentages of coatings.

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c) Comparisons between sintered and unsintered iron compacts

The sintering process was carried out at 800°C for 9 minutes. Comparisons between sintered and unsintered densities, volumes and VHN calculated results for different percentages of Fe-Cu and Fe-Cu-Sn powder compacts are summarised in Tables 19 to 24.

2. Ceramic Coated Powder Compacts

a) Compaction stress, theoretical density, tensile strength, tensile strain and elastic modulus

The summary of calculated compaction stress, theoretical densities, tensile strength, tensile strain and elastic modulus of ceramic compacts coated with different percentages of Ni and Cu are summarised in Tables 32, 33, 34, 35, 40 and 41. The results from these tables are shown in the following comparative graphs:

   i) Graphs of theoretical density percentage versus compaction stress, are shown in Figures 29 and 30.
  ii) Graphs of theoretical density percentage versus tensile strength, are shown in Figures 31 and 32.
 iii) Graphs of theoretical density percentage versus tensile strain, are shown in Figures 33 and 34.
  iv) Graphs of theoretical density percentage versus elastic modulus are shown in Figures 35 and 36.

Tables 36 to 39 show the calculated pressed densities for different compaction loads applied on ceramic compacts coated with different percentages of Ni and Cu.

b) Comparisons between sintered and unsintered ceramic compacts

The sintering process took place at 1000°C for 10 minutes. This was carried out on WC-Ni, SiC-Ni and Al_2O_3-Cu ceramic powder compacts.

Tables 42, 43 and 44 show a comparison between the pressed and sintered densities of these compacts for different compaction loads.

4.5 MICROSTRUCTURE EXAMINATION

1. Examination of Microstructure by Optical Microscope

Examination of the microstructures by this method was carried out on the original and coated iron and ceramic powders of Al_2O_3, WC, TaC and SiC. Polished and sectioned surfaces of particles (coated and uncoated) of compacts made from these were also examined by this method. These are classified as shown below:

a) Loose powders

Figure 38 shows the appearance of iron powders coated with Cu and Cu-Sn made by the displacement method. Figures 55 to 59 show the appearance of ceramic powders coated with different percentages of Cu, Ni and Co metal coating by the electroless technique.

b) Polished cross-sections of coated and uncoated particles

Figure 37 shows the microstructure of a cross-section of pure iron particles. Figure 39 shows the cross-section of iron particles coated with Cu and Cu-Sn particles, made by the displacement method.

Figures 60 to 63 show the microstructure of cross-sectioned ceramic particles coated with copper. Figures 64 to 66 show the cross-sectioned ceramic particles coated with nickel. All these microstructure photographs were taken at different magnifications up to x100.

c) Sintered and unsintered iron coated compacts

The sintered and unsintered iron compacts were examined under the optical microscope at different magnifications.

Figure 48 shows the microstructure of Fe-Cu coated compacts. Figure 49 shows the Fe-Cu-Sn coated compact microstructure, while the microstructure of their sintered compacts can be seen in Figure 53a and b.

2.   Examination of Particles Using the Scanning Electron Microscope

Photographs of powders examined by SEM were produced for uncoated and coated iron particles with different percentages of Cu and Cu-Sn coatings made by the displacement method. The broken green compacts of these powders with different densities were also observed under the SEM. Uncoated ceramic particles, and particles coated by the electroless method, were also examined at different magnifications.

a)   Loose powders

Figure 40 shows the characteristics of uncoated iron powder. Figures 41 to 44 show the appearance of Fe-Cu coated particles with different percentages of Cu.

Figures 45 to 47 show the appearance of Fe-Cu-Sn coatings for different magnifications and percentages of coatings.

The appearance of the four types of uncoated ceramic particles: SiC, Al_2O_3, WC and TaC are shown in Figures 67 to 72 respectively.

The microstructures of copper coated ceramic particles are shown in Figures 73 to 83.

The appearance of particles of Al_2O_3, SiC, WC and TaC with nickel coatings are shown in Figures 84 to 91.

The cobalt coated WC particles are shown in Figure 92.

b)   Broken green compacts

The fracture surface of a broken pure iron compact is shown in Figure 50.

The fracture surfaces of broken Fe-Cu compacts with different percentages of Cu coating and different densities are shown in Figure 51.

Figure 52 shows the fracture surface of broken Fe-Cu-Sn compacts with different percentages of coating.

3. Examination of Microstructure by Electron Probe Microanalysis

Analysis was carried out on the loose powders, green and sintered compacts of Fe-Cu and Fe-Cu-Sn powders with different percentages of coatings using the Cambridge Microscan 5.

Figure 54(a) shows the traces from copper coated iron particles in a sintered compact; whilst Figure 54(b) and (c) show the traces from iron particles coated with copper-tin in a sintered compact.

CHAPTER 5

DISCUSSION OF RESULTS

5.0 PLATING TECHNIQUES

There are many coating techniques that could be used for coating powders, as mentioned in Chapter 2, of which only two wet methods were chosen, i.e. the chemical displacement method and the electroless (or chemical) method. The deposits obtained on powders using these two methods are discussed under the following sections.

5.1 THE CHEMICAL DISPLACEMENT COATING TECHNIQUE

The metallic deposition obtained by this coating technique for powders was achieved by two handling methods; these were the fluidised bed and the bath techniques. These methods are discussed separately below:

a)   Fluidised Bed Plating Deposition

The procedure carried out for deposition of the copper coating on iron powder by this method is shown in the flowchart in Figure 5. Three coating solutions, each with different copper sulphate concentrations, were used for coating iron particles in the size range: -150 +125 µm at room temperature, see Table 8. Three types of coated powders were produced from these solutions containing different percentage weights of copper in each. Figure 16 shows a graph of copper-sulphate (g/l) against different percentage weights of copper deposited on iron particles; where the increase in Cu^{++} concentration caused an increase in the copper deposition.

An estimation of the movement of iron particles in the fluidised bed was observed and presented in Table 4 and plotted in Figure 6. Here a relationship between the flow rate of the fluidised bed and the height of particles, shows that when the flow rate increased up to 2 cm/s, the height of the particles expanded sharply from the bed.   If the

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flow rate was increased over 2 cm/s, then the particles would leave the bed and flow into the reservoir shown in Figure 4.

A few points were observed, while this technique was carried out, which could be considered as advantages. This technique provided a multiple coating in such a manner that the iron particles were continuously suspended over the bed. This ensured that a continuous and quicker replacement of copper ions, which were being removed from the solution, during the chemical displacement reaction which is:

$$Cu^{++} + Fe \longrightarrow Fe^{++} + Cu \text{ (deposit)}$$
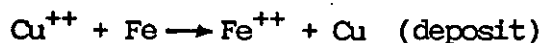
Uniformly coated powder particles were produced and free copper particles from the coating solution were also observed. No contamination however was observed during this coating process. When the latter came to an end, the blue colour of the copper solution totally disappeared, this was more clearly observed for dilute coating solutions.

The powder/solution mixtures could be agitated even further if a mechanical stirrer was introduced into the mixture. Here violent agitation of the powder was achieved using both mechanical and solution flow stirring. This did not seem to make any difference to the coating process, since the particles were continuously suspended in the flowing solution due to the action of the fluidisation.

b) Bath Plating Deposition

The procedure carried out for deposition of Cu-Sn coatings on iron powder particles by this method is shown in the flow chart given in Figure 7. These coated powders were produced with different percentage weights of Cu-Sn coatings at room temperature on iron particles in the size range -150 +125 μm. The bath composition for this type of coating is shown in Table 5(b). One sample of coated powder was produced by this technique for copper deposition on iron particles; the bath composition and the coating procedure was similar

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to that shown in the flow chart in Figure 5 for the fluidised bed technique, except one further amount of copper sulphate of 7.5g was added.

Here it was found that mechanical agitation in a small bath produced a similar agitation of the powder particles so that they were all kept in suspension in the plating solution.

No real problems were found in using this simple coating method and the particles produced clearly showed their metallic coatings, as can be seen in Figure 38.

5.2 ELECTROLESS (OR CHEMICAL) COATING

The electroless plating process is an excellent technique available for the fabrication of ceramic-metal compositions, because it gives a uniform and often adherent metal coating on ceramic surfaces (see references 111-116).

The electroless plating technique was used in this research for deposition of Ni, Cu and Co coatings on different types of ceramic particles. The ceramic materials chosen were: SiC, WC, Al_2O_3 and TaC; because oxides and carbides, such as these, have many industrial uses. The deposits obtained on powders using this technique are discussed under the following sections.

1.   Electroless Nickel Deposition

Deposition of electroless nickel plating on SiC (29, 250 μm), WC (-150 +45 μm), Al_2O_3 (45 μm) and TaC (45 μm) particles was carried out producing different percentages of Ni deposition. Since these ceramic particles are non-conductive materials, a special treatment had to be carried out in order to make their surfaces suitably sensitised and activated[117,118]. Table 7 shows the bath compositions used for sensitising and activating the above ceramic particles prior to introducing them into the electroless bath.

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Figure 8 shows a flow chart of the processes for nickel coating on ceramic powders in a bath. It was observed in this study that no limit to the weight percentage of coating was found for the powder sample; as long as the baths were freshly replaced and were not contaminated, or became unstable, during the deposition process.

Electroless nickel plating, particularly of silicon carbide particles, was investigated in order to optimise the plating parameters: temperature, time, particle size, quantity of solution, method of agitation and pH level. These parameters are discussed in detail in this order below.

a)   Temperature and Time
The quantity of metal plated (Ni) increased significantly with increasing temperature, as shown in Figure 17. At temperatures above 45°C, rapid nickel plating was observed in the initial stages. Here it can be seen that the plating rate slowed down rapidly after 5 minutes.

When the nickel solution, which had been used for 5 minutes at 80°C, was then reused to plate pre-activated SiC particles, no plating was observed.

The consumption of reactants may be responsible for the limitation of Ni plating[119]. Attempting to continue the plating beyond 30 minutes resulted in the precipitation of a greenish/white powder and a decrease in the quantity of metal plated.

Therefore it can be said that the quantity of metal plated is only increased with the increase in the plating duration for temperatures below 45°C as Figure 17 shows.

b) Particle Size

Bright metal plating was obtained for two sizes of SiC particles; these were 29 and 250 μm as shown in Figure 18. There was a slight increase in yield with decreasing particle size, from about 1.35 wt% Ni for particles of size 250 μm, to about 2.2 wt% Ni for particles of size 29 μm. This is due to the fact that the finer particles of core powder have a larger specific surface area than the coarser particles; this will tend to form composite particles with a greater percentage of nickel than the coarser fraction. Hence, a fairly close size range of core powder is advantageous, if a uniform analysis of all particles is desired.

This is in agreement with some previous studies[102] which showed the same effect of the percentage nickel deposition on the particle size.

c) Quantity of Coating and Method of Agitation

A comparison between the effect of coating solution and the method of agitation, i.e. manual stirring or whisk by motor stirrer is shown in Figure 19. When the solution was whisked by a motor stirrer, the quantity of metal plated increased with an increase in the quantity of solution. On the other hand, when manual agitation was used, the quantity of metal plating was lower and showed a maximum before reducing to a much lower level. Therefore, the manual agitation action proved to be inferior to that of motor action, which must be due to the inability of the former to give a continuous supply of fresh solution to the particle surfaces.

d) pH Level

When the pH level of the plating bath was measured using the pH meter at 45°C, over a period of 60 minutes, the resultant graph can be seen in Figure 20. The pH level was reduced slowly from 9.5 to approximately 9.0 at 30 minutes; as more time was allowed, a very small reduction in pH level occurred and then remained constant at 8.9. This constant level of pH is maintained because of the presence

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of excess ammonia in the bath[39,120,121,122], which stabilises the bath in an alkaline state.

The typical appearances of nickel coated particles are given in Figure 55(b) for Al_2O_3, Figures 56 and 58(b) for SiC, Figure 57(c) for WC and Figure 59(b) for TaC.

2. Electroless Copper Deposition

Deposition of electroless copper plating on SiC (29, 250, 400 μm), Al_2O_3 (45 μm), WC (-150 +45 μm) and TaC (45 μm) particles was carried out producing different percentages of copper coating.

Figure 9 shows the flow chart for the procedure of coating these particles by the electroless copper plating method.

Details of the sensitising and activating baths are shown in Table 7, these were used prior to introducing the above ceramic particles into the electroless copper bath.

It was noticed that any increase in the amount of reducing agent, i.e. formaldehyde, above the stated amount caused the process of copper deposition to be stopped. This was due to the contamination of the bath composition, particularly at elevated temperatures. This is a common fault, since the electroless coating process is based upon complex chemical reactions, especially in the presence of formaldehyde, which are sensitive to any change in parameters, such as temperature, additives, pH etc[123,124,125].

It should be noted that this plating process is carried out mainly in an acid solution during sensitising and activation, and in neutral conditions during reduction of the plating on the surface of the particles.

The typical appearance of copper coated particles is given in Figure 55(a) for Al$_2$O$_3$; Figure 57(b) for WC; Figure 58(a) for SiC and Figure 59(a) for TaC.

3. Electroless Cobalt Deposition

The electroless cobalt deposition was carried out on SiC (29, 250 μm) and WC (-150 +45 μm) particles only.

Figure 10 shows the steps in the electroless cobalt coating deposition for these ceramic particles.

Sensitising and activating bath solutions were also used for these particles, prior to introducing them into the electroless cobalt bath, see Table 7.

Very little difference was detected in this cobalt coating process, as opposed to the nickel coatings method in the previous section. One main difference in the cobalt bath solution was that it gave a larger yield of dull cobalt, and that the coating operation had to be restricted to around 50°C due to the instability of the solution at higher temperatures.

This is very common for cobalt deposition, since it is produced by reduction involving nascent hydrogen (rather than formaldehyde) using sodium hypophosphite solutions[126].

The typical appearance of the cobalt coating on WC particles is seen in Figure 57(a). The appearance of cobalt on SiC particles is similar.

5.3 CHARACTERISTICS AND PROPERTIES OF THE COATED POWDERS

This section covers a study of the effect of metallic coatings on some of the physical and mechanical properties of the coated powders and their compacts. The examinations were carried out on compacted coated

powders in the unsintered and sintered condition, as well as on the loose powders.

These properties are discussed in detail in the following sections.

1. Physical Properties of Coated Powders

The physical properties of iron powders plated with copper and copper-tin coatings, which were made by the chemical displacement method, was studied and compared with those properties of uncoated iron powder. These physical properties can be assessed by measuring the flow rate and the apparent densities of the coated powders. The apparent densities of the coated powders, in general, are dictated primarily by the specific gravity of the components, the particle shape and size of the core, and the thickness of the coatings. While the screen size distribution is dependent on the quantity of core plus any agglomeration of the powder, due to the method of coating.

Table 9 shows the effect of the coating techniques and the type of coatings on the apparent density and flow time of iron coated powders. It can be clearly seen that the apparent density of the coated iron particles decreases in comparison with the uncoated iron particles; whilst the flow time increases.

This behaviour is also observed as the weight percentage of coating increases, whether the coating is by displacement in the bath or in the fluidised bed system.

This could be due to the surface roughness produced by the deposited powder particles; this roughness causes the specific surface area to be increased and this leads to a reduction in the apparent density. The increase in surface area and roughness creates interparticle friction, which causes a delay in flow time. A study of the differences in the surface character of the particles in Figure 40 (Fe particles) when compared with Figures 41-44 (Fe/Cu particles) and Figures 45-47 (Fe/Cu/Sn particles) explains this flow behaviour.

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2. Mechanical Properties of Coated Iron Powders

This section covers iron particles plated with copper and copper-tin coatings with different percentages of coating. The effect of coating on the mechanical properties of these particles will be discussed below.

a) Effect of coating on compactability of composite powders

The compaction process was carried out as explained in Section 3.5. Figure 21 shows a graph of pressed density against compaction stress for uncoated iron particles compared with different percentages of copper coated iron particles. Tables 10 and 11 show the results presented on this graph.

It can be clearly seen from Figure 21 that the effect of copper coating on the iron particles during compaction increases the density as the compaction pressure increases.

It can also be seen that an increase in the percentage of copper coating increases the pressed density as would be expected. This is due to the copper being a ductile material and its presence improves the compactability of the iron powder, which is a great advantage when a higher density is required under the same pressure.

Figure 22 shows a graph of pressed density against compaction stress for uncoated iron particles compared to Cu-Sn coated iron particles. These results are also presented in Tables 10 and 12. The behaviour of this graph shows a slight effect on increasing the pressed density in comparison to the copper coated iron particles. This could be due to the fact that Cu-Sn coatings have little effect on the ease of pressing of iron powders.

Similar results have been found in earlier researches by Foba and Coleman[127], when the effect of coating on the pressed density of iron particles was studied.

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Tables 13 and 14 show this behaviour of the pressed density with coating content in a different form, when a compaction load was applied on copper coated, and copper-tin coated, iron particles produced by the displacement method using bath and fluidised bed techniques.

b)   Effect of coating on tensile (or green) strength of pressed compacts

The process of measuring the green strength was described in Section 3.8 earlier.

Figure 23 shows a graph of pressed density against green strength for the iron and copper coated iron particles, using the results from Tables 10 and 11. The curves for copper coated iron powder compacts indicate a marked increase in green strength as the green or pressed density increases, as would be expected. This substantial increase in strength could be due to the high ductility of the copper coating which allows more particle deformation before failure.

Moon and Choi[128] indicated that the green strength should be related to the contact area between the powder particles. Therefore, an increase in the green density due to greater particle movement and deformation, gives rise to more particle interlocking, which will promote the green strength. Hence the relationship between density and green strength.   James[129] claims that the fundamental bonding forces of attraction are more likely to maximise the green strength, as an increase in compaction pressure is recorded.

Figure 24 shows a graph of the pressed density against green strength for iron particles and copper-tin coated iron particles, plotted from the results in Table 12. It can be seen from this graph that a similar behaviour is obtained, as shown in Figure 23, however lower pressed densities here in Figure 24 have the same green strengths.

This may be due to the presence of Sn in the Cu-Sn plating which appears to increase the resistance against the pressure applied and therefore decreases the pressed densities. The bond between the particles, however, may be stronger for the Cu-Sn coating, hence the increase in green strength.

Tables 15 and 16 show more results of measured green strengths and pressed densities for copper and copper-tin coated iron particles, which demonstrate a similar behaviour to those results obtained in Tables 11 and 12.

c)   Effect of coating on hardness of unsintered compacts
The process of measuring the hardness of compacts using the Vickers hardness test is described in Section 3.9.2.   Since hardness is an indication of the solidity and firmness of a material, all iron particles within a compact will have properties dependent on their coating and hence different hardness values for each type of composite powder.

Figure 25 and Tables 10 and 11 show the values of Vickers Hardness Number (VHN) against the green density for iron particles with different percentages of copper coatings.   It is noticeable that an increase in the green density causes an increase in the VHN, as would be expected.

The copper coatings on the iron particles cause an increase in the density of the compacts under compaction pressure, as seen in Figure 21;   however Figure 25 shows that for compacts with the same pressed density for example 7.2 g/cc, those compacts with high Cu contents have lower values, 85 compared with 120 VHN for pure iron compacts. This would again be expected, as the copper content increases the ductility of the compacts.

Figure 26 shows a graph of the VHN against green density for iron particles and copper-tin coated iron particles and Tables 10 and 12

represent the results used in plotting these curves. The behaviour of these materials appears to be very similar, there is difficulty in separating the curves for each composite powder compact. This indicates that the change in the Sn contents is not as dramatic as for the Cu contents. Compacts at the same density here are generally harder than for the Fe/Cu compacts. Compare hardness values (between 120 and 140 VHN) at 7.2 g/cc in Figure 25 with Figure 26.

Figure 27 shows the graphs of VHN against green strength of iron particles with different percentages of copper coatings; these graphs were plotted from the results shown in Tables 10 and 11. The behaviour of the curves show that the increase in the value of the green strength causes an increase in the value of VHN. This could be justified by the great bonding between the particles of the compact, which is caused by the coating effect. Therefore, all different percentages of copper coated particles show an improvement of VHN over uncoated iron particles.

Figure 28 shows VHN against green strength for different percentages of copper-tin coated iron particles, and the results of these graphs are shown in Tables 10 and 12. The behaviour of these curves are similar to those of the copper coated iron particles with a noticeable improvement in VHN over the uncoated iron particles.

Tables 17 and 18 show the results of direct readings of pressed density and VHN for both copper and copper-tin coated iron particles, showing an increase in pressed density and a corresponding increase in the VHN.

d)   Effect of coating on the mechanical properties of sintered
      compacts
The copper and copper-tin coatings on iron particles with different percentages of coatings were sintered and some observations of their mechanical properties were studied in comparison to the unsintered coated iron compacts.

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Tables 19 and 20 show the comparison between the unsintered and sintered densities of copper coated and copper-tin coated iron powder compacts. The sintering was carried out at 800°C for 9 mins in an industrial furnace. The sintering operations involved two low melting point metals, these were tin (232°C) and the higher melting point metal copper (1083°C). In this exercise, where the sintering time is quite short, only the first stage of sintering occurs, then the presence or absence of a liquid phase is very significant in the final pore structure and pore distribution within the compact. In this study, no attempt was made to analyse the effect of sintering parameters on microstructure, instead only general trends in some properties under specified conditions will be discussed.

Referring to Tables 19 and 20, it can be seen that a change in density has occurred in the copper and copper-tin coated iron particles during sintering; especially when a greater compaction stress was applied and the percentage coating was increased. It is interesting to note that in Table 20, for a compaction pressure of 767 MN/m^2, no change in density occurs for the three copper-tin coated powders during sintering.

Tables 21 and 22 show a comparison between the sintered and unsintered volumes for copper and copper-tin coated iron powder compacts. Since the volume is a function of density, therefore a change in density causes an inverse change in volume. Depending on the composition of the coating the change in volume could be a shrinkage or an expansion[130]. This phenomenon was investigated by other researchers in a series of experiments. Pelzel[131], Gummeson and Fross[132] and Bockstiegel[133], have all investigated sintered iron-copper materials and referred to the volume increase during sintering, when copper powder is added to the iron powder. Silbereisen[134] stated that the increased volume of the iron-copper solid solution cannot alone be responsible for this effect and he claimed that it must be due to the swelling between the single crystals, since the volume increase is

quite different in different temperature ranges and is also influenced by the structure of the powders.

The change in volume of the copper-tin coated iron powder compacts was occurred as a result of sintering and caused by the tin having a low melting point (232°C) coupled with a low vapour pressure at elevated temperatures. These helped and accelerated the penetration of the Cu-Sn alloy melt between the green boundaries at early stages of the sintering process[135,136,137].

In this study, the greatest change in volume and density was achieved for those compacts having highest percentage of coating; also in the case of the copper-tin composite powders, for all three compositions given in Tables 20 and 22, a compaction pressure of 768 MN/m^2 produced a compact which had no volume change.

Tables 23 and 24 show a comparison between sintered and unsintered VHN, for copper and copper-tin coated iron powder compacts. It can be seen that the VHN values decrease during the sintering, this may be due to the formation of secondary pores which occurred at early stages of sintering[138]. This porosity seems to have a remarkable effect more significant than that produced by the composition of the compacts and the sintering. Numerous investigations have been carried out to determine the effects of porosity on the mechanical properties of sintered materials[139,140,141].

e) **Effect of lubrication on the pressing of iron and coated iron powders**

The influence of lubrication on the metal powder/die interface can assist or retard densification according to the applied pressure and the lubricant content[142]. The behaviour of iron powder/stearate systems in compaction and ejection processes has been studied by a number of researchers[143,144,145,146]. In this study, diluted zinc stearate[147] (see Table 3 for specification) in ether was used for the die wall lubrication in the pressing of iron and coated iron powder

compacts with different Cu and Cu-Sn coating percentages. Comparison between the lubricated and unlubricated compaction parameters and ejection stresses of these compacts identically compacted were investigated.

Iron Powders. Table 25 shows a comparison between lubricated and unlubricated parameters of iron compacts. It can be seen that the effect of lubrication resulted in an increase in the density of the metal powder due to a decrease in height/diameter ratio for these compacts. The presence of lubricant during compaction caused a reduction in the coefficient[148,149] of friction of powder compact sliding against the die wall and increased the interlocking and the interparticle movement during pressing.

Similarly, the effect of a die wall lubricant caused a reduction in the ejection stress values of the lubricated compacts in comparison to the unlubricated ones, Table 25 shows this change to be from 5.84 to 2.48 kg/mm^2.

Copper Coated Iron Powders. The effect of zinc stearate on copper coated iron powders with different percentages of coating made using the fluidised bed technique, is shown in Tables 27-29. In these results, the lubricated density is always greater than the unlubricated density, and the lubricated ejection stress is less than the unlubricated ejection stress following similar trends to that of the uncoated iron powder in Table 25.

It is interesting to note that for Fe-1.5 wt% Cu powder compositon shown in Table 26, made by using the displacement method in a bath (rather than the fluidised bed method) a reduction in the lubricated density is observed, while the rest of the parameters follow similar behaviour to those compositions mentioned before. This could be due to some difference in the characteristics of the coating in this material.

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Tables 30 and 31 show the effect of lubrication on the pressing of copper-tin coated iron powders with different coating percentages. Here there is a great difference in behaviour of the parameters in comparison to the copper coated iron compacts. In particular, the ejection stresses for the same compaction pressure are very much lower in the unlubricated cases. There is also little difference between the lubricated and unlubricated results. This would indicate that the tin coating is acting as a very good high pressure lubricant during the ejection, but not during the compaction stage.

Increase in the percentages of coating composition generally seems to have some effect on the compaction stage, since the pressed densities increase in the case of the copper coatings, and the ejection stresses fall with increasing copper content in the case of the copper coated iron powders.

For the copper-tin coated iron powders there is little difference due to composition in the pressed densities, but the ejection stresses are very much lower in both lubricated and unlubricated cases. The stresses appear to be dependent on composition.

3. Mechanical Properties of Coated Ceramic Powders

This section covers a study of the mechanical properties of the compacts made from metal coated ceramic powders. The composition of these powders is as follows: Al_2O_3 - 1.9 wt% Ni; Al_2O_3 - 6.34 wt% Ni; WC - 5.71 wt% Ni; SiC - 8.33 wt% Ni; TaC - 12.8 wt% Cu and Al_2O_3 - 11.7 wt% Cu.

Previous work has been carried out by many other researchers on the coating of ceramic particles (see references 111-116), particularly on the preparation and production of these coated particles. However, very little information has been reported on the pressing of metal coated ceramic particles.

In this study, an attempt was made to press the coated ceramic powder and the effect of metallic coating on their mechanical properties has been examined.

a) Effect of coating on compactability of composite powders

The details of the die used for compaction of coated ceramic powders is described in Section 3.5.2.

Nickel Coated Ceramic Powders. Figure 29 shows a graph of compaction stress against percentage theoretical density for Al_2O_3 - 6.34 wt% Ni, Al_2O_3 - 1.9 wt% Ni, WC - 5.71 wt% Ni and SiC - 8.33 wt% Ni green compacts. The resultant mechanical properties of these compacts are shown in Tables 32 to 35.

The increase of the Ni percentage coating on Al_2O_3 particles seems to cause a reduction in the compressibility of these powders and a reduction of their theoretical density. This reduction is presented in such a way that, at lower compaction pressures, greater reduction in the theoretical density occurs, while at higher compactionpressures, smaller reductions were introduced.

The Ni coated tungsten carbide and silicon carbide compacts show a greater increase in their theoretical densities over those of the alumina/nickel compacts. This could be explained by assuming that there is a greater deformation of the nickel bond between the SiC and WC particles (surrounded by nickel), than that between the alumina/nickel particles. The particle sizes of these composite powders will also affect the theoretical densities under increased pressure.

Copper Coated Ceramic Powders. Figure 30 shows a graph of compaction stress against percentage theoretical density for TaC - 12.8 wt% Cu and Al_2O_3 - 11.7 wt% Cu green compacts. It is clear that the increase in compaction stress causes an increase in theoretical density as would be expected. Also the effect of copper coating on the tantalum

carbide particles on the theoretical density, seems to be greater than the effect of copper on the alumina particles under the same pressure. This could be explained in a similar manner to those graphs in Figure 29, but note here that the particle sizes are the same, both being 45 μm.

Tables 36 to 39 show more results of the pressed densities with increasing compaction pressure for all the previously mentioned coated ceramic powders.

b) **Effect of coating on tensile (or green) strength of pressed compacts**

Figure 31 shows a graph of tensile strength against theoretical density for nickel coated ceramic powder compacts. The general trend of these curves shows an increase in the tensile strength with an increase in the theoretical density.

For the Al_2O_3 - 6.34 wt% Ni powders, the increase in theoretical density caused a corresponding increase in the tensile strength, this relationship is presented by a straight line. Whereas, the Al_2O_3 - 1.9 wt% Ni powders show a greater increase in tensile strength with little increase in the theoretical density in comparison to the Al_2O_3 - 6.34 wt% Ni powder compacts. For the WC - 5.71 wt% Ni and SiC - 8.33 wt% Ni green compacts, the high values of increased theoretical density were related to low values of increased tensile strength; leaving these compacts much weaker than those of the Al_2O_3 - Ni powders.

The behaviour of these graphs suggests that the compacts with higher theoretical density give lower tensile strengths with the increased percentage of nickel present within the compacts, leaving the compacts more weak and brittle. The results of these graphs are shown in Tables 32-35.

Some studies were carried out on the effect of nickel coating on the bonding WC compacts in comparison to the milled nickel bonded powders, and it was found that the compacts produced by this method crumbled due to a very poor compactability. Therefore, greater pressures should be applied to improve the green strength[150], however this may cause fracture of the carbide particles.

Figure 32 shows a graph of tensile strength against percentage theoretical density for green compacts made from TaC - 12.8 wt% Cu, and Al_2O_3 - 11.7 wt% Cu powders. It can be seen that an increase in the theoretical density of these compacts causes an increase in their tensile strength.

The effect of copper coating within the structure of the alumina compacts seems to have little effect on its tensile strength in comparison to the TaC-Cu compacts, where the latter shows much greater tensile strength. This could be due to the increase in the homogeneity of copper coating within the TaC particles which makes this material more ductile and able to resist more pressure. The particle sizes of both powders is the same, 45 μm, so a study of the coating character and its morphology may give a clue to this anomaly. The results of these graphs are shown in Tables 40 and 41.

c) Effect of coating on tensile strain

Figure 33 shows a graph of tensile strain against theoretical density for Al_2O_3 - 6.34 wt% Ni; Al_2O_3 - 1.9 wt% Ni; WC - 5.71 wt% Ni; and SiC - 8.33 wt% Ni green compacts. It can be seen from these graphs that generally an increase in the values of theoretical density causes a decrease in tensile strain.

The alumina compacts coated with two different percentages of nickel show that the increased percentage of nickel causes an increase in the elongation allowable within the compact, presumably by reducing the cohesive forces between the individual particles.

For WC and SiC compacts coated with nickel, similar behaviours are shown, with enough elongation allowed within the compacts to agree with their tensile strengths shown in Figure 31. The results of Figure 33 are presented in Tables 32-35.

Figure 34 shows a graph of tensile strain against theoretical density for TaC - 12.8 wt% Cu and Al_2O_3 - 11.7 wt% Cu green compacts; where the general behaviour of these curves are similar to those shown in Figure 33.

It can be clearly seen that the compacts with higher theoretical densities have little elongation allowed with the compact. The effect of the presence of copper with the TaC compacts seems to make them more ductile and hence, increases the elongation, i.e. increases the tensile strength. Whereas, the presence of copper within the alumina compacts seem to have less effect on the tensile strain. See Tables 40 and 41 for results.

The graphs in Figures 33 and 34 agree with the graphs in Figures 31 and 32 where an increase in the tensile strain of a compact at a given percentage theoretical density under the same pressure conditions, gives a corresponding decrease in the tensile strength value for the same compact.

d) Effect of coating on modulus of elasticity
Figure 35 shows a graph of modulus of elasticity (E) against theoretical density for Al_2O_3 - 6.34 wt% Ni; Al_2O_3 - 1.9 wt% Ni; WC - 5.71 wt% Ni and SiC - 8.33 wt% Ni green compacts. The general trend of these graphs shows an increase in the value of E as the theoretical density increases. This is predictable, since the elastic modulus is directly proportional to the tensile stress and inversely proportional to the tensile strain; which therefore agrees with the results previously mentioned.

The effect of the increase in percentage of nickel within the alumina compacts is reflected in the values of E, where the greater percentage of nickel gives higher E values. For the WC and SiC compacts coated with nickel, similar behaviour is detected. The results presenting the graphs are shown in Tables 32-35.

Figure 36 shows a graph of E against the theoretical density for TaC - 12.8 wt% Cu and Al_2O_3 - 11.7 wt% Cu green compacts. It can be clearly seen that the lower values of percentage theoretical density produce low values of E, whereas the higher value of theoretical density produces much greater E values.

The greater percentage copper within the TaC compacts reflects a greater value of E than that for Al_2O_3. This is due to the greater cohesion or bonding forces within the particles of the compacts caused by the presence of copper, leading to a much stiffer material. The results of these graphs are shown in Tables 40 and 41.

Note that all values of elastic modulus for the compacted coated ceramic powders are far less than those for the solids in the coating materials in terms of stiffness. For example the values of E are 210 and 120 $GN/m,^2$ respectively[151]. Porosity within the compacts has a great influence in this respect and also so has the weaker forces of cohesion between the particles. These factors allow greater freedom of movement of powder particles and hence reduce the stiffness.

e) Effect of coating on sintering of ceramic powder compacts
The effect of sintering on the density of WC - 5.71 wt% Ni compacts can be seen from Table 42. Here the pressed densities under different compaction pressures increased after sintering. This could be due to the fact that densification occurred during sintering and that some of the pores within the compacts were removed, even though no liquid phase was present. Table 43 shows the effect of sintering of SiC - 8.33 wt% Ni compacts, where a decrease in the sintered density occurred. This could be due to the presence of nickel within the

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compact, which allowed an expansion of the volume of this compact during sintering, possibly by particle rearrangement, leaving the compacts more porous and therefore producing smaller densities.

Table 44 shows the effect of sintering on the densities of Al_2O_3 - 11.7 wt Cu compacts, where the sintered densities were increased under low compaction pressure, but reduced under higher compaction pressures, in comparison to the pressed densities. This could be due to the formation of secondary pores within the structure of alumina compacts during the early stages of sintering. This leaves these compacts with a highly porous structure and an accompanying increase in volume and therefore a decrease in the sintered densities.

Note that due to the shortage of material, the behaviour of the sintered densities of TaC-Cu, Al_2O_3-Ni compacts were not studied.

5.4 EXAMINATION OF COATED POWDERS AND PRESSED COMPACTS

1.   Microstructures of Metal Plated Iron Powders
This section covers a study of the microstructures of loose, unsintered and sintered iron powder compacts plated with copper and copper-tin coatings.

a)   Loose composite powders
The iron powder was examined also by optical microscopy. Figure 37 shows a cross-section of polished iron powder particles for different magnifications, where a rough idea of the relative shape and size of these particles can be seen.

Figure 38 shows the iron powders coated by the displacement method with 7.1 wt% Cu and 4.3 wt% Cu - 2.1 wt% Sn alloy photographed under the optical microscope at the same magnification. The appearance of these coated powders can be clearly seen, the particles are smoothly coated, with an orange colour for the copper coating and a bronze colour for the Cu-Sn alloy coating.

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Polished sections of these powders are shown in Figure 39; here the photograph, Figure 39(a), shows the iron powder particles surrounded by a thin layer of copper, and Figure 39(b) shows the Cu-Sn layer surrounding the iron particles.

SEM photographs were produced for uncoated and coated iron particles with different percentages of copper and copper-tin platings. Figure 40 shows pure iron particles for different magnifications. Comparing these photographs with those in Figure 41, the Fe - 1.5 wt% Cu coating seems to have a much rougher surface texture than those shown in Figure 40. Increasing the percentage of copper coating up to 9.8 wt%, as shown in Figures 42-44, seems to increase the roughness of the surface texture of the particles, leaving them with random and uniform layers of multiple coatings. These coatings were produced by the chemical displacement method using the fluidised bed technique.

Figures 45-47 show the SEM photographs of iron particles coated with different percentages of Cu-Sn layers. The nature of these coatings seems to leave the surface texture of the particles more porous and slightly rough in comparison to those produced with copper coating only. These coatings were carried out using the chemical displacement method by the bath technique.

b) Unsintered compacts pressed from coated powders
Electromicroscopy photographs were produced for the green compacts made from iron powders coated with copper and copper-tin layers. Figure 48 illustrates a cross-section of a green compact made from Fe - 9.8 wt% Cu powders coated by using the chemical displacement method in the fluidised bed. It can be seen that the iron particles (blue in colour) are surrounded by the copper (light brown), while the dark patches are the pores formed within the compact.

A cross-section of the compacts made from copper-tin coated powders made by the chemical displacement method in a bath is shown in Figure 49, where the iron particles (in blue) are surrounded by 0.7 wt% Cu -

0.2 wt% Sn alloy (bronze in colour) and the dark patches are the pores within the structure of the compact.

Other photographs were produced showing the broken surfaces of compacts made of iron, copper and copper-tin coated powders. Figure 50 shows a broken surface of a pure iron compact, where the iron particles do not seem to be much affected by compaction, in comparison to the loose iron particles shown in Figure 40(b); except for the change in the mechanical locking of the particles due to the compaction force.

Figures 51 and 52 show photographs of the broken surfaces of green compacts made from different percentages of copper and copper-tin coated powders made by the chemical displacement method using the fluidised bed technique and by the chemical displacement method in a bath technique. The failure of these compacts is due to the application of tensile forces, these cause an increase in the forces between the particles, and therefore cause the coated layers between these particles to be pulled apart. These forces seem to be reduced with increasing percentage of coating for densities ranging between 6.98 to 7.35 g/cm^3, probably due to the increase in ductility of these coatings.

c) Sintered compacts pressed from coated iron powders
Electromicroscopy photographs presenting a cross-section of sintered compacts made from copper and copper-tin coated iron powders are shown in Figure 53.

A cross-section of the Fe - 9.8 wt% Cu sintered compact is shown in Figure 53(a), which differs from that of the unsintered compacts shown in Figure 48. This change was due to the copper not reaching its melting point of 1083°C at the first stage of sintering after 9 mins at 800°C, this is when the presence or absence of the liquid phase becomes very significant in the final pore structure and pore distribution within the compact. These pores are clearly evident in Figure 53(a).

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Figure 53(b) shows a cross-section of the sintered compact made from 0.7 wt% Cu - 0.2 wt% Sn coated iron particles, where the presence of tin within the alloy has an effect on accelerating the sintering of the compact at elevated temperature[135,136]. The effect of sintering within the matrix of the compact is shown where the presence of the melting Cu-Sn alloy is seen surrounding the iron particles. The dark patches shown are the presence of growing pores during sintering in comparison to those of unsintered compacts shown in Figure 49.

2. Electron Probe Microanalysis (EPMA) of Some Coatings

The microstructure of some samples were studied using electron probe testing equipment in order to assess the homogenisation of these samples during the sintering process. Compacts made from copper and copper-tin coated iron powders were studied.

Figure 54(a) shows a trace for a Fe-Cu particle, where very sharp peaks of the coating on the powder particle can be detected, these indicate that diffusion has occurred.

Figure 54(b) shows a trace for a Cu-Sn iron coated particle, where the absence of Sn is due to the richness of Cu within the compact. The broadening of the peaks and the overlap of the peaks for coating metals and iron substrate are evidence of little diffusion occurring.

Similarly Figure 54(c) shows the absence of Cu for another Cu-Sn alloy iron coated particle. Here the areas particularly rich in Sn, show an absence in Cu. The Sn peaks are quite sharp, this is possibly due to the low melting point of the tin (232°c) which led to the occurrence of diffusion as a result of sintering. All the compacts for iron powders coated with Cu and Cu-Sn layers were sintered at 800°C for 9 mins.

These facts indicate that at the sintering temperature of 800°C, the Cu and Sn did not react to form a "bronze" alloy as expected, but rather acted as independent materials within the iron matrix in the compacts.

3. Microstructures of Metal Coated Ceramic Powders

This section covers a study of the microstructure of loose coated and uncoated ceramic particles examined using the optical microscopy and the scanning electron microscopy methods.

a) Optical microscopy of loose composite powders

Optical microscope photographs of alumina powders with 45 μm particle size and coated with 11.7 wt% Cu and 6.34 wt% Ni made by electroless plating are shown in Figure 55.

Figure 55(a) shows the alumina particles coated with copper, where the rounded orange particles are uniformly coated; while the white particles are uncoated Al_2O_3 particles. This is because the surfaces of these particles were not properly activated during the pre-treatment by palladium chloride, before introducing them to the electroless copper plating solution.

Figure 55(b) shows the alumina particles coated with Ni. The rounded dark green particles are the uniformly coated alumina, while the white particles are the uncoated alumina for the same reasons explained above.

Figure 56 shows SiC powder with 250 μm particle size and coated with 3.5 wt% Ni by electroless deposition. The coated particles have very sharp edges, and the deposition of these particles is relatively bright in colour.

Figure 57(a) shows the WC powder with 45-150 μm particle size and coated with 12.72 wt% Co by electroless deposition. The cobalt forms

dark greenish bright depositions, and the WC particles have irregular cubic shape and sharp edges.

Figure 57(b) shows the WC coated with 8.74 wt% Cu. The copper deposition is orange in colour and is uniformly coated. The dark particles are due to the presence of oxide on the surface of the particles exposed to air.

Figure 57(c) shows the WC particles, coated with 5.71 wt% Ni. The deposition of nickel is seen as a bright green deposition on the particles.

Figure 58 shows the SiC powder with particle size of 29 μm coated with Cu and Ni. Figure 58(a) shows SiC coated with 62.4 wt% Cu, where the orange copper coating covers the SiC particles by multiple electroless coating depositions.

Figure 58(b) shows the SiC coated with 8.33 wt% Ni. The greenish coloured particles and the bright coloured particles are due to the reflection of the light on the nickel coating on particles with very sharp edges.

Figure 59 shows the TaC powder with particle size of 45 μm and coated with Cu and Ni. The TaC powder coated with 12.8 wt% Cu by the electroless method is shown in Figure 59(a), where the orange colour is the copper coating.

Figure 59(b) shows the TaC coated with 4.8 wt% Ni, and the greenish colour is due to the nickel coating. The nature of these electroless coatings is to produce rough surfaced particles.

A cross-section of the ceramic coated powders was examined by the optical microscope producing the photographs discussed below. Different percentages of copper were deposited by the electroless method.

The electroless copper coating is uniformly deposited around the particles, no matter how irregular their shapes are. This is considered to be one of the greatest applications of the electroless method. Since the ceramic particles are very hard, during the polishing process of these powders, some of the polished particles came out leaving small cavities of copper within the matrix as can be seen in Figure 60. This is why care must be taken during polishing so that excess polishing pressure is prevented.

A cross-section of Al_2O_3 (45 μm), SiC (250 μm and 400 μm), WC (45-150 μm) coated with different percentages of Ni by the electroless deposition method can be seen in Figures 64 to 66. These show the nickel coating surrounding the ceramic particles, where the Ni is uniformly coated. Since the ceramic particles are very hard materials, sectioning and polishing was very difficult. During the polishing process, only the Ni coating of the surface particles was peeled off, leaving the ceramic particles surrounded by nickel. This can be clearly seen in Figure 65.

For the same reasons, comparison between different magnifications with different particle sizes for all ceramic coated powders cannot be made here.

b)   Scanning electron microscopy of loose composite powders
The SEM photographs of uncoated ceramic particles at different magnifications of SiC (29, 250, 400 μm), Al_2O_3 (45 μm), WC (45-150 μm), TaC (45 μm) are shown in Figures 67 to 72.

All different sizes of SiC particles are irregular in shape, with very sharp edges and smooth glassy surfaces, dark grey in colour. This is expected as these are prepared by crushing of the raw material to the required sizes.

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The Al_2O_3 particles are agglomerated and rounded in shape, with rough surfaces and white in colour. This because these particles are made by sintering of fine powders.

The WC particles are irregular in shape, with triangular sharp and smooth surfaces, which meet in more than one angle and have a shining silver colour. Again this is due to the crushing of the raw material to this size range.

The TaC particles form a tree-like matrix with very rough surfaces and have an olive-greenish colour.

Note that all these ceramic powders mentioned above were examined by SEM as received from Goodfellow Metals Ltd[152] with no further modifications.

The SEM photographs for different magnifications are of electroless copper platings on ceramic particles which have been obtained by single and multiple plating to obtain different percentages of coating. This copper plating was applied to particles of: Al_2O_3 (45 μm), TaC (45 μm), WC (45-150 μm) and SiC (29, 250, 400 μm); these are shown in Figures 73 to 83 respectively.

The copper coating deposition on the surface of alumina particles was seen as islands of metal when 3.4 wt% Cu was deposited. This was achieved by a single plating process. When the percentage of copper deposition was increased to 7.1 wt% by a multiple plating process, the coating started to build up gradually, forming a thick layer of deposition. This can be seen in Figure 74(b), where a rough but uniform coating around the particles was achieved. The white lines across the matrix were probably paper fibres produced during the filtering process when paper was used. This occurred because of the roughness of the copper deposition surface at this stage of coating.

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When the percentage of copper was increased to 11.7 wt%, a thick coating was produced around the surface of the particles, leaving a relatively smoother surface.

The copper deposition on TaC produced a rough surface on the particles and a relatively uniform coating around them, as shown in Figure 76, where TaC is coated with 12.8 wt% Cu.

The copper deposition on WC particles by single and multiple plating processes was achieved with different percentages of copper ranging from 8.74 to 35 wt%. The coating was uniform and smooth even with small percentage weights of copper coating, the white spots represent the free copper on the surface of the coating.

The copper deposition on SiC particles was achieved by single and multiple plating processes. This was possible for copper contents up to 62.4 wt%. Figure 71 shows a totally uniform and smooth coating surrounding the SiC particles, in comparison to Figure 79 for the same size of particles, where very little copper deposition was observed. With the larger size of SiC particles, a uniform coating was also observed, with free copper deposition on the surface of the coating.

The SEM photographs for different magnifications of nickel plating on ceramic particles, was obtained by single and multiple plating stages to give different percentage weights of coating. This was applied on Al_2O_3 (45 μm), TaC (45 μm), WC (45-150 μm) and SiC (29, 250 μm), which are shown in Figures 84 to 91.

Different percentage weights of nickel plating were deposited on alumina particles by single and multiple coating processes. Like the copper deposition on alumina, the nickel depositions gradually built up forming uniform and relatively rough, bright grey layers of coating around the surface of the particles. With the increase of nickel percentage deposition by a multiple plating process, a thicker layer was obtained. See Figures 84 to 86.

Single and multiple processes of nickel deposition on SiC particles with different percentage weights of nickel were carried out as shown in Figures 87 to 89.

Although the 29 and 250 µm SiC particles had irregular shapes, the metal film coated the rough surface fairly well. The quantity of metal plating was achieved up to 8.33 wt%. A bright coating was obtained. However, the coating had cracks all over the surface of the particles and showed partial peeling off.

The plating of 5.77 wt% Ni on WC (45-150 µm) particles is shown in Figure 90. The colour of the coating shown is a relatively bright grey. Most of the particles had large defects in the coatings, the coatings were cracked all over the surface of the particles.

Figure 91 shows a 8.4 wt% Ni deposited on TaC particles. It can be seen that the coating on the surface of these particles is rough with free nickel deposits. The defects on these surfaces are minor in comparison to those on the WC and SiC particles, see above.

Figure 92 shows the 12.71 wt% Co layer plated on WC particles. Although the particles were irregular in shape, a totally uniform and smooth coating is observed, which had a bright brownish colour. Very few cracks were observed on the surface of these coatings.

CHAPTER 6
CONCLUSIONS AND RECOMMENDATIONS FOR
FUTURE RESEARCH

CONCLUSIONS

The coating of iron and ceramic powders by wet methods, if considered early enough in the production of composite powder materials, may lead to better and more efficient production. In some cases it may lead to products which would be unobtainable by other methods.

6.1 IRON POWDERS

a) Iron particles can be coated with metals, which include Cu and Cu-Sn layers, by a chemical displacement method; using a fluidised bed or bath techniques. Up to 9.8 wt% of Cu and 4.3 wt% Cu - 3.1 wt% Sn metal coatings have been made. It is presumed that high weights of coating are also possible, but were not attempted in this research.

b) A fluidised bed technique used for coating of iron particles is an excellent method to provide continuous and coherent coatings with even thickness around each iron particle.

c) The rate of copper deposition is enhanced by the increasing copper concentration values in the plating solution (see Figure 16).

d) In the fluidised bed technique, the height of the iron particles sharply increased in the powder bed, as the flow rate increased within the fluidised bed, to a critical flow rate, which is dependent on the apparatus design.

e) The coating of iron particles with Cu-Sn layers could be an alternative method for producing bronze powders. However, the sintering conditions must be revised in order to achieve correct distribution of the Cu/Sn material.

6.2 CERAMIC POWDERS

a) Ceramic powders of carbides and oxides can be coated with metals, which include Cu, Ni and Co, by an electroless plating method. Up to 62.4 wt% of Cu, 8.33 wt% of Ni and 12.72 wt% of Co metal coating was achieved in this research. It appears that higher levels than this are also possible.

b) The electroless plating process is found to be an excellent technique for fabrication of ceramic-metal composites, providing them with a uniform and adherent metal coating on the ceramic surfaces.

c) The method of coating applied is highly dependent on the pre-treatment of the coated particles in the sensitising and activating baths.

d) Studies of the electroless nickel plating on SiC particles leads to the following conclusions:
   1. the nickel deposition on SiC depends on the duration of plating with temperature as a parameter (see Figure 17);
   2. the decrease in particle size leads to greater nickel deposition (see Figure 18);
   3. the method of agitation affects the rate of deposition, as well as the quantity of coating solution (see Figure 19);
   4. there is a slight drop in pH level in baths with time during the electroless deposition process (see Figure 20).

6.3 PHYSICAL PROPERTIES OF COATED POWDERS

The measurement of apparent density and flow rate of Cu and Cu-Sn coated iron particles seems to be affected by every percentage of coating and appears to be dependent on the method of coating.

6.4 MECHANICAL PROPERTIES OF COATED POWDERS

The composite powders take up properties of the coating, once a critical thickness is achieved. This coating then controls the mechanical behaviour of the compacted powders.

a) The following conclusions are drawn from the mechanical properties of Cu and Cu-Sn coated iron particles:

1. The green density increases with an increase in the percentage of Cu and Cu-Sn layer deposition, as well as an increase in compaction stress (see Figures 21 and 22).

2. The tensile strength of iron coated particles, increases with an increase in their green densities (see Figures 23 and 24).

3. The hardness of the green compacts increases with an increase in their densities (see Figures 25 and 26).

4. The hardness increases with an increase in the tensile strength of green compacts (see Figures 27 and 28).

5. Generally speaking, porosity within the green compact structure seems to be the main parameter affecting the hardness rather than the percentage of coating on these compacts.

b) The following conclusions are drawn from the tested mechanical properties of coated ceramic particles of: Al_2O_3, WC, SiC and TaC; coated with Cu and Ni:

1. Since ceramic particles are known to be difficult to compact, the coating of these particles made their compaction possible.

2. An increase in compaction stress causes an increases in the theoretical density of the green compacts, as would be expected (see Figures 29 and 30).

3. The tensile strength of green compacts increases with an increase in their theoretical densities (see Figures 31 and 32).

4. The tensile strain of the green compacts decreases with an increase in their theoretical densities (see Figures 33 and 34).

5. The modulus of elasticity increases with an increase in the theoretical density (see Figure 35 and 36).

6.5 EJECTION OF COATED POWDER GREEN COMPACTS

The use of ductile and soft materials, such as Cu and Sn, to coat materials such as iron, make the ejection process of the compacts easier, without using any type of lubrication. Increasing the percentage of coating has also made the ejection process even easier.

Insufficient work was carried out to assess the ejection stress for ceramic coated compacts. Nevertheless, it was noticed that the presence of coating, particularly of Cu, made the ejection process easier.

6.6 PROPERTIES OF SINTERED COMPACTS

a)   The sintered iron compacts at $800^{\circ}C$ coated with 1.5 and 7.1 wt% of Cu, resulted in a slight increase in density, whereas for 8.6 and 9.8 wt% of Cu a reduction in density resulted, as expected from work studied in the literature.

b)   Sintering of iron compacts at 800°C coated with Cu-Sn layers
       results in a loss of the low melting metal, i.e. tin, either by
       evaporation or by sweating. This effect resulted in a non-uniform
       densification. Furthermore the Cu and Sn did not react to form a
       "bronze" alloy as expected, but rather acted as independent
       materials within the iron matrix of the sintered compacts.

c)   Sintering of compacts of SiC coated with Ni at 1000°C, resulted
       in a reduction of density due to expansion in the volume of the
       compacts, whereas, sintering of WC coated with Ni at 1000°C,
       showed an increase in the density of the composites.

d)   Sintering of compacted Al_2O_3 coated with Cu, showed little
       increase in density when compacted at 3500 kg compaction load;
       and a reduction in densities when compacted at 4000 kg.
       Consolidation of this composite occurred when sintered at 1000°C.

6.7 MICROSTRUCTURE OF COATED POWDERS

The percentage volume of the continuous coated metal matrix depends on
the quantity of coated metal. The method of coating determines the
structure of the deposit, as well as the microstructure of the green
compacts.

For the compacts of iron coated powders, failure occurred at the neck
region, or within the coated metal matrix. The pore structure and pore
distribution of Fe-Cu, and Fe-Cu-Sn compacts sintered at 800°C, is
very different to that of the unsintered green compacts.

The formation of brittle or ductile fracture for the coated ceramic
particles occurs depending on the composition of   prior particle
contact points and that of the neck regions formed during sintering.

The nature of microstructure of coated ceramic particles depends on the type of metal coating. If a multiple plating process using electroless plating techniques is used, this provides a larger quantity of metal plating with carefully engineered and controlled microstructure.

Some difficulties were found with nickel coatings, where some cracks occurred in the coatings.

6.8 RECOMMENDATIONS FOR FUTURE RESEARCH

This study covered the production of coated powders by wet methods, which occupies a special place in powder metallurgy and related industries. More work could be done on different types of core and coating materials covering different areas, such as their specifications, production and uses as raw material.

Recommendations for further work to be carried out by future researchers are listed below:

1. Development of an efficient fluidised bed system for commercial scale of displacement coatings of powders particularly for bronze (Cu-Sn) alloys.

2. Development of a new method for the measurement of tensile strain within diametrically compressed compacts.

3. Improvement of electroless bath plating technique used for coating ceramic particles, particularly at higher temperatures.

4. Improvement of electroless nickel plating bath to reduce the phosphorus content as the impurity.

5. Further studies on non-conductive particles, particularly spherical powders.

6. Wider investigation on WC particles coated with cobalt and nickel, particularly when using fine WC particles.

7. Further investigation on compaction of ceramic coated powders, with greater pressure application e.g. using cold or hot isostatic pressing equipment.

8. A study of the effect of coated ceramic powders on the density distribution in the unsintered state.

9. Further work on the ejection stress of the ceramic coated powders with or without lubrication.

10. Further work on the sintering of ceramic coated powders, particularly at higher temperatures.

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APPENDICES

APPENDIX I

Technical information on the type of
Vyne material used as a semi-permeable membrane
during plating by fluidised bed system

Vyon

The Vyon name embraces a range of semi-rigid microporous polymer materials with properties tailored to suit a variety of industrial applications.

PRODUCT INFORMATION

Materials

Vyon products include Vyon F, Vyon D, Vyon H and Porvent manufactured from High Density Polyethylene. The grade of HDPE used has an exceptionally high resistance to chemicals and is not attacked by aqueous solutions of salts, acids or alkalis or by most common solvents. It also complies with the German Federal Office of Health (BGA) recommendation III for use with food products and conforms to the food and health regulations of most other countries.

Further details of chemical resistance are available from Porvair if required.

While HDPE has been chosen for the basic product range because of its versatility, products made from other polymers including poly vinyl chloride/vinyl acetate co-polymer (Vyon C), ultra high molecular weight HDPE and polypropylene are available to cater for special needs.

Properties/characteristics

The structure of Vyon materials provides porosity by way of a series of tortuous paths, the volume porosity of standard Vyon being of the order of 35%. Because of this they are able to permit flow rates much higher, and retain particles much smaller, than their pore size would indicate.

While it is not possible to give an absolute particle retention rating the table below gives average pore sizes and typical retention figures intended as a general guide. It should be noted that these figures refer to standard products but materials with an average pore size of up to 180µ can be made to order.

Pore size and Physical properties				
Material thickness (mm)	Average pore size (microns) bubble pressure method (BS 1752:1963)	Typical retention (microns)	Minimum tensile strength (kgf/cm²)	Minimum elongation at break (%)
VYON F 0.75	90	30	10	10
1.0	80	27	15	10
1.5	70	24	25	10
2.0	65	24	30	10
2.5	60	22	35	10
3.2	60	22	40	10
4.75	55	20	40	10
VYON D 3.2	30	15	70	10
4.75	30	15	70	10
VYON C 5.0	30	15	100	3
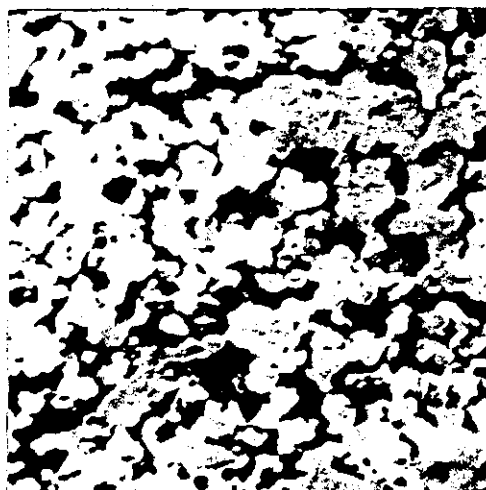
VYON CROSS SECTION

Permeability/flow rates

The graphs below indicate average water and air flow characteristics of standard products. Flexibility exists however to produce special materials with restricted or enhanced permeability if required. For example, 5 mm thick materials have been made with air permeability as high as 150 cfm per square foot at 0.2″ water gauge pressure differential.

AIR FLOW THROUGH VYON 'F' EXCEPT WHERE INDICATED
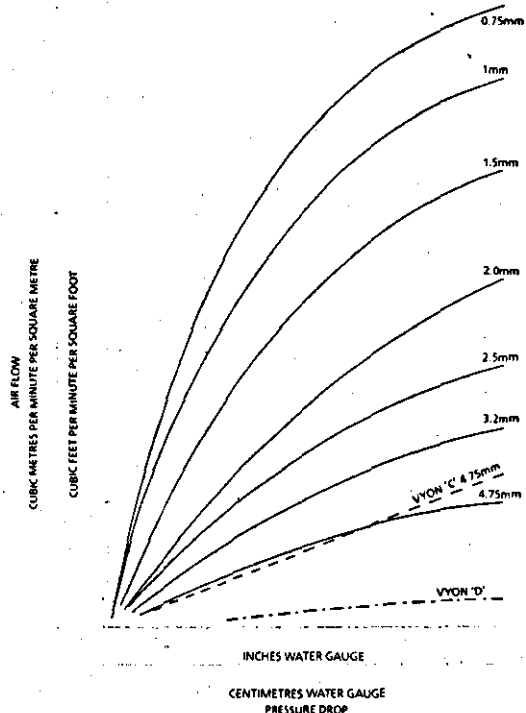
WATER FLOW THROUGH VYON F
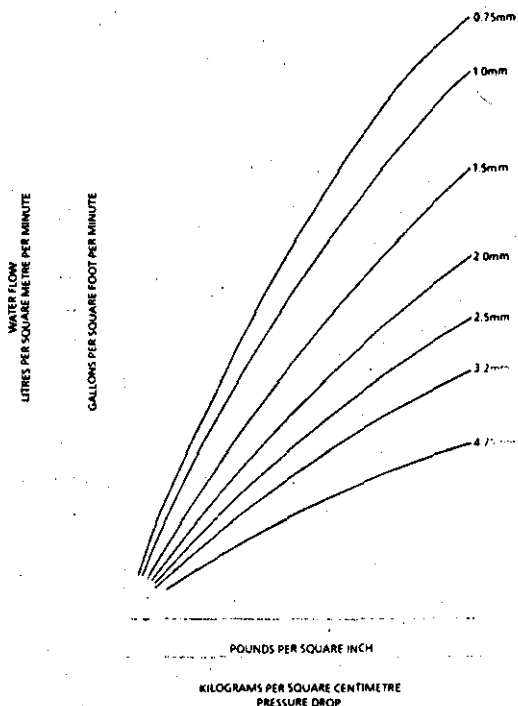
Recommendations

The preferred operating temperature range for Vyon products manufactured from HDPE is −70°C to 80°C but they may be used for short periods at temperatures up to 100°C in lightly stressed applications.

Users are advised to take advantage of the facilities and experience of Porvair's Industrial Products Division if complex fabrications are required. However, simple shapes can be readily produced by the user having conventional workshop facilities.

Vyon materials may be readily cleaned using water, most solvents or reverse flow techniques.

Availability

Vyon products are available in a multitude of configurations to meet customers' needs. These include;

▸ Standard sheets

▸ Rolls

● Mouldings

● Discs or other shapes cut from flat sheet

● Tubes (open ended or fitted with caps, flanges, threaded adaptors, etc)

● Cones.

INDUSTRIAL PRODUCTS DIVISION

APPENDIX II

Technical information on the embedding
material used for ceramic powders

SCAN-DIA EMBEDDING SYSTEM

SCANDIPLAST No. 9101

EMBEDDING COMPOUND

Normally, characteristic to this group of self-setting plastics is that, if the temperature is raised to a critical level (50°C approx.), the polymerisation will accelerate and develop like a chain reaction under a violent production of heat, by which the compound will crack and destroy itself completely. SCANDIPLAST, however, is processed in a manner so it is possible to control the reaction and achieve a fast hardening without a destruction of the material.

SCANDIPLAST 9101 is characterized by

a remarkable hardness and resistance to abrasion, better than for example most hot curing plastics and acrylic resins,

a definite ability to fill out tiny cracks and pores,

excellent transparency and complete absence of bubbles,

extremely easy and fast preparation, especially in connection without embedding moulds SCANDIFORM (DBGM-protected) and the curing oven No. 9180/83.

INSTRUCTION FOR USE.

IMPORTANT!

Decisive for a good result is perfectly clean and degreased surface of the metallic sample to be embedded.
Due to the slight contraction of the resin during the hardening especially even and glossy surfaces will not form a sufficiently good bond to the resin if not properly prepared.

impregnation is a convenient method of filling interstices (using a Witt pot).

ScandiPLAST is suitable for embedding specimens up to approximately 40 mm diameter using the above method. Larger specimens can of course be embedded and we would be pleased to provide these instructions upon receipt of your detailed requirements.

If, despite having followed the above instructions, the results are not to your complete satisfaction, please do not hesitate to contact us, as we are at your disposal for any further assistance or information you may require.

Don't forget our other products :

Scan-Dia diamond paste
Scan-Dia polishing cloths
Scan-Dia automatic polishers

D.R. BENNETT LTD.
105A, BARKBY ROAD
LEICESTER LE4 7LG
Tel: (0533) 766715

Instructions for Use

ScandiPLAST is an ideal cold-setting embedding agent
incorporating the very latest developments. It consists
of two liquid components: the embedding material itself
and the hardener. After thoroughly cleaning and degreasing
the specimens (preferably with Scandia cleaning and adhesive
solution No. 9105), one drop of hardener per cc of embedding
material is added and thoroughly incorporated by stirring;
the mixture is then poured into embedding moulds, preferably
of the ScandiFORM (silicone rubber) type. ScandiPLAST sets
within 50-40 minutes at room temperature, if heated gently to
about $40^{O}C$ it will set within 15-20 minutes, and setting can be
carried out very easily by using setting box No. 9181.

It is preferable to leave the specimens to set in the moulds
until they have cooled down to room temperature. While warm,
the embedding material has a tendency to absorb oxygen from the
atmosphere, so preventing 100% polymerisation of the surface.
The specimens then tend to become sticky on contact with
alcoholic liquids.

Where it is necessary for the hardness of the embedding material
to equal that of the specimen, Aequidur hardener adjuster may
be added to part of the stirred mix before pouring. The specimen
is embedded in a small amount of ScandiPLAST/Aequidur mix in
the mould which is then topped up with more mixture. Aequidur
hardener adjuster is available in three grades - soft, medium and
hard, enabling any degree of hardness to be obtained, from that
of the softest aluminium to hard alloys or cermets. By following
these instructions a first-class embedded specimen is attainable,
having blister-free transparent embedment with even the finest
hairline cracks completely filled with resin. A clean, non-tacky,
glass-smooth surface results which has good etch-acid/solvent
resistance, but which is soluble in chloroform or acetone.

SCANDIFORM - SCANDIPLAST

Embedding in ordinary metal-
or plastic moulds. Adhesion
between mould and resin.
Result: bad adhesion
between sample and
resin. The gap ac-
cumulates abrasives
and etching reagent.
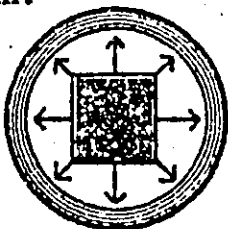
Embedding in SCANDIFORM.
No adhesion between mould
and resin. Result:
Resin contracts
around the sample.
Good adhesion be-
tween resin and
sample.
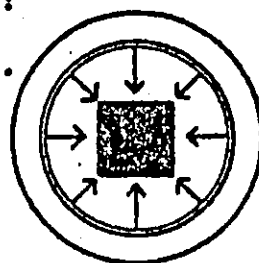
Normal procedure by embedding in SCANDIFORM:

2) Pour mixed
resin slowly
into mould.

1) Place degreased
sample into the mould.
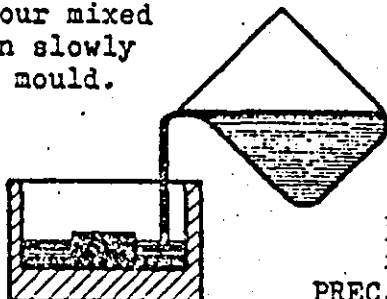
3) After harden-
ing, release specimen.
PRECAUTION! To prevent damage of
the mould, do not wrest or wring
to get the specimen out! Turn
mould upside-down and press
with thumb from bottom side.
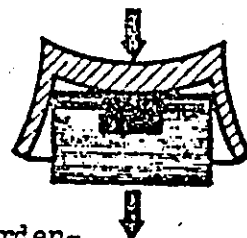
To ensure extra tight embeddings by
tricky samples (rings, nuts etc.)

1) The two-step or "layer-cake" method.
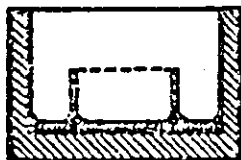
Start with very
thin layer at
the bottom and
let it harden.
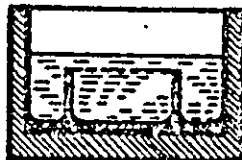
Then fill up to
ordinary measure and
harden again as usual.

2) The SEALANT method. Especially safe and perfect.

Cover sides of sample with ultra-thin layer of SCAN-DIA SEALANT 9103.
Let it harden 2-3 min. Then embed in SCANDIPLAST as usual.
(special instruction available)

3) The EQUIDUR method.
The adding of EQUIDUR, particularly the type S (9130/32), prevents con-
traction of resin and ensures tight embeddings (special instruction avail.)

To ensure extra hard surfaces of resin, see instruction for use of EQUIDUR.

Embedding of thin wires and alike.
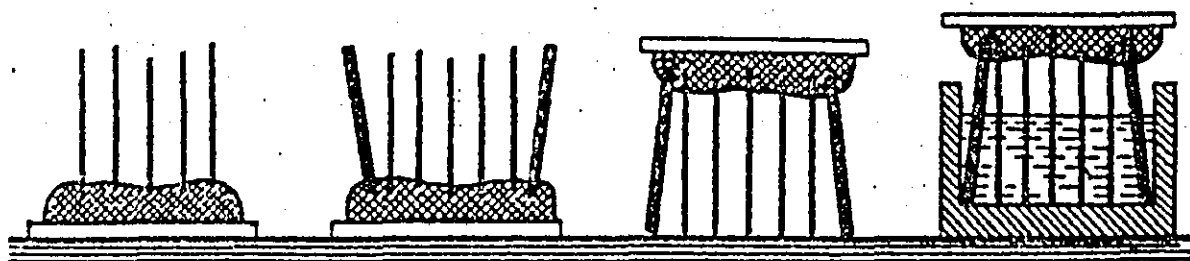
1 Place a number of
the cut wires in
plastiline, not
too deeply.

2 Finish with
a "tripod"
of nails.

3 Press the
bundle against
flat surface.

4 Place in resin-
filled mould
and harden. Wait
10 min. before
heating!

After hardening, cut surplus of wires and nails.
Grind back surface coarsely.

APPENDIX III

APPENDIX III

EQUATIONS USED IN PROPERTY CALCULATIONS

a) **Density of Compacts,** ρ

$$\rho = \frac{4m}{\pi d^2 t} \quad (g/cm^3)$$

where m = mass

d = diameter

t = thickness

b) **Compaction Stresses,** σ_C

$$\sigma_C = \frac{4F}{\pi d^2} \quad (MN/m^2)$$

where F = compaction force.

c) **Tensile Stresses,** σ_T

$$\sigma_T = \frac{2 L_M g}{\pi dt} \quad (MN/m^2)$$

where L_M = maximum applied load

g = acceleration due to gravity.

d) **Tensile Strain,** ε_T

$$\varepsilon_T = \frac{e_M}{d} \times 100 \quad (\%)$$

where e_M = extension at L_M.

e) Modulus of Elasticity, E

$$E = \frac{\sigma_T}{\varepsilon} \quad (MN/m^2)$$

f) Vickers Hardness Number, VHN

$$VHN = \frac{1.854}{L^2} \cdot P$$

where P = indenter load (kg)

L = diagonal length of indentation (ocular reading mm).

Conversion tables were supplied for translating the ocular reading (mm) into VHN, avoiding the use of the above equation.

TABLES

TABLE 2: SPECIFICATIONS OF MANNESMANN WPL 200 IRON POWDER[136]

Method of manufacture	Atomised
Apparent density (g/cm^3)	2.508
Tap density (g/cm^3)	3.40
Flow rate (sec/50g)	45.6
Surface area (cm^2/g)	207.0

TABLE 3: PROPERTIES OF ZINC STEARATE[136]

Ash (%)	15.1
Free fatty acid (%)	0.5
Water soluble salts (%)	0.2
Moisture (%)	0.5
Softening point (^{O}C)	124.0
Specific gravity	1.09
Particle size	99.9% through
(BS sieve)	325.0 mesh

TABLE 4: ESTABLISHMENT OF FLOW CHARACTERISTICS THROUGH THE SUPPORT
FILTER AND POWDER BED IN THE FLUIDISED BND

Details of system: Bed support: 4.75 mm thickness
                    Powder bed: -150 + 125 µm fraction
                    Amount: 100 gm
                    Cross-sectional: 78.5 cm
                    Pump: Centrifugal

Pump Voltage V	Flow Rate cm/s	Height of Particles Observation mm
90	0	0 No movement
100	0.53	0.3 Onset of fluidisation
110	0.92	2
120	1.24	18
130	1.75	30
140	2.0	41
150	2.3	Particles expand over 100% sharp dividing

TABLE 5:   COMPOSITIONS OF SOLUTIONS USED FOR DISPLACEMENT METHOD
COATING ON IRON POWDERS IN FLUIDISED BED SYSTEM AND BATH

A) Copper Plating Solutions

Copper sulphate heptahydrate $CuSO_4.5H_2O$	2.5 g/l
Ferrous sulphate septahydrate $FeSO_4.7H_2O$	5 g/l
Sulphuric acid H_2SO_4 (concentrated)	5 cc/l
Bath temperature (room temperature)	$\sim 20^\circ C$

B) Copper-tin Plating Solution

Stannous chloride dihydrate $SnCl_2.H_2O$ (SLR grade)	5 g/l
Copper sulphate heptahydrate $CuSO_4.5H_2O$	7.5 g/l
Sulphuric acid H_2SO_4 (concentrated) (specific gravity: 1.84)	5 cc/l
Bath temperature (room temperature)	$\sim 20^\circ C$

TABLE 6: ELECTROLESS PLATING SOLUTIONS USED FOR PLATING ALUMINA, TUNGSTEN CARBIDE, TANTALUM CARBIDE AND SILICONE CARBIDE POWDER PARTICLES

A) Copper Plating Solutions

Copper sulphate heptahydrate $CuSO_4.5H_2O$ (SLR grade)	15 g/l
Ammonium carbonate NH_2CO_3 (SLR grade)	10 g/l
Potassium sodium tartarate monohydrate $KNaC_4H_4O_6.H_2O$	30 g/L
Sodium hydroxide NaOH added to a pH of	12.5-13.0
Formaldehyde $CH_2(OH)_2$ (37-40 vol/wt%)	5 cc/l
Bath temperature	$27^{\circ}C-30^{\circ}C$

B) Nickel Plating Solution

Nickel chloride septahydrate $NiCl_2.7H_2O$	25 g/l
Sodium pyrophosphate decahydrate $NaP_2O_7.10H_2O$ (SLR grade)	50 g/l
Ammonia (aq) (20 vol%) add up to	9.5 pH
Sodium hypophosphite $NaHPO_2$ (SLR grade)	25 g/l
Bath temperature	$30^{\circ}C-40^{\circ}C$

C) Cobalt Plating Solution

Cobalt chloride $CoCl_2$	20 g/l
Sodium pyrophosphate decahydrate $NaP_2O_7.10H_2O$ (SLR grade)	72 g/l
Ammonia (aq) (25 vol%) add up to	10 pH
Sodium hypophosphite $NaHPO_2$ (SLR grade)	30 g/L
Bath temperature	$45^{\circ}C-55^{\circ}C$

TABLE 7: PRE-TREATMENT BATHS USED FOR ELECTROLESS PLATING PROCESS ON CERAMIC PARTICLES

Sensitising Solution :

- Stannous chloride: $SnCl_2.2H_2O$ 10 g/l

- HCl (aq):   40 cm^3/l

- Bath temperature: 22°C

- Treatment time   3 mins

Activating Solution:

- Palladium chloride: $PdCl_2$ 1 g/l

- HCl (aq)   5 cc/l

- pH: 1-1.5

- Bath temperature   22°C

- Treatment time: 2 min

Solution compositions used to find effect
of copper sulphate amounts on amount
of copper deposited. (See Fig. 16.)

| | Compositions | | |
	1	2	3
$CuSO_4 5H_2O$ g/l	2.5	5	10
$FeSO_4 H_2O$ g/l	5	5	5
H_2SO_4(conc) cm^3/l	5	5	5

Total volume   10 litre (distilled water)

Temperature   room temperature ∼20°C

Powder charge   100g of iron particles in −150 +125μm size range

Fig. 16

TABLE 9

APPARENT DENSITIES AND FLOW RATES FOR SINGLE
AND ALLOY COATED IRON POWDER FRACTION
(-150 + 125 μm)

Powder Type wt%	Apparent Density (g/cm^3)	Flow Time (s)
Fe	2.54	36
Fe-1.5 Cu[a]	2.41	41
Fe-7.1 Cu[b]	2.26	44
Fe-8.6 Cu[b]	2.05	47
Fe-9.8 Cu[b]	1.94	58
Fe-0.7 Cu-0.2 Sn[a]	2.50	39
Fe-3.5 Cu-3.1 Sn[a]	2.34	42
Fe-4.3 Cu-2.1 Sn[a]	2.24	43

a: Coated by displacement in bath

b: Coated by displacement in fluidized bed system (FBE)

TABLE 10: PROPERTIES OF PURE IRON POWDER UNSINTERED COMPACTS
(Average values)

Powder Type	σ_c (MN/m^2)	ρ (g/cm^3)	VHN	σ_T (MN/m^2)
IRON	480.3	6.70	65.6	24.5
	574.5	6.90	94.0	27.4
	670.1	7.09	113.5	33.3
	766.2	7.24	126.7	38.2
	865.2	7.33	148.8	40.7

Powder Type	σ_c (MN/m²)	ρ (g/cm³)	VHN	σ_T (MN/m²)
Fe-1.5Cu	480.3	6.72	74.3	25.4
	576.0	6.96	97.5	31.3
	672.5	7.13	119.9	36.2
	767.9	7.27	132.9	39.2
	864.6	7.36	151.2	42.0
Fe-7.1Cu	480.3	6.75	77.2	25.8
	577.7	7.07	105.31	32.3
	674.1	7.22	123.1	36.4
	768.0	7.38	138.1	41.2
	861.8	7.49	153.2	42.9
Fe-8.6Cu	480.2	6.80	78.4	26.0
	575.6	7.25	102.7	32.3
	671.8	7.5	119.7	37.1
	768.4	7.70	142.3	42.1
	864.9	7.9	154.2	47.0
Fe-9.8Cu	479.6	7.03	78.5	29.8
	575.6	7.43	105.87	39.2
	671.8	7.66	135.3	46.1
	769.6	7,88	163.5	50.0
	867.5	8.09	181.17	53.9

TABLE 12: PROPERTIES OF COPPER-TIN ALLOY COATED IRON POWDER
UNSINTERED COMPACTS (average values)

Powder Type	σ_C (MN/m^2)	ρ (g/cm^3)	VHN	σ_T (MN/m^2)
Fe-0.7Cu -0.2Sn	480.9	6.74	76.2	30.0
	575.6	6.96	105.1	35.3
	672.3	7.12	125.3	38.2
	768.2	7.26	144.8	43.1
	863.2	7.41	155.5	45.0
Fe-3.5Cu -1.3Sn	480.3	6.75	79.0	28.6
	577.3	7.00	107.0	33.3
	672.7	7.18	132.5	39.2
	768.1	7.33	148.8	43.7
	865.0	7.45	161.6	47.0
Fe-4.3Cu -2.1Sn	479.3	6.77	79.1	28.3
	572.4	7.00	109.7	33.6
	656.7	7.16	134.1	42.1
	756.4	7.32	152.8	44.5

TABLE 13

PRESSED DENSITY OF IRON POWDER COATED WITH COPPER BY
DISPLACEMENT PLATING METHOD USING BATH AND FLUIDISED BED
SYSTEM TECHNIQUES

Compact Composition wt%	Compaction Load kg	Pressed Density g/cc
Fe - 1.5 Cu (bath)	2510 3010 3505 4000 4520	6.71 6.97 7.13 7.28 7.38
Fe - 7.1 Cu (FBE)	2510 3025 3540 4030 4500	6.80 7.13 7.20 7.40 7.55
Fe - 8.6 Cu (FBE)	2510 3000 3495 4030 4500	6.88 7.29 7.35 7.63 7.96
Fe - 9.8 Cu (FBE)	2510 3000 3495 4030 4540	7.04 7.38 7.55 7.77 8.12

TABLE 14

PRESSED DENSITY OF IRON POWDER COATED WITH COPPER-TIN
ALLOY BY DISPLACEMENT PLATING METHOD USING BATH TECHNIQUES

Compact Composition wt%	Compaction Load kg	Pressed Density g/cc
Fe-0.7 Cu-0.2 Sn	2515	6.76
	3005	6.98
	3515	7.10
	4010	7.29
	4495	7.42
Fe-3.5 Cu-1.3 Sn	2510	6.77
	3997	7.03
	3520	7.17
	4010	7.35
	4520	7.44
Fe-4.3 Cu-2.1 Sn	2495	6.78
	2980	7.04
	3650	7.18
	3975	7.36
	4550	7.58

TABLE 15

TENSILE STRENGTH OF PRESSED DENSITIES OF IRON POWDER COATED WITH COPPER USING DIAMETRIC COMPRESSION TESTS

Compact Composition wt%	Pressed Density g/cc	Tensile Strength MN/m^2
Fe - 1.5 Cu	6.71	24.81
	6.94	31.88
	7.15	37.86
	7.29	39.63
	7.35	41.49
Fe - 7.1 Cu	6.58	22.46
	7.10	33.64
	7.25	47.57
	7.40	41.59
	7.55	44.34
Fe - 8.6 Cu	6.88	26.58
	7.21	33.25
	7.43	37.37
	7.88	48.46
Fe - 9.8 Cu	7.40	28.44
	7.42	40.90
	7.51	47.18

TABLE 16

TENSILE STRENGTH OF PRESSED DENSITIES OF IRON POWDER
COATED WITH COPPER-TIN ALLOY USING
DIAMETRIC COMPRESSION TESTS

Compact Composition wt%	Pressed Density g/cc	Tensile Strength MN/m^2
Fe-0.7 Cu-0.2 Sn	6.73 7.04 7.14 7.30	30.11 35.41 38.94 43.06
Fe-3.5 Cu-1.3 Sn	6.74 7.03 7.47	28.93 32.66 48.46
Fe-4.3 Cu-2.1 Sn	6.96 7.46 7.26 7.40	32.17 44.53 45.32 47.08

TABLE 17

VICKERS HARDNESS TESTS ON GREEN COMPACTS MADE FROM
COPPER DEPOSITION ON IRON POWDER PARTICLES
BY DISPLACEMENT METHOD

Compact Composition wt%	Pressed Density (g/cc)	VHN (Average)
Fe - 1.5 Cu	6.31 6.83 7.11 7.45	68.8 92.3 112.0 148.0
Fe - 7.1 Cu	6.8 7.05 7.31 7.59	80.1 95.3 134.0 160.0
Fe - 8.6 Cu	7.19 7.35 7.65	96.2 113.0 133.0
Fe - 9.8 Cu	7.01 7.22 7.55	75.4 97.3 123.0

TABLE 18

VICKERS HARDNESS TESTS ON GREEN COMPACTS MADE FROM COPPER-TIN ALLOY DEPOSITION ON IRON POWDER PARTICLES BY DISPLACEMENT METHOD

Compact Composition wt%	Pressed Density (g/cc)	VHN (Average)
Fe-0.7 Cu-0.2 Sn	6.8	88.3
	7.15	132.1
	7.21	138.4
	7.33	147.7
Fe-3.5 Cu-1.3 Sn	6.61	68.3
	7.08	105.1
	7.20	138.0
	7.38	152.0
Fe-4.3 Cu-2.1 Sn	6.71	77.6
	7.18	139.3
	7.36	158.5
	7.43	161.1

TABLE 19

COMPARISON BETWEEN SINTERED AND UNSINTERED DENSITY
FOR COPPER COATED IRON COMPACTS

Sintered for 9 mins at 800°C

Compact Composition wt%	Compaction Stress MN/m^2	Pressed Density g/cm^3	Sintered Density g/cm^3
Fe - Cu 1.5	575.6 673.5 769.4 863.5	6.98 7.14 7.29 7.38	7.00 7.16 7.31 7.41
Fe - Cu 7.1	576.1 671.6 767.5 861.3	7.03 7.22 7.38 7.53	7.04 7.25 7.35 7.45
Fe - Cu 8.6	575.6 667.7 767.5 863.5	7.29 7.41 7.69 7.88	7.17 7.29 7.39 7.52
Fe - Cu 9.8	575.6 673.5 767.5 871.1	7.38 7.58 7.77 8.12	7.35 7.50 7.49 7.67

TABLE 20

COMPARISON BETWEEN SINTERED AND UNSINTERED DENSITY FOR
COPPER-TIN ALLOY COATED IRON COMPACTS

Sintered for 9 mins at 800°C

Compact Composition wt%	Compaction Stress MN/m^2	Pressed Density g/cm^3	Sintered Density g/cm^3
Fe-Cu 0.7-0.2 Sn	576.6	6.98	7.01
	674.4	7.10	6.97
	768.5	7.17	7.17
	862.5	7.42	7.42
Fe-3.5 Cu-1.3 Sn	575.6	7.0	7.07
	671.6	7.20	7.21
	767.5	7.33	7.34
	863.5	7.47	7.46
Fe-4.3 Cu-2.1 Sn	580.1	7.00	7.06
	683.0	7.17	7.23
	767.2	7.35	7.36
	882.3	7.47	7.46

TABLE 21

COMPARISON BETWEEN UNSINTERED AND SINTERED VOLUME
FOR COPPER COATED IRON POWDER COMPACTS

Sintered for 9 mins at 800°C

Compact Composition wt%	Compaction Stress MN/m^2	Unsintered Compact	Sintered Compact
		Volume mm^3	Volume mm^3
Fe - 1.5 Cu	575.6	429.4	424.7
	673.5	421.2	418.6
	769.4	411.5	408.0
	863.5	406.4	404.8
Fe - 7.1 Cu	576.1	418.1	415.1
	671.6	415.1	412.0
	767.5	406.4	406.4
	861.3	402.3	403.8
Fe - 8.6 Cu	575.6	415.1	415.6
	667.7	407.4	406.9
	767.5	399.7	402.8
	863.5	369.1	392.1
Fe - 9.8 Cu	575.6	405.4	399.8
	673.5	398.2	394.2
	767.5	392.1	388.1
	871.1	381.3	380.5

TABLE 22

COMPARISON BETWEEN UNSINTERED AND SINTERED VOLUME
FOR COPPER-TIN ALLOY COATED IRON POWDER COMPACTS

Sintered for 9 mins at 800°C

Compact Composition wt%	Compaction Stress MN/m^2	Unsintered Compact Volume mm^3	Sintered Compact Volume mm^3
Fe-0.7Cu-0.2Sn	576.6	429.4	427.8
	674.4	423.8	427.3
	768.5	418.1	418.1
	862.5	403.8	403.8
Fe-3.5Cu-1.3 Sn	575.6	426.8	424.2
	671.6	419.2	417.1
	767.5	411.5	411.0
	863.5	403.8	404.4
Fe-4.3Cu-2.1 Sn	580.1	428.4	424.7
	683.0	416.6	410.5
	767.2	406.4	392.2
	882.3	401.3	397.8

TABLE 23

COMPARISON BETWEEN SINTERED AND UNSINTERED HARDNESS VALUES
FOR COPPER COATED IRON POWDER COMPACTS

Sintered for 9 mins at 800°C

Compact Composition wt%	Compaction Stress MN/m²	VHN (Unsintered Compact)	VHN (Sintered Compact)
Fe - Cu 1.5	575.6	100.9	85.3
	673.5	125.0	77.0
	769.4	136.0	71.5
	863.5	151.0	90.7
Fe - Cu 7.1	576.1	108.0	75.4
	671.6	122.0	77.0
	767.5	137.2	91.1
	861.3	151.0	85.4
Fe - Cu 8.6	575.6	98.3	63.3
	667.7	116.0	70.6
	767.5	140.7	80.2
	863.5	154.6	76.9
Fe - Cu 9.8	575.6	115.0	81.9
	673.5	129.2	67.3
	767.5	161.2	75.7
	871.1	184.3	74.0

TABLE 24

COMPARISON BETWEEN SINTERED AND UNSINTERED HARDNESS VALUES FOR COPPER-TIN ALLOY COATED IRON COMPACTS

Sintered for 9 mins at $800^{\circ}C$

Compact Composition wt%	Compaction Stress MN/m^2	VHN (Unsintered Compact)	VHN (Sintered Compact)
Fe-Cu 0.7- - 0.2 Sn	576.6 674.4 768.5 862.5	103.3 127.2 144.5 155.0	104.5 77.3 70.1 64.6
Fe-3.5 Cu- -1.3 Sn	575.6 671.6 767.5 863.5	109.7 132.0 151.7 164.0	102.8 96.6 86.5 79.9
Fe-4.3 Cu- -2.1 Sn	580.1 683.0 767.2 882.3	107.4 134.2 156.2 162.0	104.5 96.3 89.3 83.0

TABLE 25

COMPARISON BETWEEN LUBRICATED AND UNLUBRICATED
PURE IRON COMPACTS POWDER

Detail: - 150 + 125 μm Fraction
2000 kg load compaction

Compact Composition wt%	Parameter	Unlubricated	Lubricated
Fe	Mass (g)	2.99	3.00
	Height (mm)	9.76	9.15
	Diam (mm)	8.07	8.07
	Volume (mm^3)	498.96	467.77
	δ (g/cm^3)	5.99	6.41
	σ_C (kg/mm^2)	39.12	39.12
	σ_E (kg/mm^2)	5.84	2.48

TABLE 26

COMPARISON BETWEEN LUBRICATED AND UNLUBRICATED
COPPER COATED IRON COMPACTS
Fe - 1.5 Cu wt%

Detail: - 150 + 125 μm Fraction
2000 kg load compaction

Made by displacement method in bath

Compact Composition wt%	Parameter	Unlubricated	Lubricated
Fe-1.5Cu	Mass (g)	3.01	3.00
	Height (mm)	9.05	9.10
	Diam (mm)	8.07	8.07
	Volume (mm^3)	462.66	465.21
	δ (g/cm^3)	6.50	6.44
	σ_C (kg/mm^2)	39.12	39.12
	σ_E (kg/mm^2)	5.12	2.49

TABLE 27

COMPARISON BETWEEN LUBRICATED AND UNLUBRICATED
COPPER COATED IRON COMPACTS
Fe - 7.1 Cu wt%

Detail: - 150 + 125 μm Fraction
2000 kg load compaction

Made by displacement method in fluidised bed

Compact Composition wt%	Parameter	Unlubricated	Lubricated
Fe-7.1Cu	Mass (g)	3.00	3.00
	Height (mm)	9.37	9.15
	Diam (mm)	8.07	8.07
	Volume (mm^3)	479.02	467.77
	δ (g/cm^3)	6.26	6.41
	σ_C (kg/mm^2)	39.12	39.12
	σ_E (kg/mm^2)	5.68	1.94

TABLE 28

COMPARISON BETWEEN LUBRICATED AND UNLUBRICATED
COPPER COATED IRON COMPACTS
Fe - 8.6 Cu wt%

Detail: - 150 + 125 µm Fraction
2000 kg load compaction

Made by displacement method in fluidised bed

Compact Composition wt%	Parameter	Unlubricated	Lubricated
Fe-8.6Cu	Mass (g)	3.01	2.99
	Height (mm)	9.40	8.86
	Diam (mm)	8.07	8.07
	Volume (mm^3)	480.55	452.95
	δ (g/cm^3)	6.26	6.60
	σ_C (kg/mm^2)	39.12	39.12
	σ_E (kg/mm^2)	5.45	1.67

TABLE 29

COMPARISON BETWEEN LUBRICATED AND UNLUBRICATED
COPPER COATED IRON COMPACTS
Fe - 9.8 Cu wt%

Detail: - 150 + 125 μm Fraction
2000 kg load compaction

Made by displacement method in fluidised bed

Compact Composition wt%	Parameter	Unlubricated	Lubricated
Fe-9.8Cu	Mass (g)	2.99	2.98
	Height (mm)	9.00	8.67
	Diam (mm)	8.07	8.07
	Volume (mm^3)	460.10	443.23
	δ (g/cm^3)	6.49	6.72
	σ_C (kg/mm^2)	39.12	39.12
	σ_E (kg/mm^2)	5.27	1.59

TABLE 30

COMPARISON BETWEEN LUBRICATED AND UNLUBRICATED
COPPER-TIN ALLOY COATED IRON COMPACTS
Fe - 3.5 Cu wt%

Detail: - 150 + 125 μm Fraction
2000 kg load compaction

Made by displacement method in bath

Compact Composition wt%	Parameter	Unlubricated	Lubricated
Fe-3.5Cu -3.1 Sn	Mass (g)	3.01	3.01
	Height (mm)	9.23	9.10
	Diam (mm)	8.07	8.07
	Volume (mm^3)	471.86	465.21
	δ (g/cm^3)	6.38	6.47
	σ_C (kg/mm^2)	39.12	39.12
	σ_E (kg/mm^2)	2.03	1.73

TABLE 31

COMPARISON BETWEEN LUBRICATED AND UNLUBRICATED
COPPER-TIN ALLOY COATED IRON COMPACTS
Fe - 4.3 Cu - 2.1 Sn

Detail: - 150 + 125 μm Fraction
2000 kg load compaction

Made by displacement method in bath

Compact Composition wt%	Parameter	Unlubricated	Lubricated
Fe-4.3 Cu-2.1 Sn	Mass (g)	2.99	3.00
	Height (mm)	9.09	9.06
	Diam (mm)	8.07	8.07
	Volume (mm^3)	464.70	463.17
	δ (g/cm^3)	6.43	6.47
	σ_C (kg/mm^2)	39.12	39.12
	σ_E (kg/mm^2)	1.63	1.52

TABLE 32

PROPERTIES OF GREEN COMPACTS MADE FROM ALUMINA POWDER (45 μm)
COATED WITH NICKEL BY ELECTROLESS METHOD

Powder Type Composition wt %	Compaction Stress σ_c (MN/m^2)	Theoretical Density Percentage	Tensile Strength σ_T (MN/m^2)	Tensile Strain ϵ_T (x 100%) $\times 10^{-5}$	Youngs Modulus E (MN/m^2) $\times 10^{3}$
	380.0	63.68	0.890	8.63	0.103
	476.9	66.41	1.049	7.39	0.142
Al$_2$O$_3$-1.9 Ni	570.0	67.10	1.243	6.65	0.187
	665.0	68.15	1.372	5.91	0.232
	760.0	68.40	1.473	4.93	0.299
	855.0	68.90	1.787	3.69	0.484

TABLE 33

PROPERTIES OF GREEN COMPACTS MADE FROM ALUMINA POWDER (45 μm)
COATED WITH NICKEL BY ELECTROLESS METHOD

Powder Type Composition wt %	Compaction Stress σ_C (MN/m^2)	Theoretical Density Percentage	Tensile Strength σ_T (MN/m^2)	Tensile Strain ε_T (x 100%) $\times 10^{-5}$	Youngs Modulus E (MN/m^2) $\times 10^{5}$
	380.25	56.86	0.969	13.56	0.071
	474.05	58.79	1.074	7.39	0.145
Al_2O_3-6.34 Ni	569.50	60.19	1.193	4.93	0.241
	665.95	61.16	1.243	2.46	0.505
	760.95	63.61	1.382	2.21	0.625
	857.85	66.26	1.486	1.72	0.863

TABLE 34

PROPERTIES OF GREEN COMPACTS MADE FROM SILICON CARBIDE POWDER
(29 μm, PARTICLE SIZE) COATED WITH NICKEL BY ELECTROLESS METHOD

Powder Type Composition wt %	Compaction Stress σ_C (MN/m^2)	Theoretical Density Percentage	Tensile Strength σ_T (MN/m^2)	Tensile Strain ε_T (x 100%) $\times 10^{-5}$	Youngs Modulus E (MN/m^2) $\times 10^5$
	380.0	66.76	0.390	10.85	0.036
	475.0	68.51	0.440	8.13	0.054
SiC-8.33 Ni	570.0	69.97	0.517	6.41	0.080
	665.0	71.42	0.652	4.93	0.132
	760.0	72.59	0.821	2.71	0.303
	855.0	73.17	0.909	1.72	0.528

TABLE 35

PROPERTIES OF GREEN COMPACTS MADE FROM TUNGSTEN CARBIDE
(-150 + 45 μm, PARTICLE SIZE) COATED WITH NICKEL BY ELECTROLESS METHOD

Powder Type Composition wt %	Compaction Stress σ_c (MN/m^2)	Theoretical Density Percentage	Tensile Strength σ_T (MN/m^2)	Tensile Strain ε_T (x 100%) $\times 10^{-5}$	Youngs Modulus E (MN/m^2) $\times 10^5$
	380.94	66.55	0.215	11.11	0.019
	476.17	67.94	0.657	8.64	0.076
WC-5.71 Ni	571.41	69.47	0.807	7.40	0.109
	667.60	72.39	0.873	6.17	0.142
	761.80	72.46	0.940	4.93	0.191
	859.10	74.25	1.187	3.70	0.321

TABLE 36

PRESSED DENSITY OF ALUMINA POWDER COATED WITH NICKEL
BY ELECTROLESS PLATING METHOD

Detail: Powder charge: 1.5g		
Compact Composition wt%	Compaction Load kg	Pressed Density g/cc
Al_2O_3 - 1.9 Ni	1010	2.539
	1515	2.552
	2000	2.568
	2500	2.564
	3000	2.577
	3495	2.591
	4010	2.617
	4500	2.633
	5025	2.662
	5490	2.653
	6000	2.665
Al_2O_3 - 6.34 Ni	1005	2.267
	1525	2.271
	2015	2.278
	2500	2.289
	3010	2.285
	3490	2.303
	4000	2.341
	5000	2.339
	5515	2.348
	6025	2.363

TABLE 37

PRESSED DENSITY OF TUNGSTEN CARBIDE POWDER
COATED WITH NICKEL BY ELECTROLESS METHOD

Detail: Powder charge: 4g		
Compact Composition wt%	Compaction Load kg	Pressed Density g/cc
	1025	10.150
	1512	10.200
	2000	10.280
	2515	10.320
	3010	10.380
WC - 5.71 Ni	3520	10.481
	4000	10.424
	4495	10.526
	5000	10.650
	5525	10.610
	6005	10.770

TABLE 38

PRESSED DENSITY OF SILICON CARBIDE POWDER COATED WITH NICKEL, BY ELECTROLESS PLATING TECHNIQUES

Detail: Powder charge: 1.5g		
Compact Composition wt%	Compaction Load kg	Pressed Density g/cc
	1000	2.171
	1510	2.250
	2005	2.417
	2525	2.451
	3000	2.462
SiC - 8.33 Ni	3515	2.610
	4000	2.660
	4500	2.653
	5000	2.670
	5495	2.697
	6020	2.790

TABLE 39

PRESSED DENSITY OF ALUMINA POWDER COATED WITH COPPER
BY ELECTROLESS PLATING METHOD

Detail: Powder charge: 1.5g		
Compact Composition wt%	Compaction Load kg	Pressed Density g/cc
	1000	2.358
	1515	2.356
	2005	2.371
	2500	2.399
	3010	2.410
Al_2O_3 - 11.7 Cu	3495	2.439
	4000	2.447
	4500	2.453
	5010	2.451
	5500	2.468
	6000	2.50

TABLE 40

PROPERTIES OF GREEN COMPACTS MADE FROM TANTALUM CARBIDE POWDER
(45 μm PARTICLE SIZE) COATED WITH COPPER BY ELECTROLESS METHOD

Powder Type Composition wt %	Compaction Stress σ_C (MN/m^2)	Theoretical Density Percentage	Tensile Strength σ_T (MN/m^2)	Tensile Strain ε_T (x 100%)	Youngs Modulus E (MN/m^2)
	380.94	57.33	6.724	9.25	0.727
	476.17	64.44	9.487	4.93	1.924
TaC-12.8Cu	571.41	65.97	11.548	4.31	2.679
	666.64	67.88	13.095	2.46	5.323
	761.80	69.18	13.757	1.97	6.983
	857.10	70.25	15.576	0.74	21.049

TABLE 41

PROPERTIES OF GREEN COMPACTS MADE FROM ALUMINA POWDER
(45 μm, PARTICLE SIZE) COATED WITH COPPER BY ELECTROLESS METHOD

Powder Type Composition wt %	Compaction Stress σ_C (MN/m^2)	Theoretical Density Percentage	Tensile Strength T (MN/m^2)	Tensile Strain T (x 100%)	Youngs Modulus E (MN/m^2)
	379.06	51.99	0.609	3.69	0.165
	473.83	55.26	1.188	2.46	0.483
Al$_2$O$_3$-11.7 Cu	570.00	55.50	1.583	1.97	0.804
	665.00	56.90	1.873	1.23	1.523
	758.10	58.54	2.052	0.49	4.188
	855.00	59.71	2.568	0.24	10.700

TABLE 42

DENSITY OF PRESSED AND SINTERED COMPACTS MADE FROM
TUNGSTEN CARBIDE COATED WITH NICKEL, 5.71 wt%
BY ELECTROLESS PLATING METHOD

Sintered at 1000°C for 10 mins

Compaction Load (kg)	Pressed Density g/cc	Sintered Density g/cc
2000	10.32	10.35
2500	10.81	10.89
3000	11.03	11.05
3500	11.24	11.29
4000	11.43	11.55
4500	11.68	11.81

TABLE 43

DENSITY OF PRESSED AND SINTERED COMPACTS MADE FROM
SILICON CARBIDE POWDER COATED WITH NICKEL, 8.33 wt%
BY ELECTROLESS METHOD

Sintered at 1000°C for 10 mins

Compaction Load (kg)	Pressed Density g/cc	Sintered Density g/cc
2000	2.010	1.790
2500	2.290	1.960
3000	2.420	2.260
3500	2.480	2.020
4000	2.460	2.120
4500	2.440	2.240

TABLE 44

DENSITY OF PRESSED AND SINTERED COMPACTS MADE FROM ALUMINA POWDER COATED WITH COPPER, 11.7 wt% BY ELECTROLESS METHOD

Sintering was carried out at 1000°C for 10 mins

Compaction Load (kg)	Pressed Density g/cc	Sintered Density g/cc
2000	2.229	2.242
2500	2.367	2.368
3000	2.378	2.401
3500	2.430	2.443
4000	2.503	2.481
4500	2.55	2.544

FIGURE 1

A Range of Automotive Parts Made by
Powder Metallurgy

Gear selector arms
Gear plates
Synchroniser hubs
Flywheel/first motion shaft support bush
Sliding blocks for synchro hubs
Synchroniser cones
Speedo drive bearing
Gearbox mainshaft splined washer
Torque convertor stator plate and thrust bearing
Operating toggle lever
Cam spacer
Valve sleeve
Third and fourth speed gear bushing
Planet carrier cover
Front clutch hub
Synchroniser strut
Down shift cams
Transmission hub
Servo strut
Shifter lever
Reverse detent lever
Transmission synchroniser insert

Air conditioner pulley hub and rim

Selector rod bearing
Speedo bushes
Speedo pole clamp
Speedo pole piece

Window winder pinion

Windscreen wiper driver
Windscreen wiper bearings

Gear shift pivot ball

Seat-belt spacers
Seat-belt ratchet and pawl
Seat-belt lock

Boot lock claw
Lift gate latch retainer bracket

Oil relief valve plunger
Timing adjuster splined plate
Timing chain tensioner key
Valve seat inserts
Valve guide

Differential pinion gear
Differential companion flange

Door lock striker racks and wedges

Windscreen wiper pole shoe
Windscreen wiper pinion

Valve wedge
Manifold inlet pipe spacer
Carburettor fast/idle cam

Oil pump gears
Oil pump rotors
Oil pump drive flange

Shock absorber piston
Shock absorber piston rod guide
Shock absorber foot valve base

Distributor bearing
Contact points
Distributor cam
Distributor shaft bushing
Distributor bob weight

Rocker pivot ball
Water pump impeller insert
Camshaft thrust plate
Crankshaft sprocket
Oil pump drive gear
Connection rods
Crankshaft gear

Hubs for dynamo,
alternator and water pump pulleys
Camshaft sprocket
Camshaft power grip belt pulley
Rocker shaft support bracket
Rocker arm

Parking brake actuator and housing
Brake adjuster nuts and quadrants
Parking pawl toggle lever

Door stop roller
Door stop

Reclining seat pivots, quadrants and bushes

Steering shaft coupling
Steering lock insert
Steering column collar

Dynamo pole shoes and bearings
Alternator spacer bush
Alternator pole piece and end-frame

Brake valve spacer

Brake pedal and cross shaft bushes
Clutch pedal and cross shaft bushes

Rack and pinion steering gear yokes and bearings
Power steering hubs

Engine and gearbox
mounting insert

P.I. motor bearing
Starter motor pole shoes
and bearings

Clutch bearing support hub
Clutch spring seat

Steering link seats

Brake disc pad support
Suspension ball seat
Disc brake valve plate
Brake piston

Crank to clutch shaft coupling spacer plate
Clutch plate spring seats
Converter turbine hub
Clutch pilot bearing

CURRENT & POTENTIAL
APPLICATIONS

FIGURE. 1.

FIGURE 2

A Range of Domestic Applications
by P.M. Techniques

POWDER MET LTD.

Pawls
Gearbox Insert

Filing Cabinets
Rollers

Electric Shavers
Bearings

Typewriters
Cams
Bearings

Garden Swings
Bearings

Model Electric Trains
Pole Piece
Wheels
Bearings

Swivel Chair
Taper Block

Hair Driers
Bearings

Electric Clocks
Bearings

Film Projectors
Bearings — Shutter Cam

Turntables & Tape Recorders
Bearings
Tape Head Guide

Loudspeakers
Magnetic Core & pot

Lawn Mower
Clutch Shoes
Bushes

Power Drills
Chuck Jaws
Fixed & Rotating Ratchets
(Hammer)
Blade Clamps
(Circular Saw attachment)
Spur Gear
Bearings

Sporting Guns
& Rifles
Triggers
Front & Rear Sights
Magazine Catches
Safety Catches

Double Spur Gear

Hand Braces
Chuck Jaw
Reversing Ring

Orbital Sander
Inner Flange

Jig Saws
Blade Carrier
Slide

Hand Drills
Chuck Jaws
Hand Wheel Gear
Bevel Gears
Bearings

Floor Polishers
Motor Bearings

Refrigerators
Compressor Pump Rotors
Conrod
Piston
Bushes

Vacuum Cleaners
Motor Bearings
Fan Hub
Agitator End Hub

Washing Machines
Quadrant
Pinion
Bushes
Brake Lever

Spin Driers &
Tumble Driers
Motor Bearings
Tub Hub
Collars

Up and Over Doors
(Garage)
Guide Wheels

Central Heating
Fan Motor
Bearings
Armatures

Dish Washers
Motor Bearings

Electric Clocks
Bearings

Rotisseries
Gears

Extraction Fans
Bearings

Wall Tin Opener
Gear

Bottle Openers
Claw

Sewing Machines
Bearings
Arms
Gears
Levers

Door Locks & Closers
Bolt
Follower
Cylinder Cam
Multislot Cam
Hinge Bearings
Cylinder Body

Food Mixers & Mincers
Meat Cutter
Outlet Bush
Bearings
Mixer Spindle Bushes
Mincer Plates
Gears in Gearbox

CURRENT & POTENTIAL APPLICATIONS

FIGURE. 2.

FIGURE 3: POWDER METALLURGY PROCESSES

Raw Materials
- Elemental or Alloy Metal Powders
- Additives (Die lubricants, graphite)

Blending
- Blending

Forming
- Hot compaction
  - Isostatic
  - Extrusion
  - Die compaction
  - Spraying
  - Pressureless
  - Sintering
- Cold compaction
  - Die compaction
  - Isostatic
  - Rolling
  - Injection moulding
  - Slip casting

Sintering
- Sintering
  - Vacuum or Atmosphere

Optional Operations
- Optional Manufacturing Steps
  - Repressing
  - Resintering
  - Forging
  - Sizing
  - Coining
  - Metal infiltration
  - Oil impregnation
- Optional Finishing Steps
  - Heat Treating
  - Tumbling
  - Machining
  - Plating
  - Steam treating

Finished Product
- Finished Product

FIGURE 4

a) Fluidised bed system showing fluidised bed cell,
   plating bath reservoir, non-self priming
   centrifugal pump and transformer of pump control

b) Fluidised bed vessel filled with copper-sulphate
   coating solution, and the flange joint
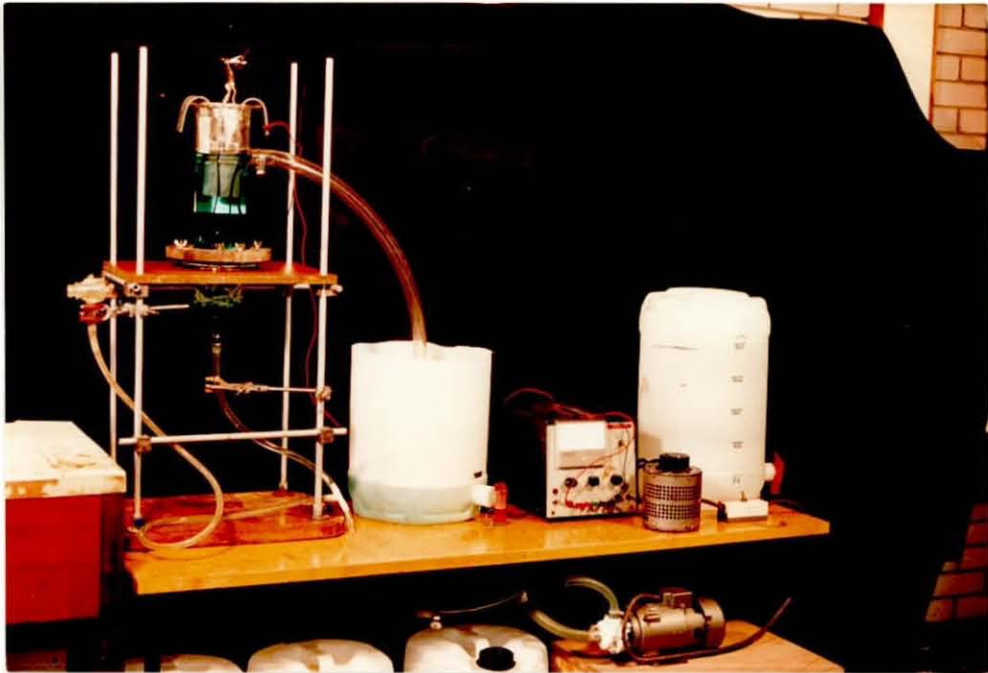
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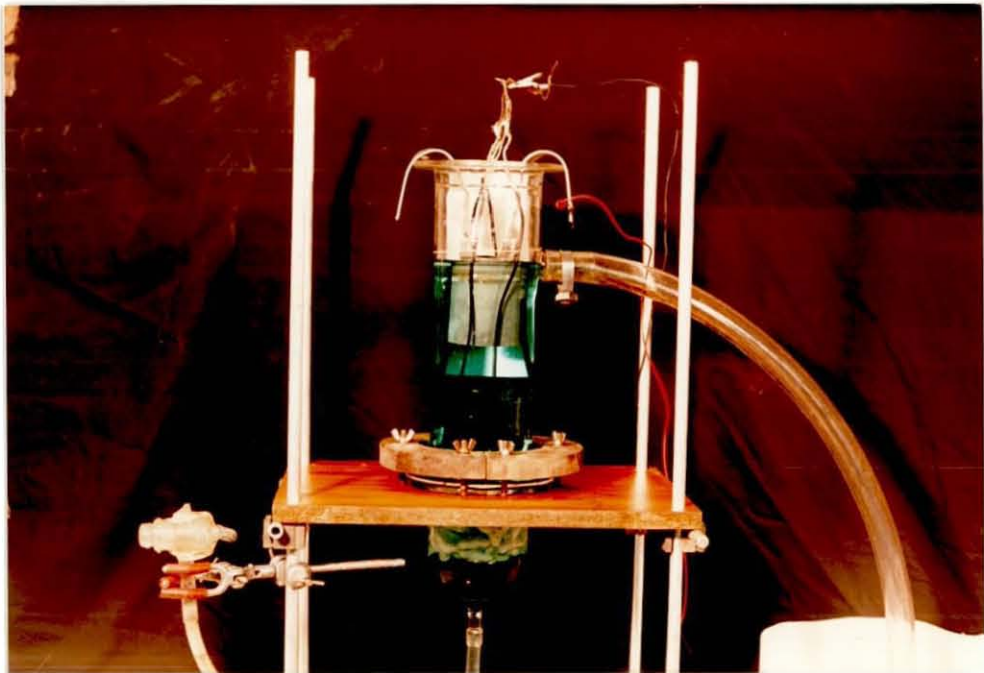
(b)

FIGURE 5

PROCEDURE FOR THE DEPOSITION OF THE Cu COATING ON IRON POWDER
BY THE DISPLACEMENT METHOD IN THE FLUIDISED BED SYSTEM

1000 ml H_2O

either 2.5, 5 or 10g
$CuSO_4.5H_2O$

$CuSO_4.5H_2O$ + 1000 ml H_2O

5g $FeSO_4.7H_2O$

$CuSO_4.5H_2O$ + $FeSO_4.7H_2O$ + 1000 ml H_2O

5g H_2SO_4

Stirring throughout fluidised bed flow

100g of Iron Powder
washed by alcohol
and then by distilled
water

Red powder + Solution

Settling and decant

Distilled water wash

Alcohol wash

Drying $50^{\circ}C$, 20 min

Red powder
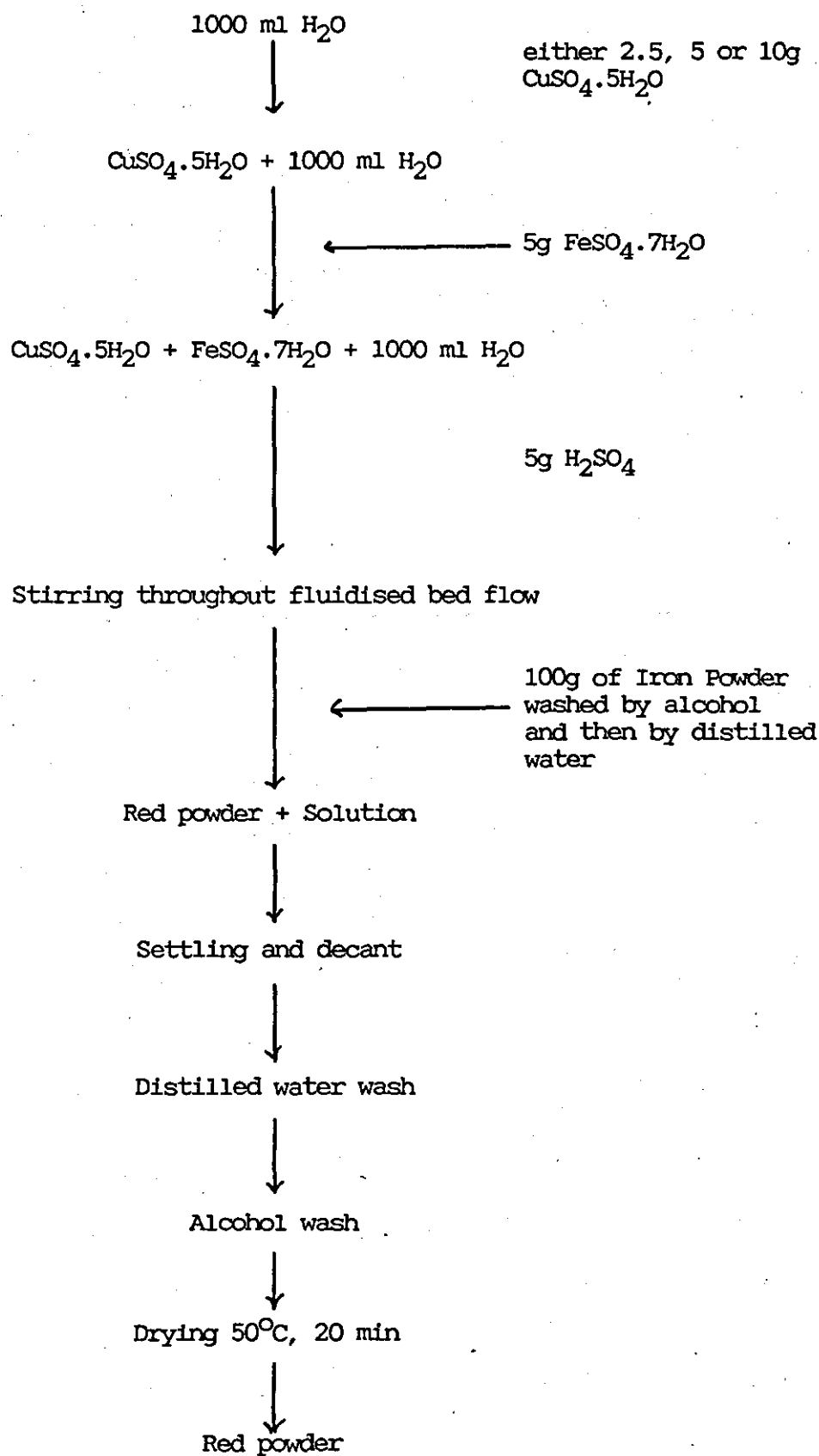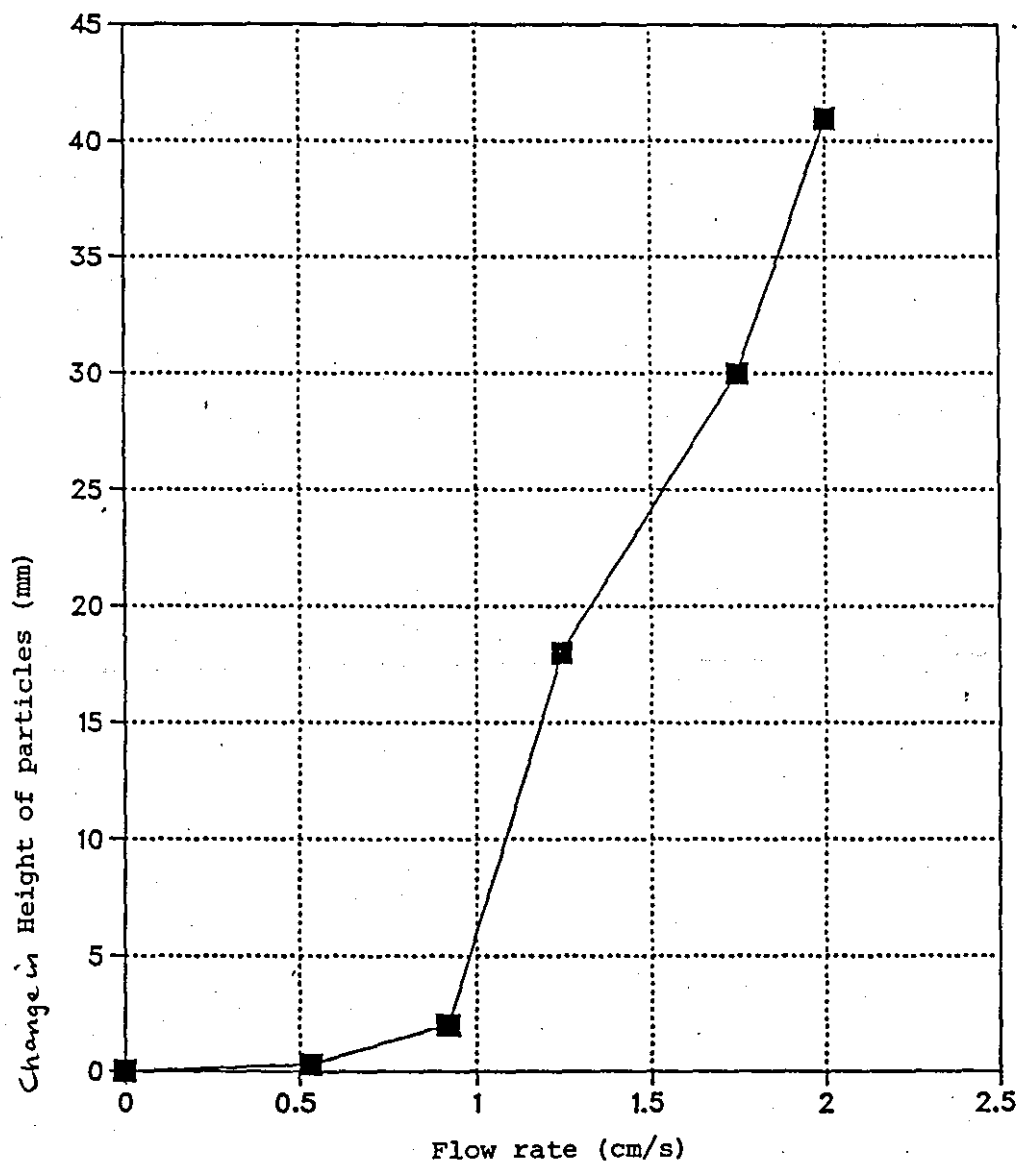
FIGURE 6: RELATIONSHIP BETWEEN FLOW RATE AND HEIGHT OF PARTICLES IN THE FLUIDISED BED SYSTEM

FIGURE 7

PROCEDURE FOR THE DEPOSITION OF THE Cu-Sn ALLOY COATING
IRON POWDER BY THE DISPLACEMENT METHOD IN BATH

1000 ml H_2O

$\longleftarrow$ 5g $SnCl_2.2H_2O$

$SnCl_2.2H_2O$ + 1000 ml H_2O

$\longleftarrow$ 7.5g $CuSO_4.5H_2O$

$SnCl_2.H_2O$ + $CuSO_4.5H_2O$ + 1000 ml H_2O

$\longleftarrow$ 5 ml H_2SO_4

Stirring mechanically
or manually

100g Iron Powder
(washed by alcohol
and then by deionised
water)

Brownish grey powder + Solution

Settling and decant

Deionised water wash

Alcohol wash

Drying 50°C, 20 min

Brownish grey powder
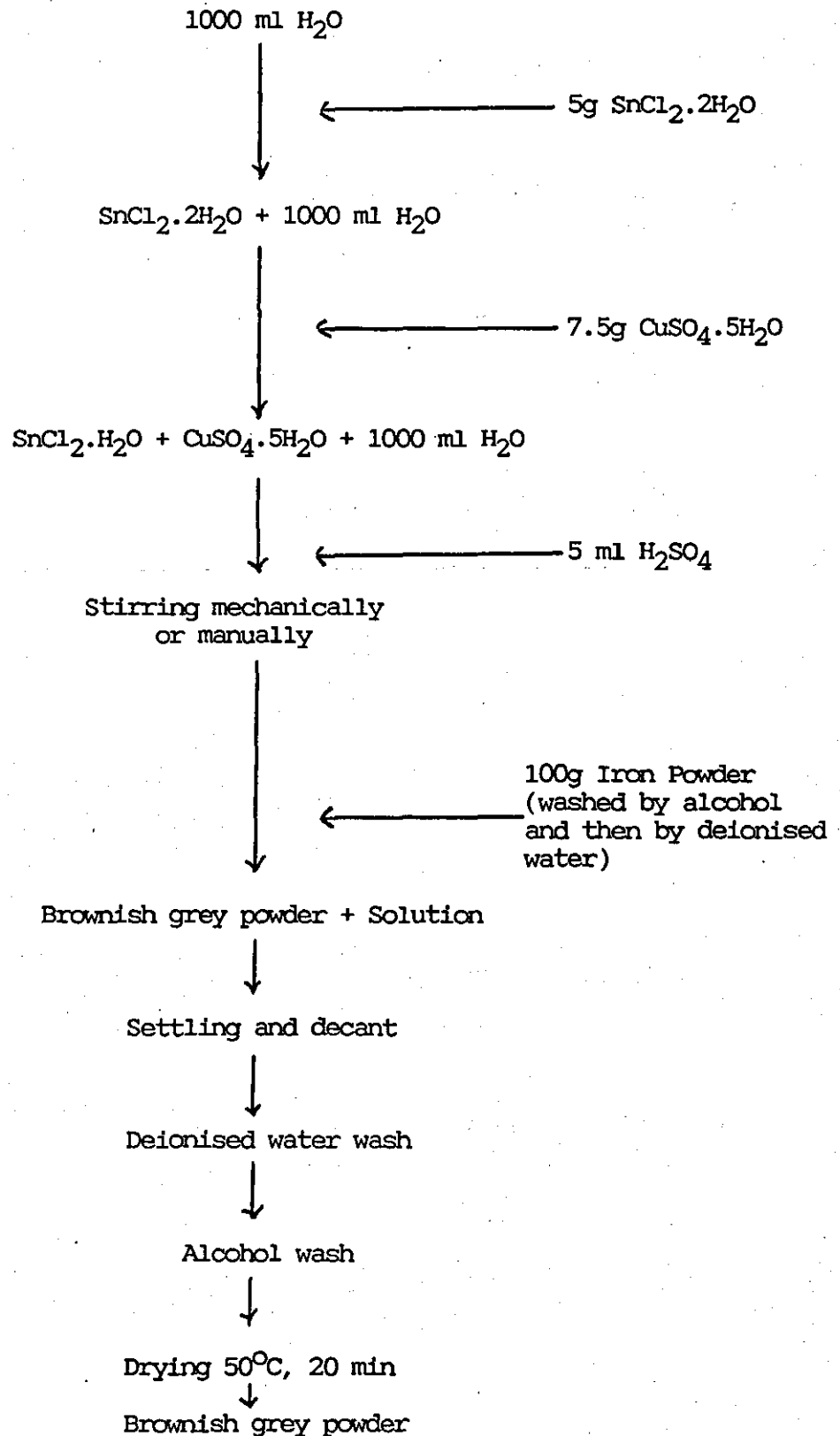
FIGURE 8

THE PROCEDURE OF ELECTROLESS NICKEL COATING PROCESS ON CERAMIC
POWDERS IN BATH

X ← grams of   - SiC
    either   - Al$_2$O$_3$
                 - WC
                 - TaC

↓

Deionized water wash

↓

Sensitising solution
wash for 3 mins

↓

Deionized water wash

↓

Activating solution wash for 2 mins

↓

Deionized water wash

↓ ← Y ml of Ni coating
     solution

Heated up to 30°C-40°C

↓ ← Na HPO$_2$ up to 25 g/l

Stirring mechanically
or manually

↓

Grey dark powder + solution

↓

Settling and decant

↓

Deionized water wash

↓

Alcohol wash

↓

Drying 50°C, 30 min

↓

Grey dark powder
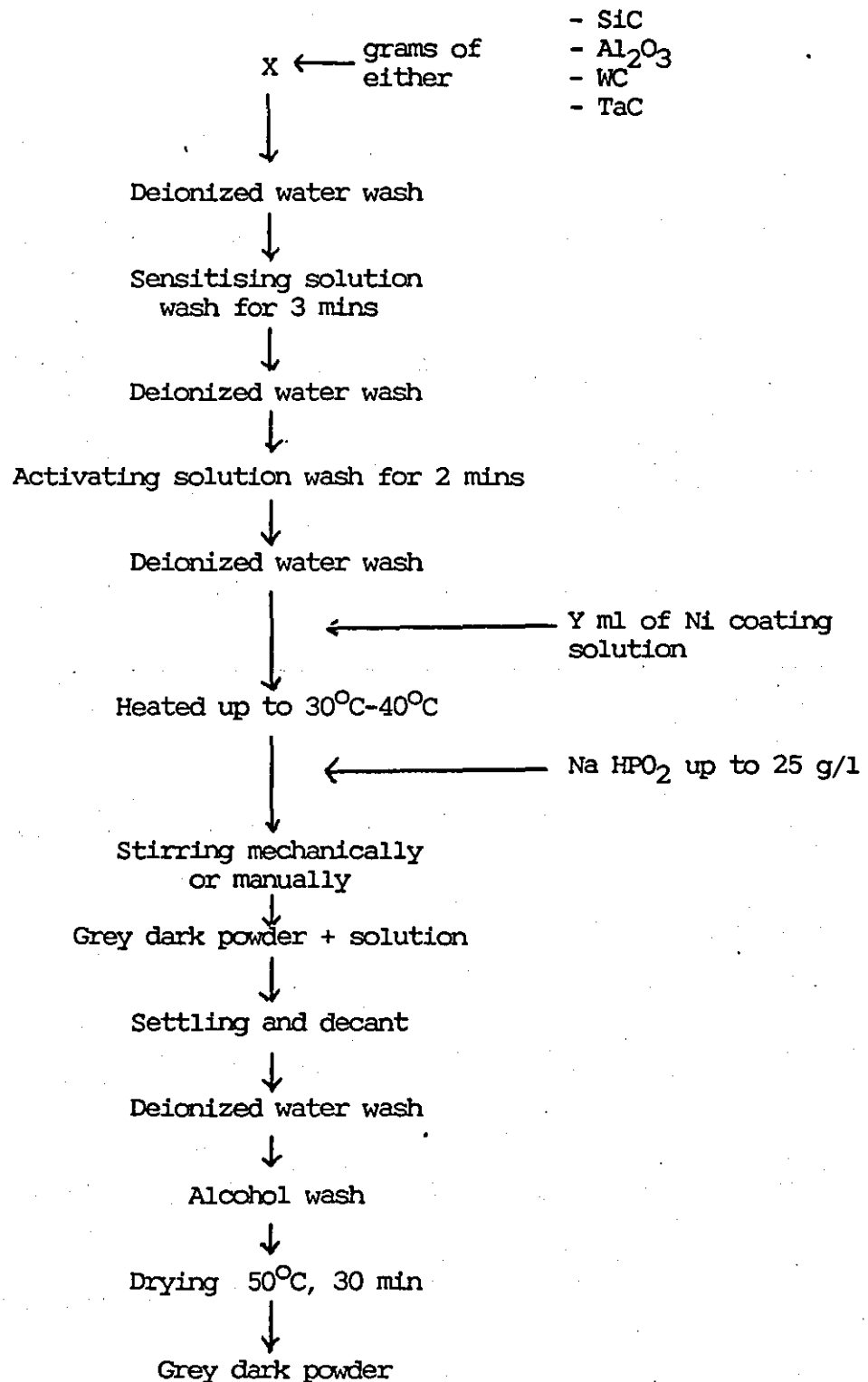
FIGURE 9

THE PROCEDURE OF ELECTROLESS COPPER COATING PROCESS ON CERAMIC POWDERS IN BATH

X ← grams of either
- SiC
- Al_2O_3
- WC
- TaC

↓

Deionized water wash

↓

Sensitising solution wash for 3 mins

↓

Deionized water wash

↓

Activating solution wash for 2 mins

↓

Deionized water wash

← Y ml of Cu solution

↓

Heated up to 27°C-30°C

← Formaldehyde (37-40 vol/wt %) added up to 50 cc/l

↓

Stirring mechanically or manually

↓

Red-orange powder + solution

↓

Settling and decant

↓

Deionized water wash

↓

Alcohol wash

↓

Drying 50°C, 30 min

↓

Red-orange powder

FIGURE 10

THE PROCEDURE OF ELECTROLESS COBALT COATING PROCESS ON CERAMIC POWDERS IN BATH

X ← grams of — SiC
        either   — WC
↓

Deionized water wash
↓

Sensitising solution
wash for 3 mins
↓

Deionized water wash
↓

Activating solution wash for 2 mins
↓

Deionized water wash
↓   ← ──────── Y ml of CO solution

Heated up to 45°C-55°C
↓   ← ──────── Na HPO$_2$ up to 30 g/l

Stirring mechanically
or manually
↓
Reflecting grey-brown powder +
brown solution
↓

Settling and decant
↓

Deionized water wash
↓

Alcohol wash
↓

Drying 50°C, 30 min
↓

Reflecting grey dark powder

FIGURE 11

a) Instron Universal Testing Machine showing:
    plotter, control panel, load cell, and pressing rig
    on the load cell

b) Pressing rig for 8 mm diameter die showing: top
    punch, die and wooden support
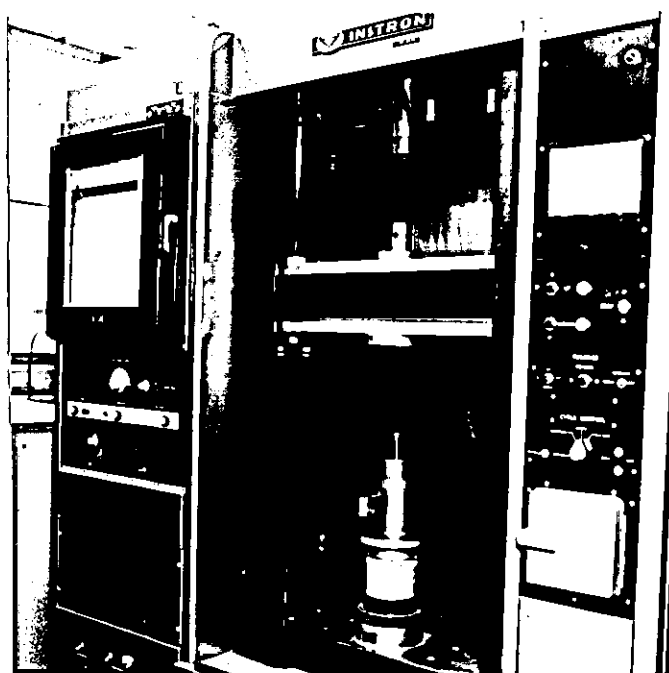
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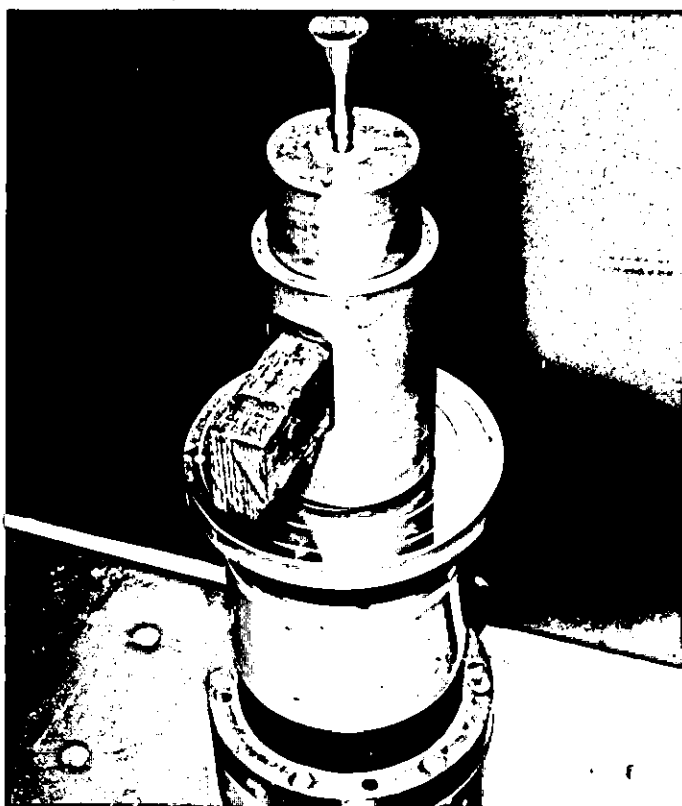
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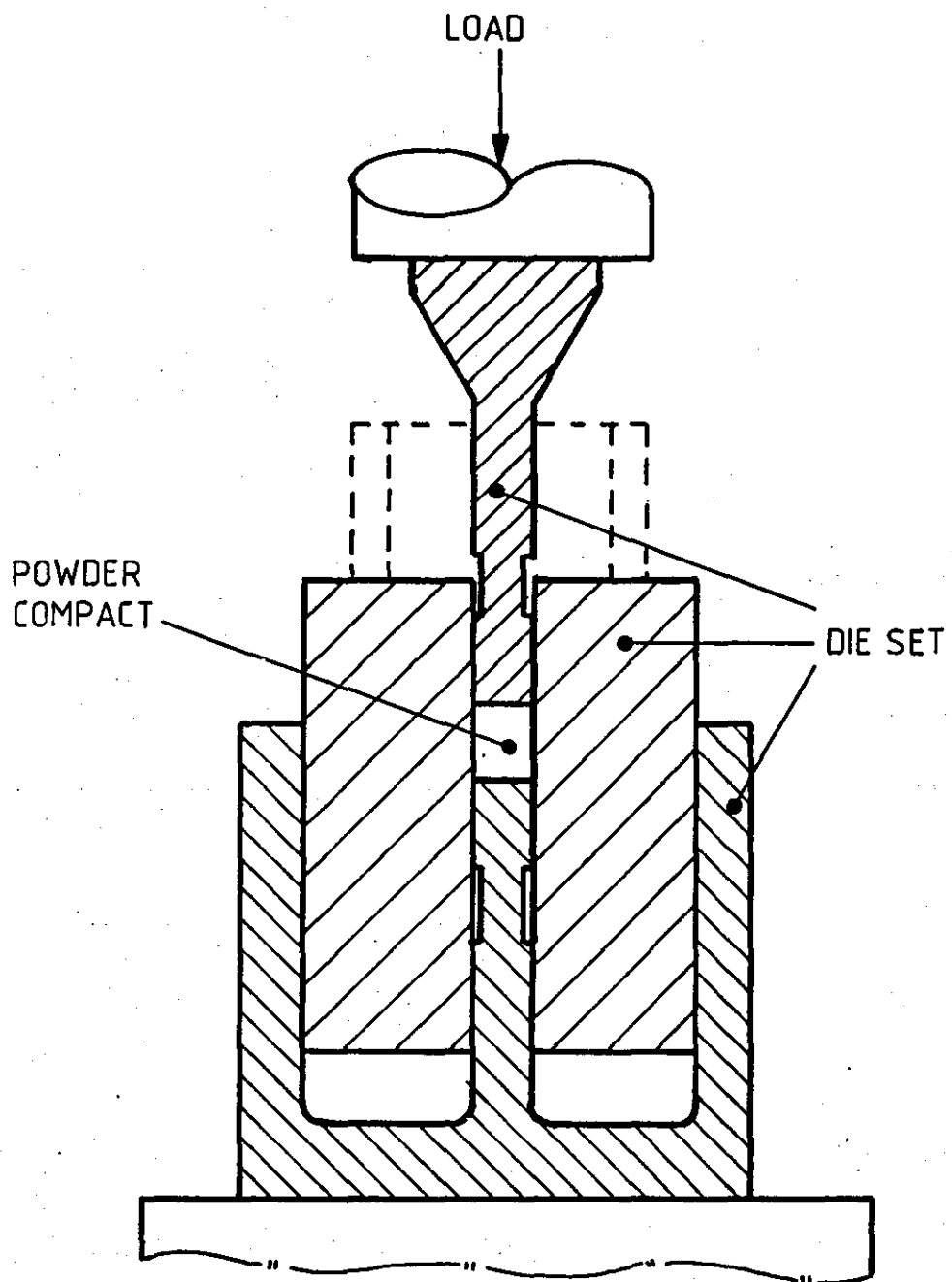
Figure 12. The sketch section of the 8mm die
used on Instron Testing Machine.

FIGURE 13

Compact in indirect diametrical compression with the strain gauge extensometer used for tensile stress, tensile strain and Modulus of Elasticity tests

via transducer

Instron Load

LOAD, σ

STRAIN, ε

specimen

Electronic Plotter

Amplifier

Strain Gauge (independent of Instron Machine)

GRAPH of

σ

ε
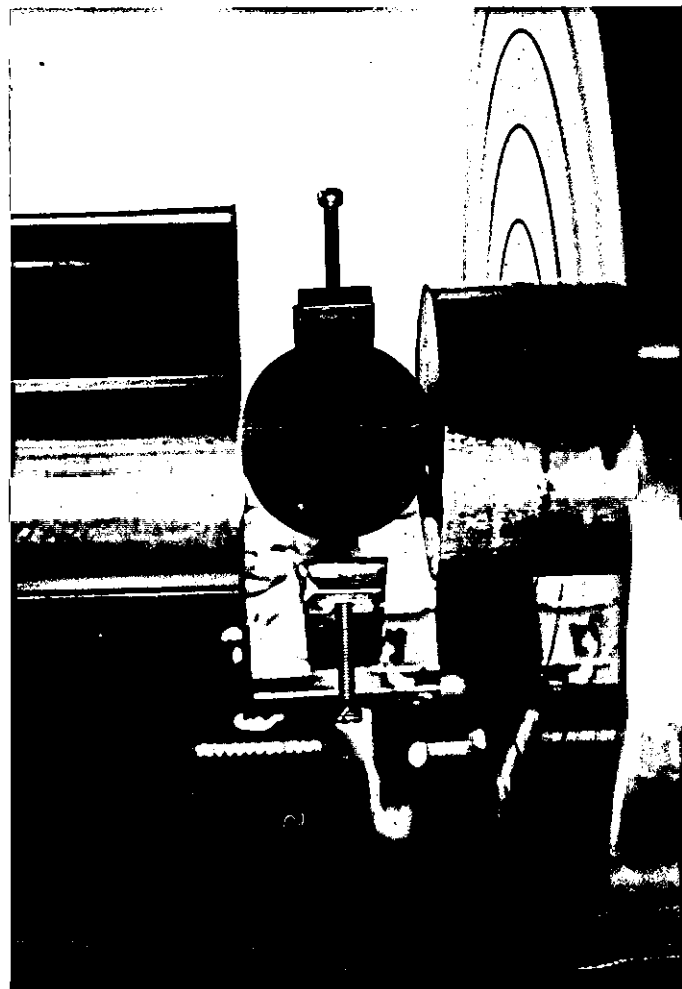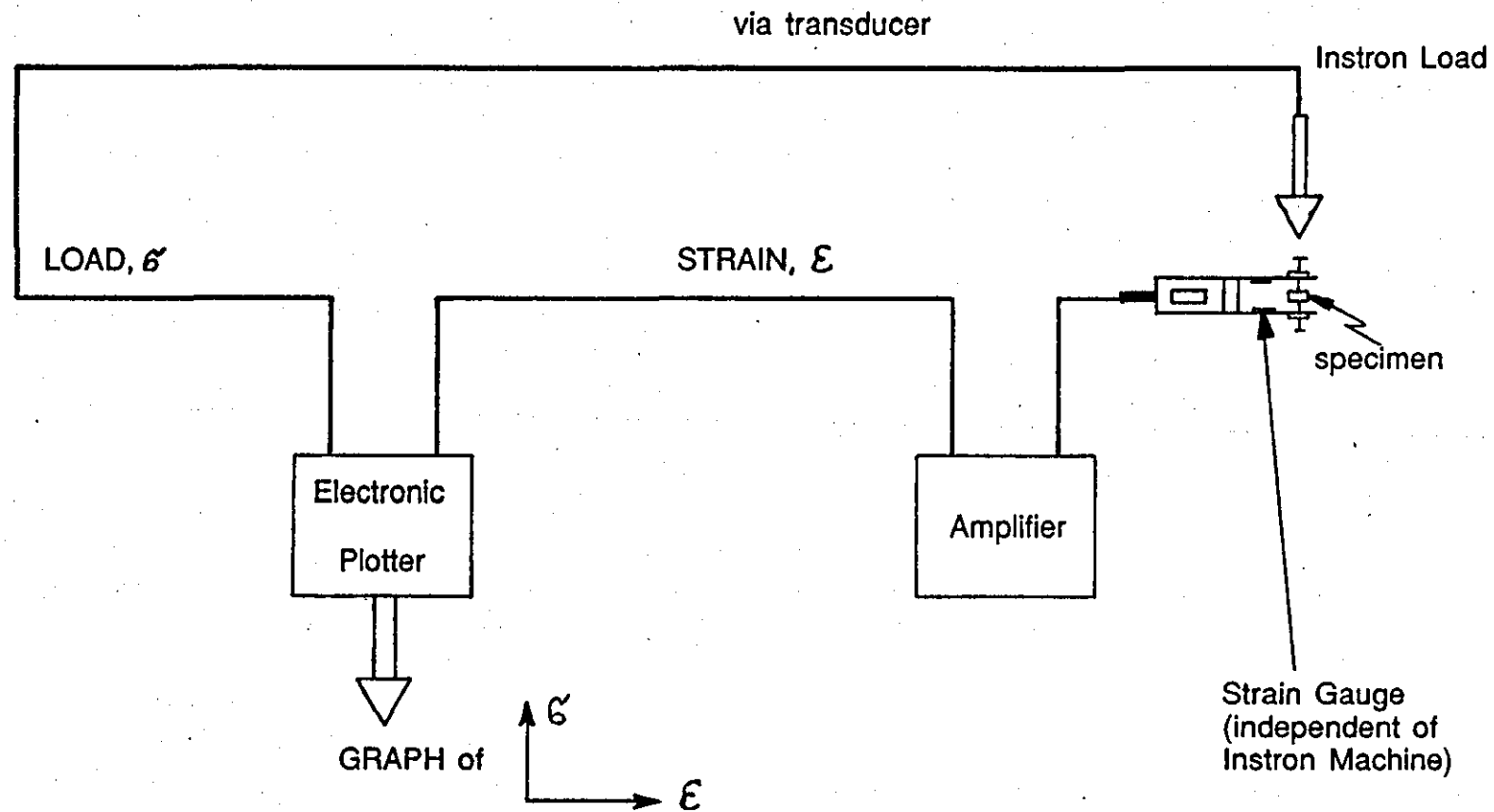
Figure 14 : The Layout of the reflective connections between the apparatus and, the input and output of strain gauge, amplifier, electronic plotter and Instron Testing Machine

MOVEMENT PROPORTIONAL
TO MOVEMENT ON THE
GRAPH PLOTTER

④ WIRES
TO AMPLIFIER

STRAIN GAUGE EXTENSIOMETER
ACTS AS A TRANSDUCER

SPECIMEN
(compact)

Figure 15 : Sketch of the Strain gauge extensiometer.

Solution compositions used to find effect
of copper sulphate amounts on amount
of copper deposited. (See Fig. 16.)

		Compositions		
		1	2	3
$CuSO_4.5H_2O$	g/l	2.5	5	10
$FeSO_4.H_2O$	g/l	5	5	5
H_2SO_4(conc)	cm^3/l	5	5	5

Total volume	10 litre (distilled water)
Temperature	room temperature ∼20°C
Powder charge	100g of iron particles in -150 +125μm size range

Fig. 16

Fig. 17.   DEPENDENCE OF Ni YIELD ON DURATION OF
COATING WITH TEMPERATURE AS PARMETER.
SiC powder: 2g/batch. Ni solution: 25ml/batch
Agitation: manual.
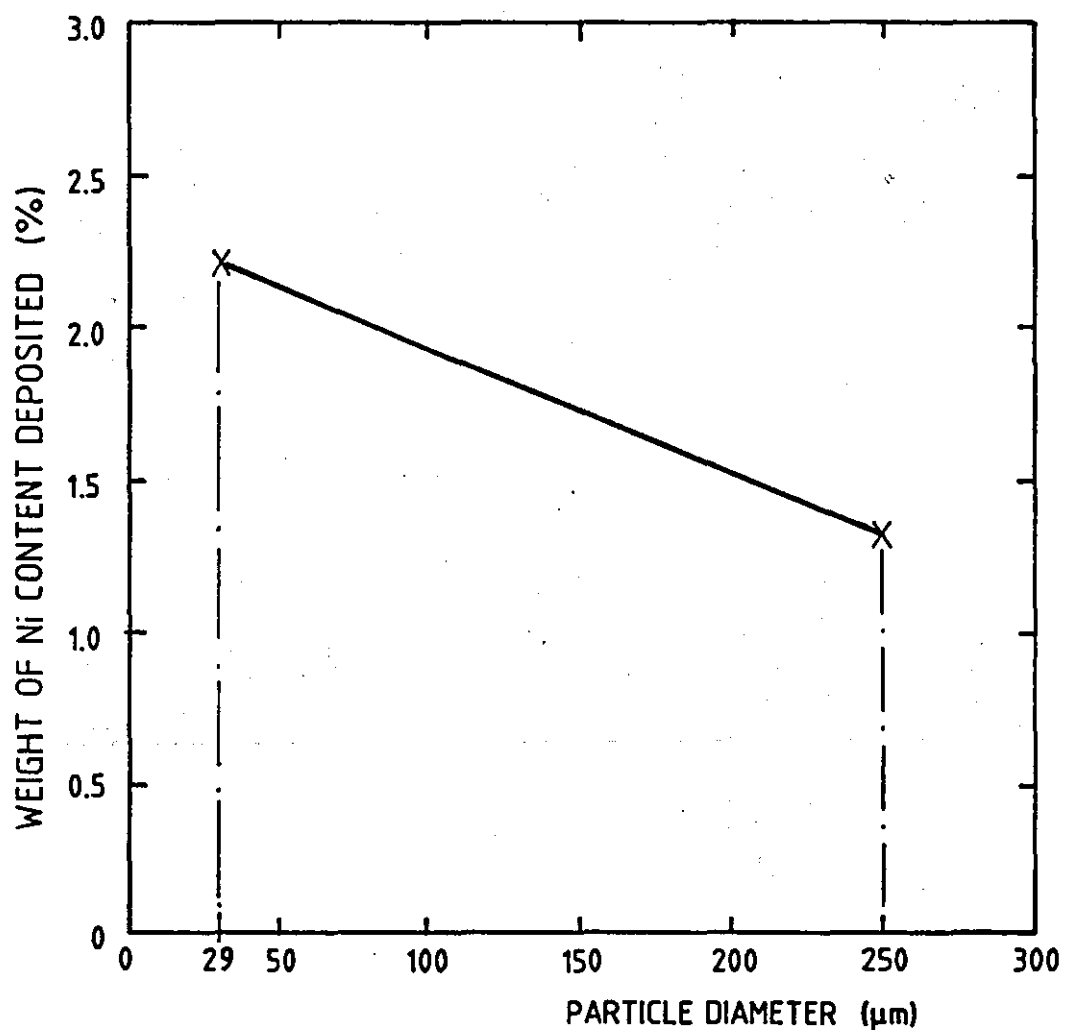
Fig. 18.   EFFECT OF PARTICLE SIZE ON Ni DEPOSITED (%)
           SiC powder : 2g/batch. Solution : 50ml.
           Temp : 45°C. Agitation : Wisk by motor.
           Time : 5 mins

Fig. 19.   EFFECT OF QUANTITY OF COATING SOLUTION
           AND METHOD OF AGITATION ON Ni YIELD
           SiC powder : 2g/batch. Temp : 45°C
           Time : 5 min
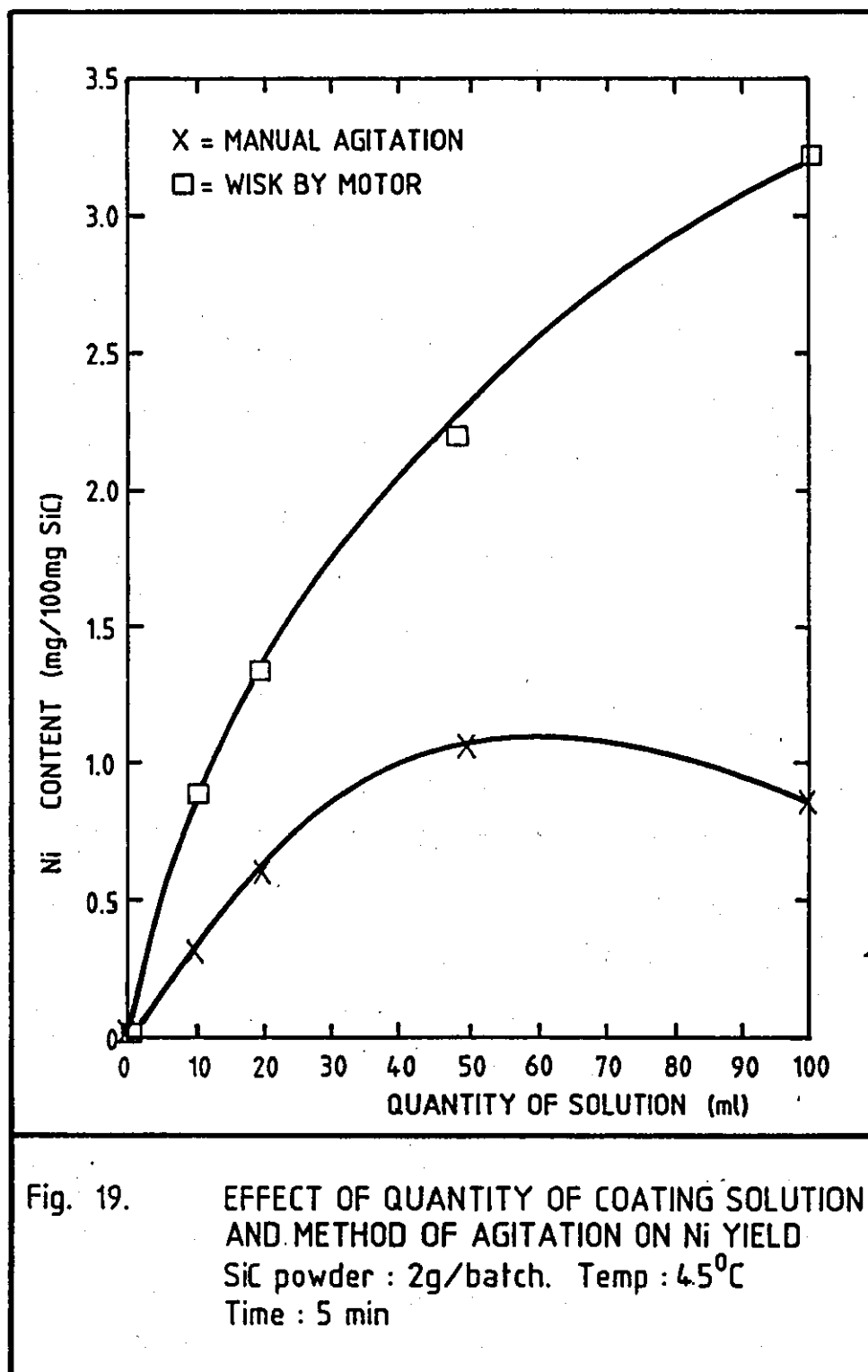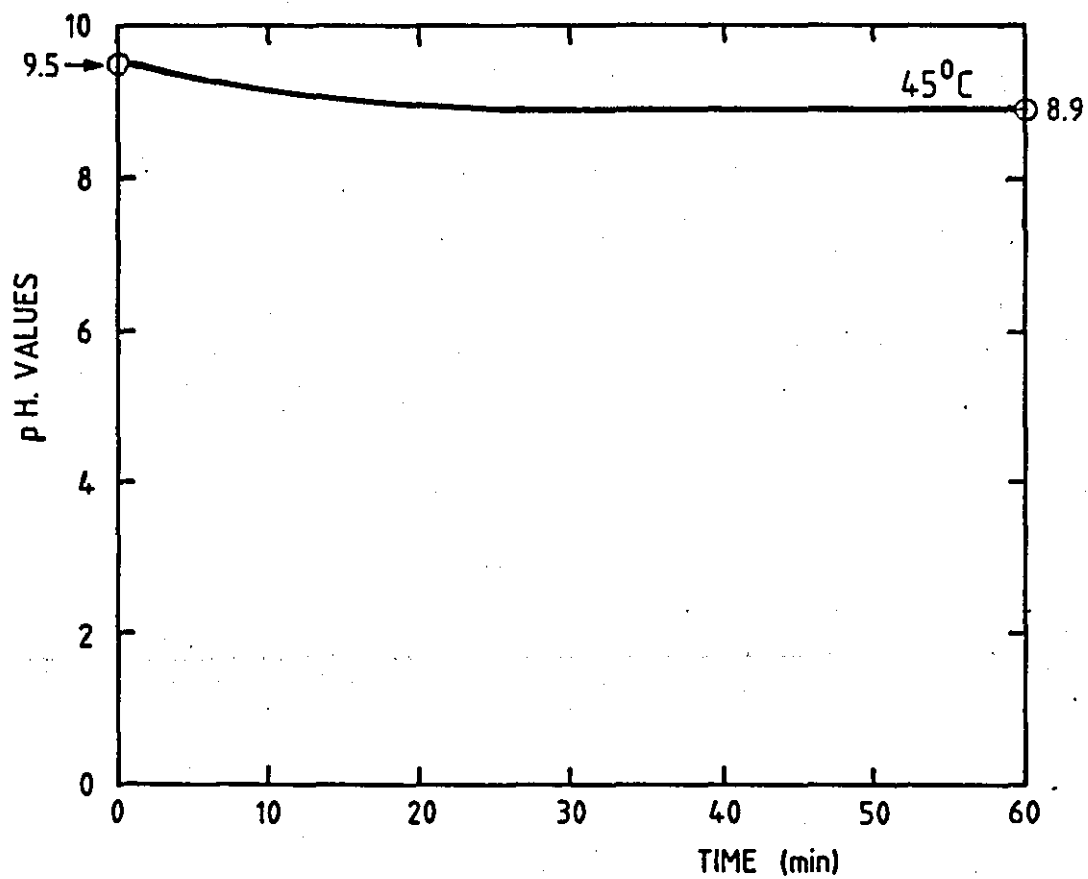
Fig. 20 .   EFFECT OF TIME DURATION ON THE
              pH OF Ni SOLUTION.
              SiC powder : 1g/batch.
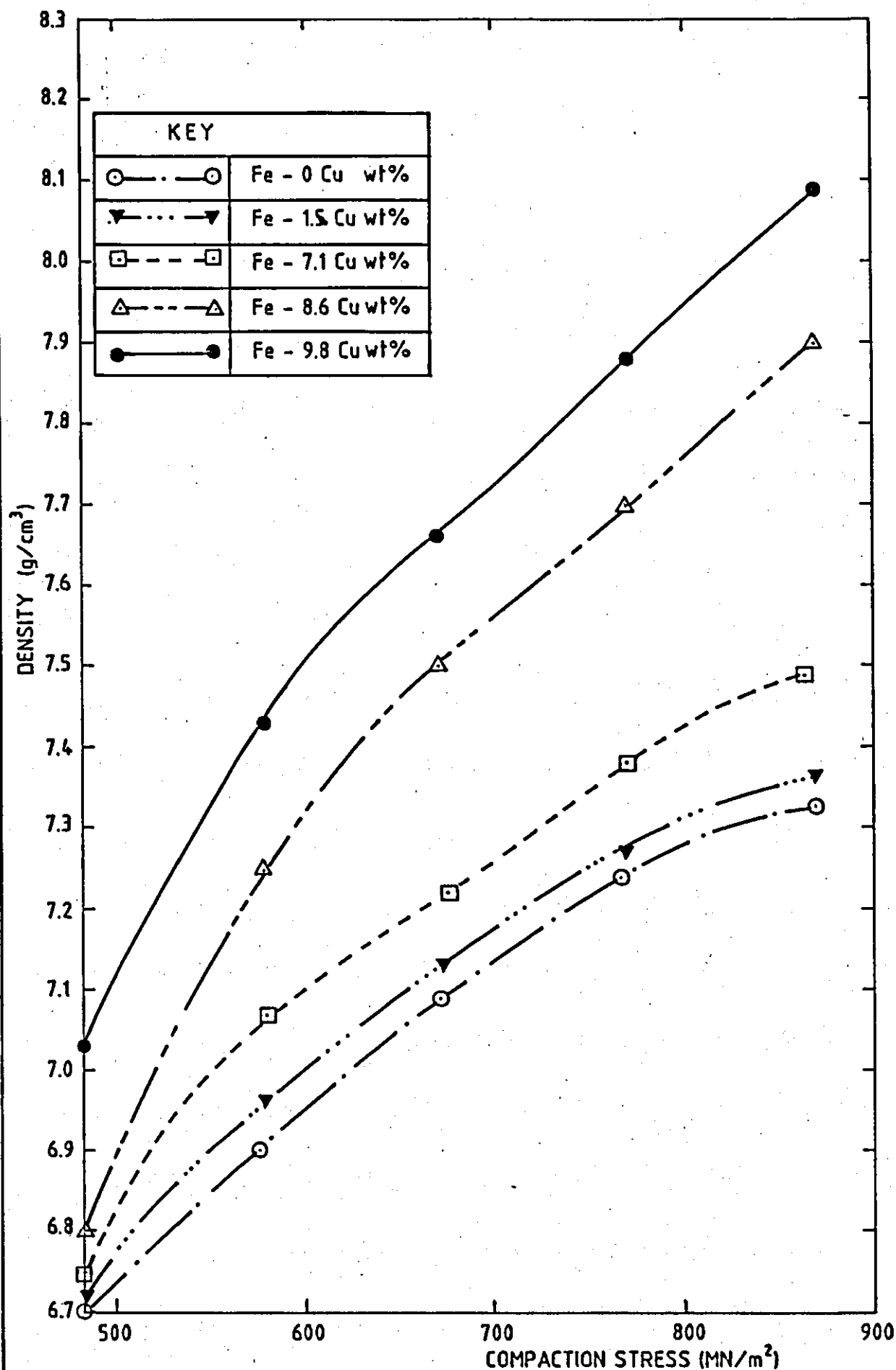
Fig. 21 DENSITY vs COMPACTION STRESS for COPPER COATED
IRON POWDER GREEN COMPACTS
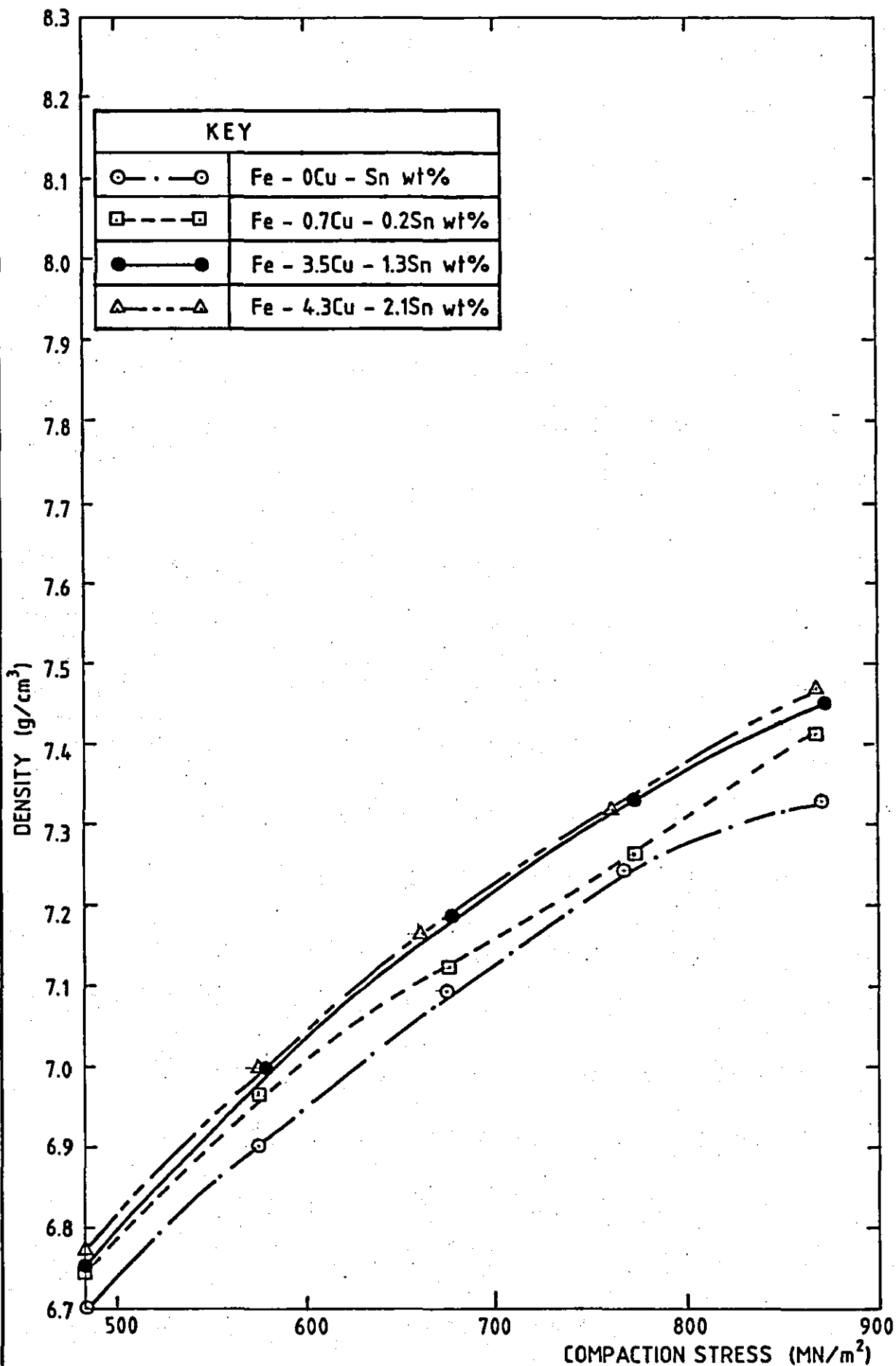
Fig. 22. DENSITY vs COMPACTION STRESS for COPPER-TIN ALLOY COATED IRON POWDER GREEN COMPACTS.
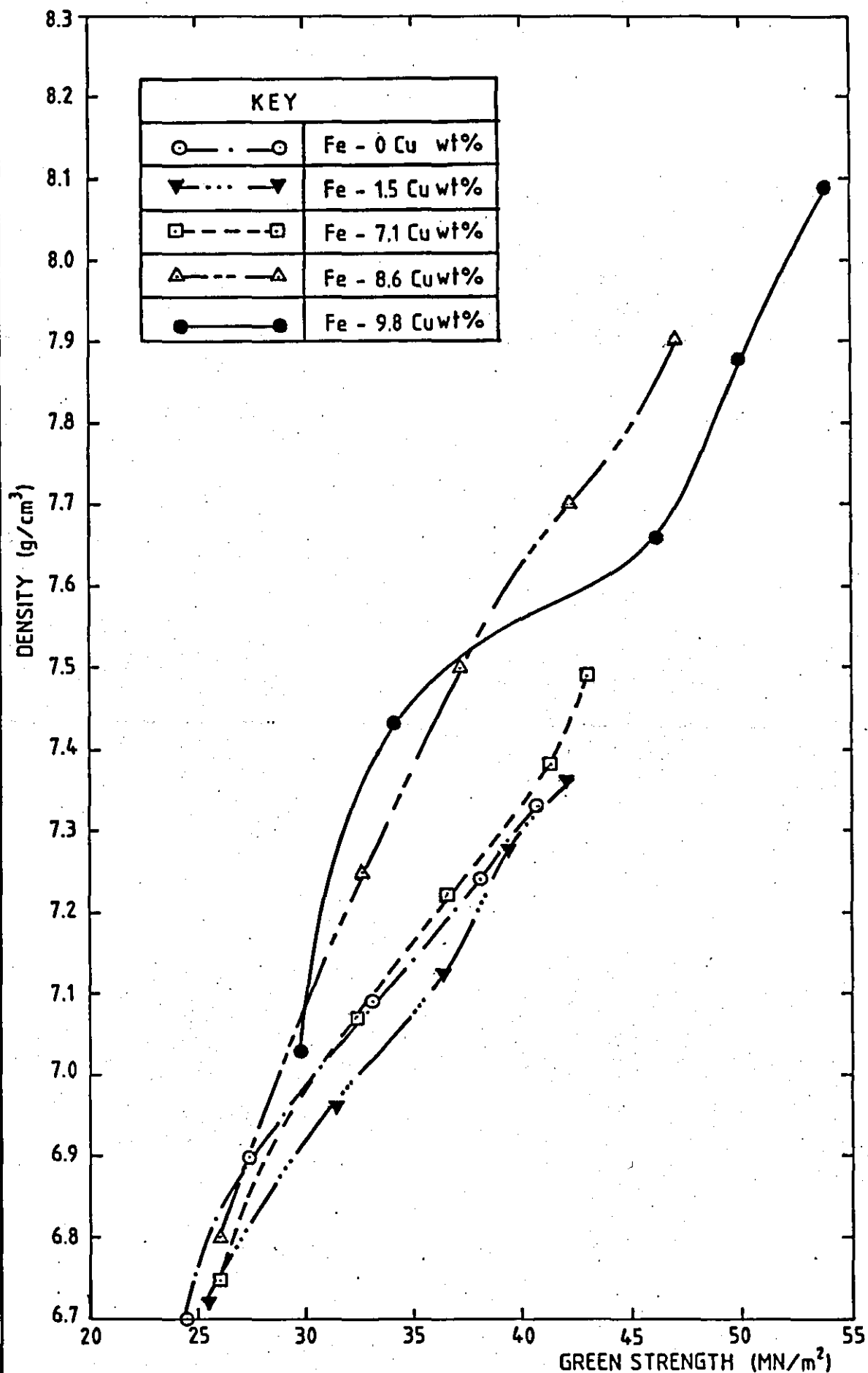
Fig. 23   DENSITY vs GREEN STRENGTH for COPPER
COATED IRON POWDER GREEN COMPACTS

KEY

⊙ —·—·— ⊙	Fe – 0Cu – 0 Sn wt%
▫ — — — ▫	Fe – 0.7Cu – 0.2Sn wt%
● ———— ●	Fe – 3.5Cu – 1.3Sn wt%
△ —·—·— △	Fe – 4.3Cu – 2.1Sn wt%

DENSITY (g/cm²)

GREEN STRENGTH (MN/m²)
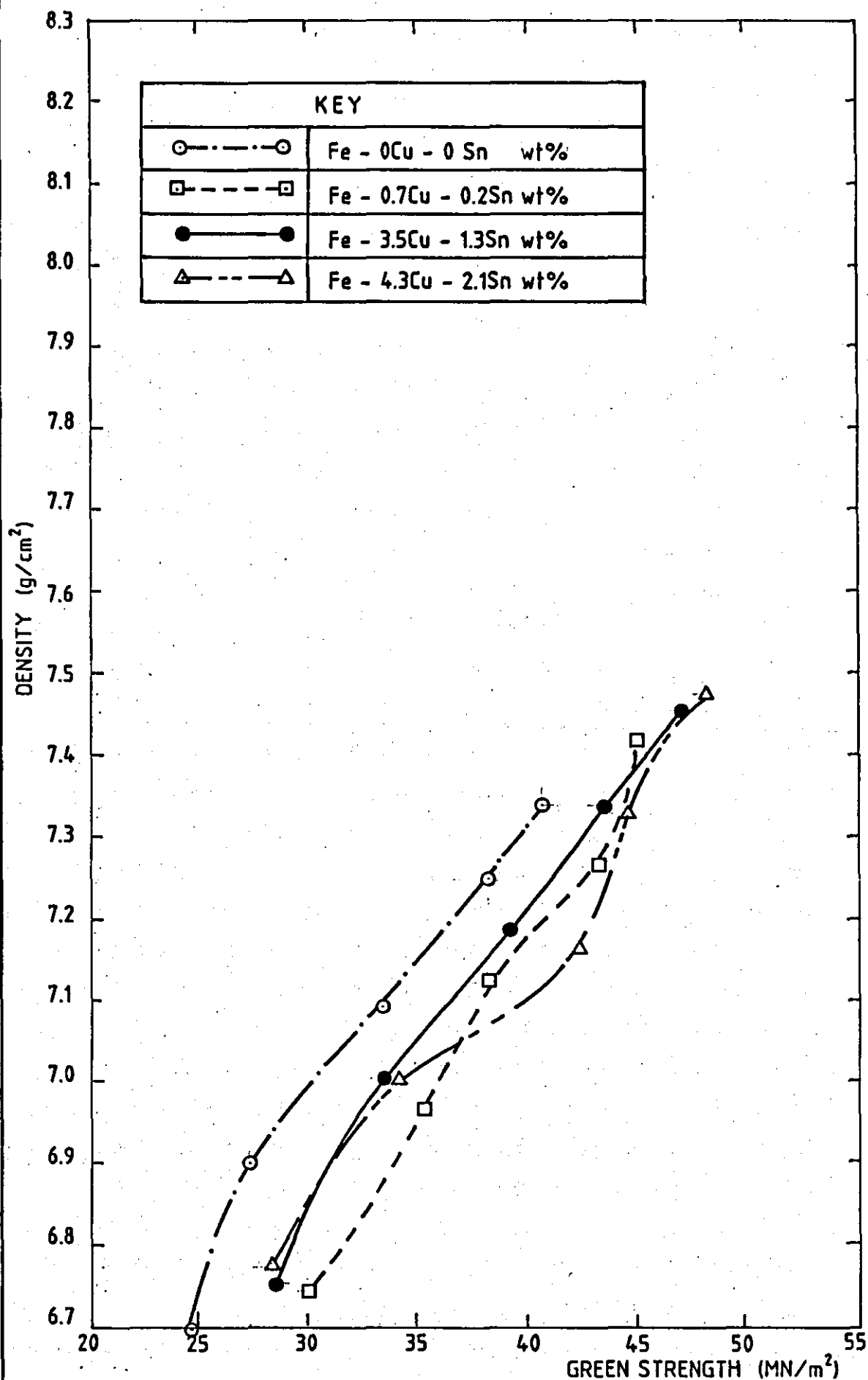
Fig. 24.   DENSITY vs GREEN STRENGTH for COPPER-TIN ALLOY
COATED IRON POWDER GREEN COMPACTS

Fig. 25 V H N vs GREEN DENSITY for COPPER
COATED IRON POWDER GREEN COMPACTS

KEY

⊙ — · — ⊙	Fe – 0 Cu wt%
▼ ···· ▼	Fe – 1.5 Cu wt%
⊡ – – – ⊡	Fe – 7.1 Cu wt%
△ – · – △	Fe – 8.6 Cu wt%
● —— ●	Fe – 9.8 Cu wt%
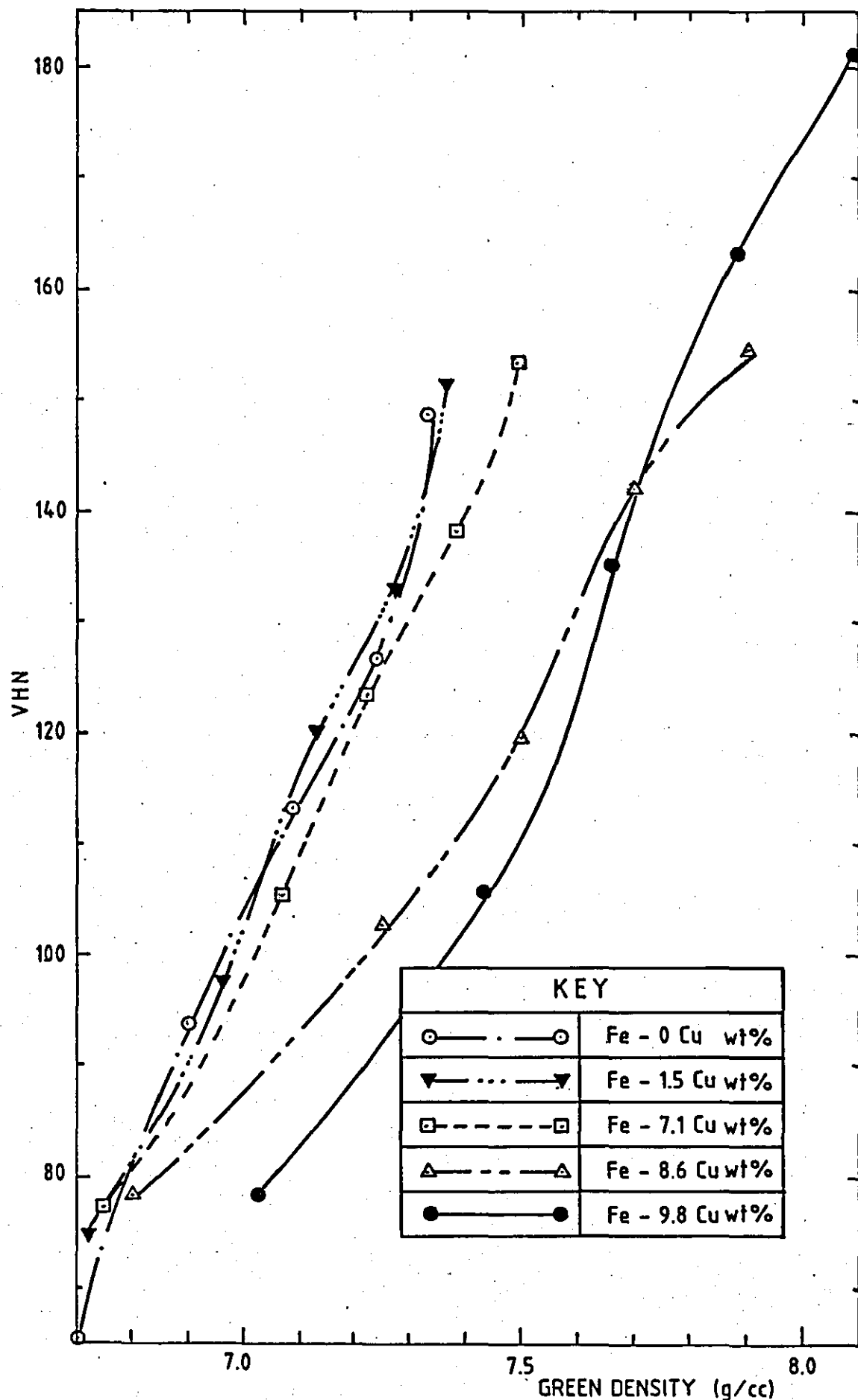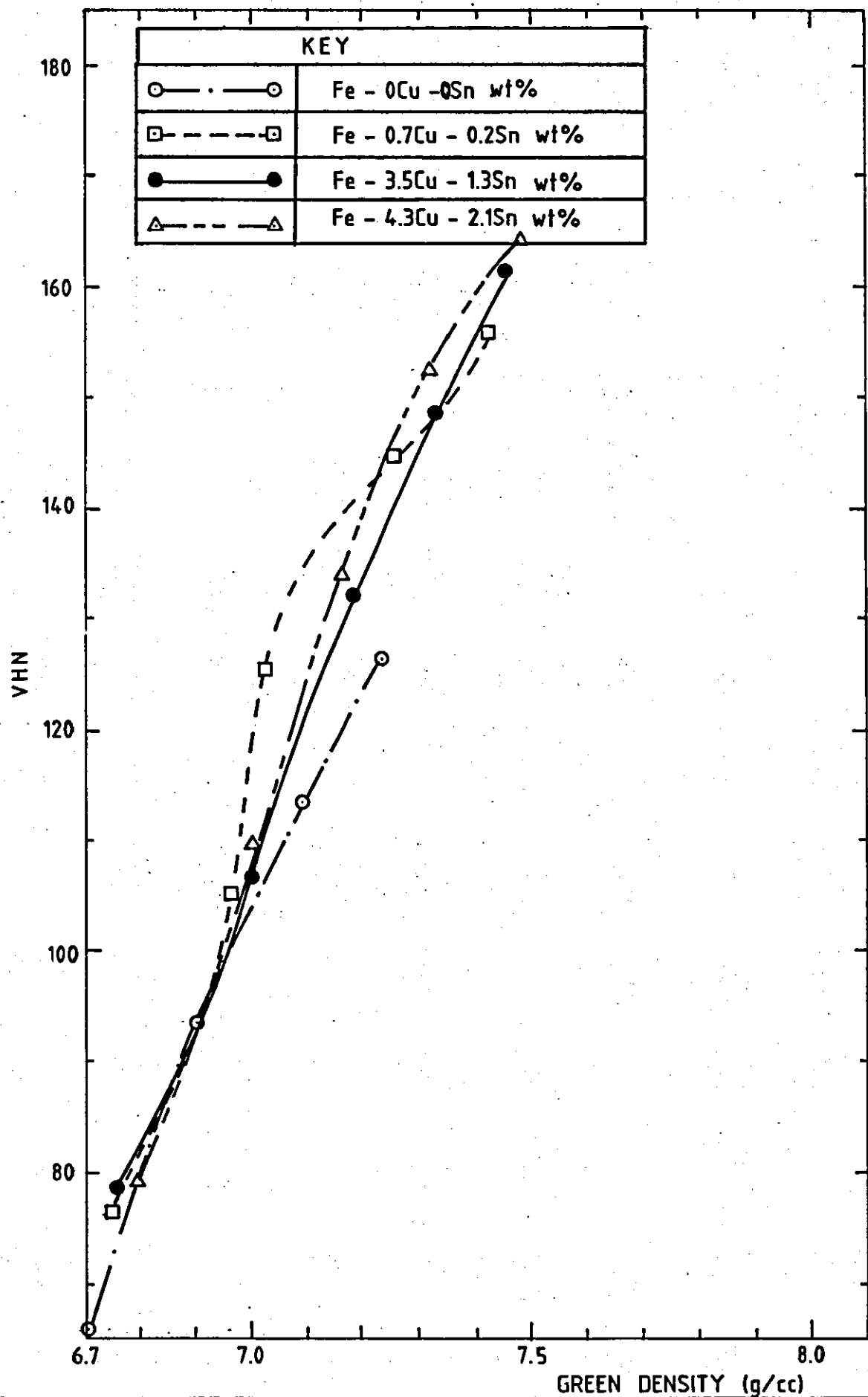
Fig. 26.　VHN vs GREEN DENSITY for COPPER-TIN ALLOY
COATED IRON POWDER GREEN COMPACTS
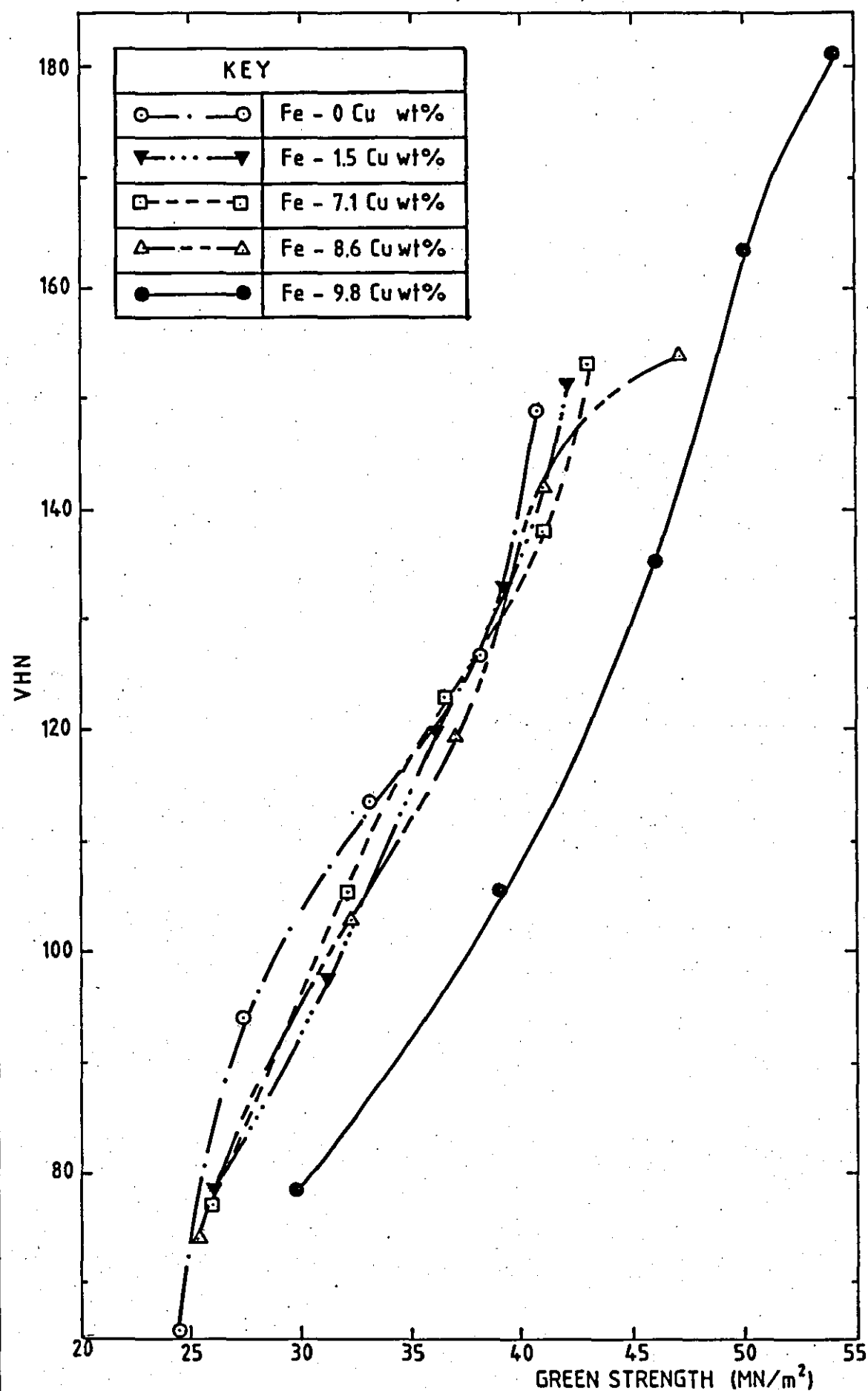
Fig. 27   V H N vs GREEN STRENGTH for COPPER
COATED IRON POWDER GREEN COMPACTS
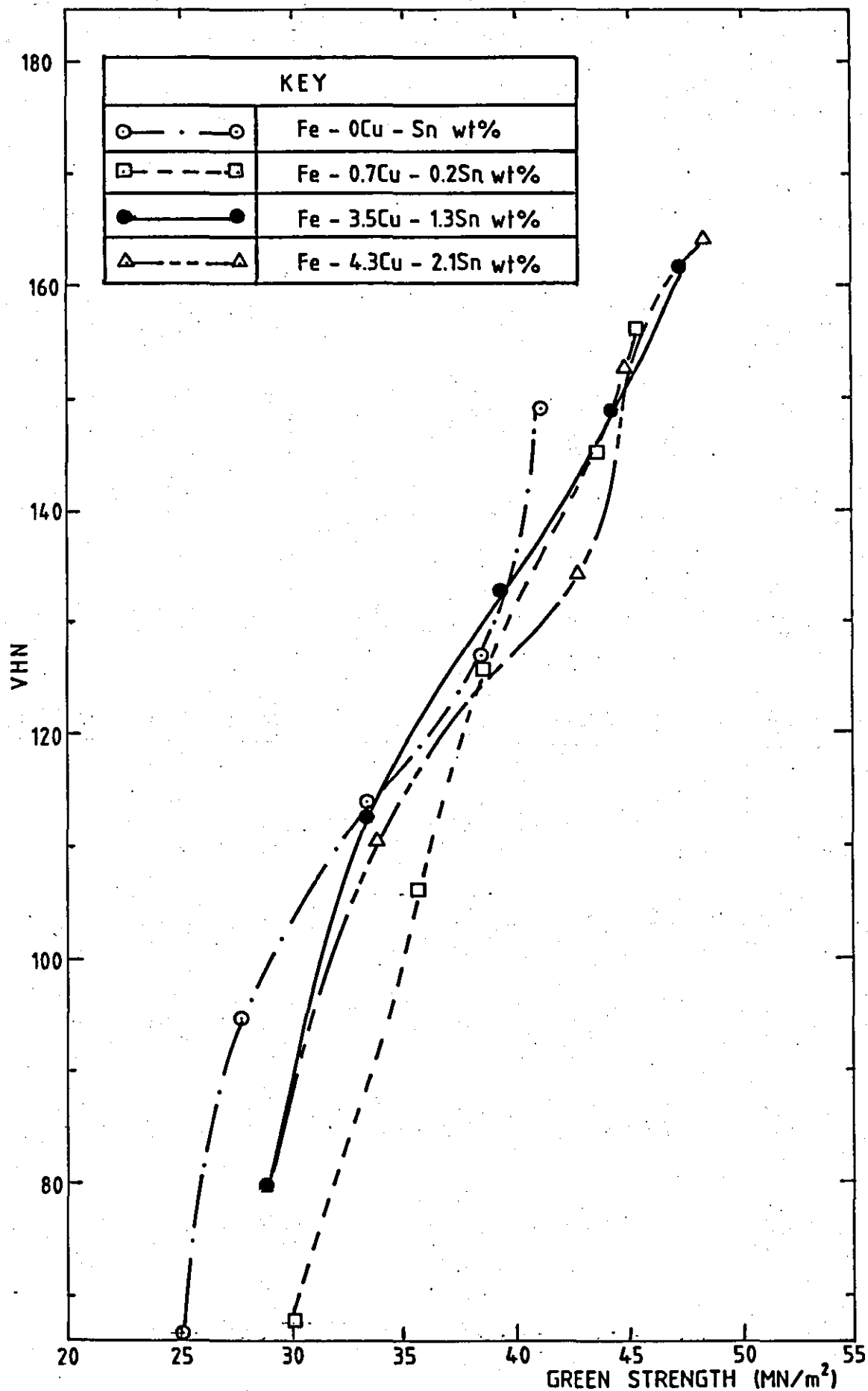
Fig. 28.   VHN vs GREEN STRENGTH for COPPER-TIN
ALLOY COATED IRON POWDER GREEN COMPACTS
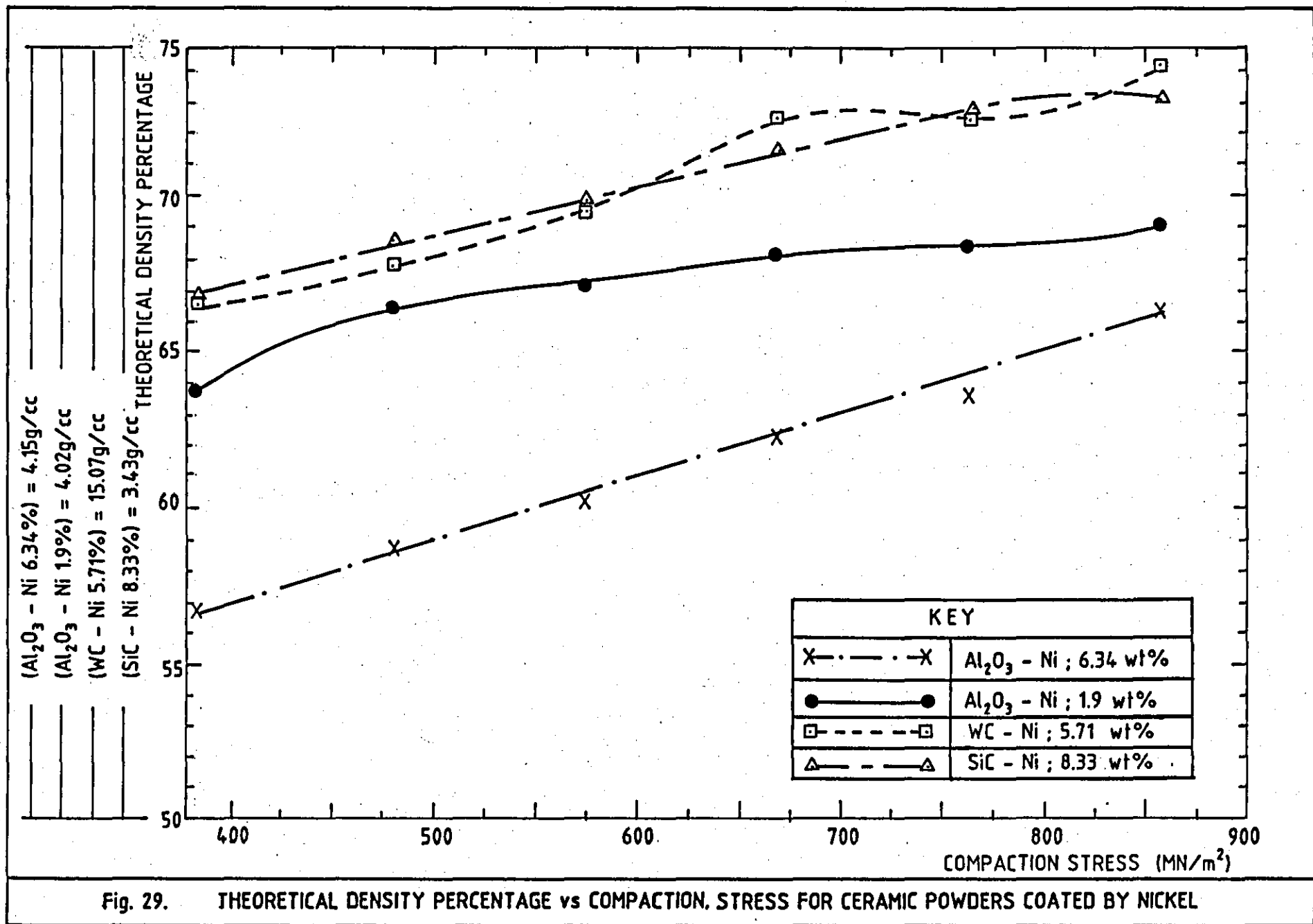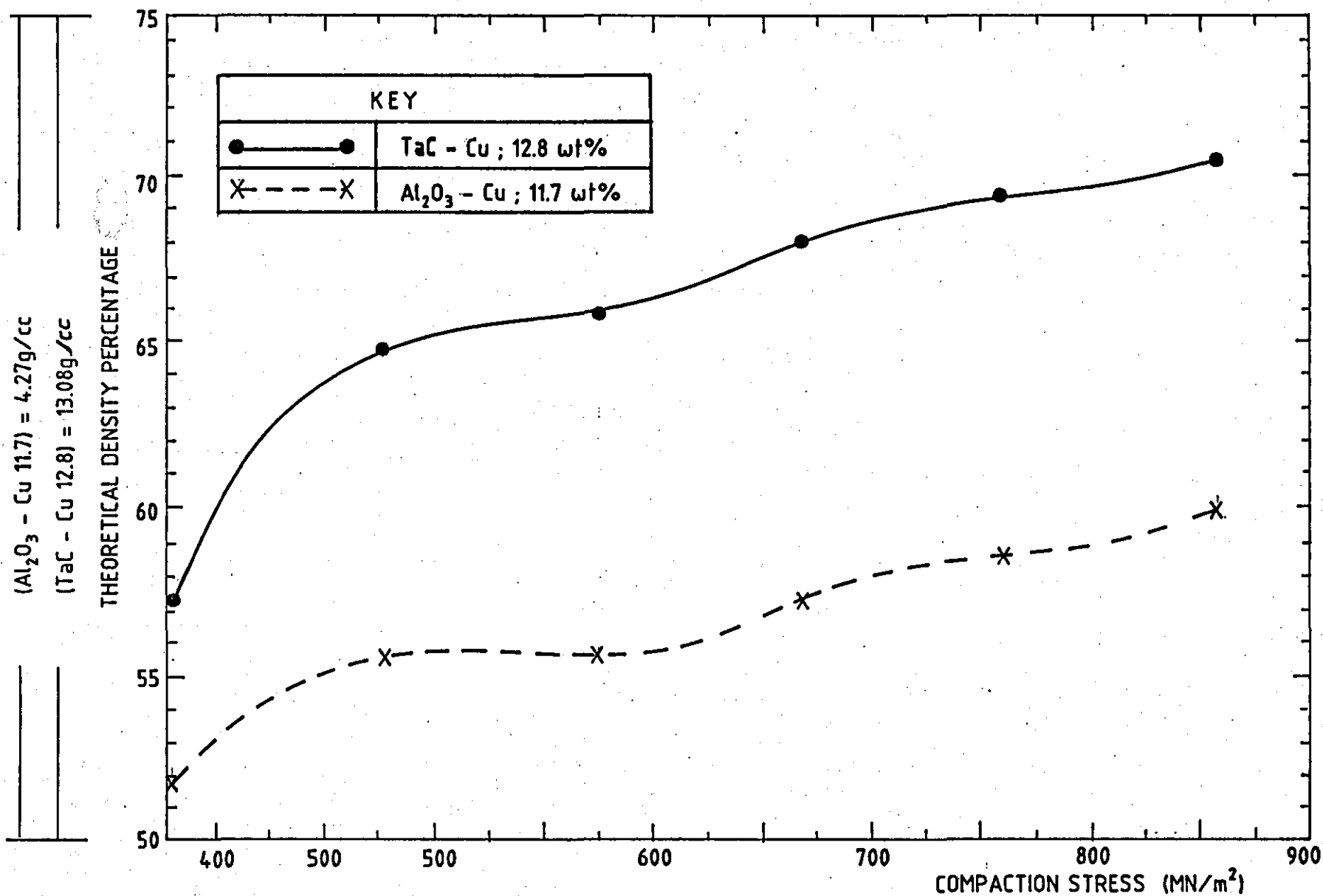
Fig. 29.   THEORETICAL DENSITY PERCENTAGE vs COMPACTION. STRESS FOR CERAMIC POWDERS COATED BY NICKEL

Fig. 30.   THEORETICAL DENSITY PERCENTAGE vs COMPACTION STRESS FOR CERAMIC POWDERS COATED BY COPPER

(Al$_2$O$_3$ - Ni 6.34%) = 4.15g/cc

(Al$_2$O$_3$ - Ni 1.9%) = 4.02g/cc

(WC - Ni 5.71%) = 15.07g/cc

(SiC - Ni 8.33%) = 3.43g/cc

THEORETICAL DENSITY PERCENTAGE

KEY	
✕——·——·——✕	Al$_2$O$_3$ - Ni ; 6.34 wt%
●————●	Al$_2$O$_3$ - Ni ; 1.9 wt%
◻----◻	WC - Ni ; 5.71 wt%
△——·——·——△	SiC - Ni ; 8.33 wt%

TENSILE STRENGTH   (NM/m^2)
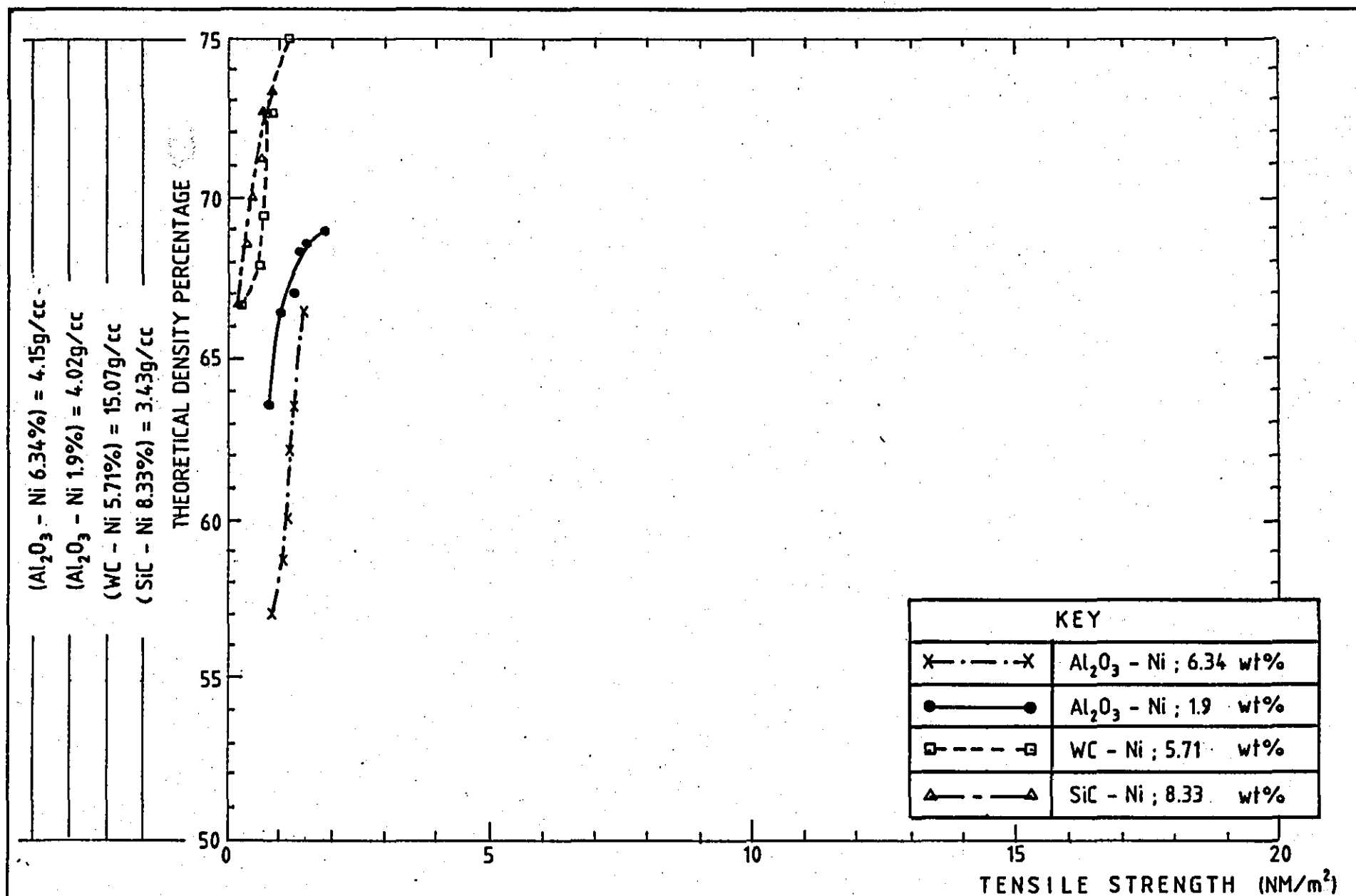
Fig. 31. THEORETICAL DENSITY PERCENTAGE vs TENSILE STRENGTH FOR CERAMIC POWDERS COATED BY NICKEL

Fig. 32. THEORETICAL DENSITY PERCENTAGE vs TENSILE STRENGTH FOR CERAMIC POWDERS COATED BY COPPER

Fig. 33.   THEORETICAL DENSITY PERCENTAGE vs TENSILE STRAIN FOR CERAMIC POWDERS COATED BY NICKEL

Fig. 34.   THEORETICAL DENSITY PERCENTAGE vs TENSILE STRAIN FOR CERAMIC POWDERS COATED BY COPPER

THEORETICAL DENSITY PERCENTAGE

(Al₂O₃ - Ni 6.34%) = 4.15g/cc
(Al₂O₃ - Ni 1.9%) = 4.02g/cc
(WC - Ni 5.71%) = 15.07g/cc
(SiC - Ni 8.33%) = 3.43g/cc

KEY	
✕ —·—·— ✕	Al₂O₃ – Ni ; 6.34 wt%
● —— ●	Al₂O₃ – Ni ; 1.9 wt%
⊡ ——— ⊡	WC – Ni ; 5.71 wt%
△ ——— △	SiC – Ni ; 8.33 wt%
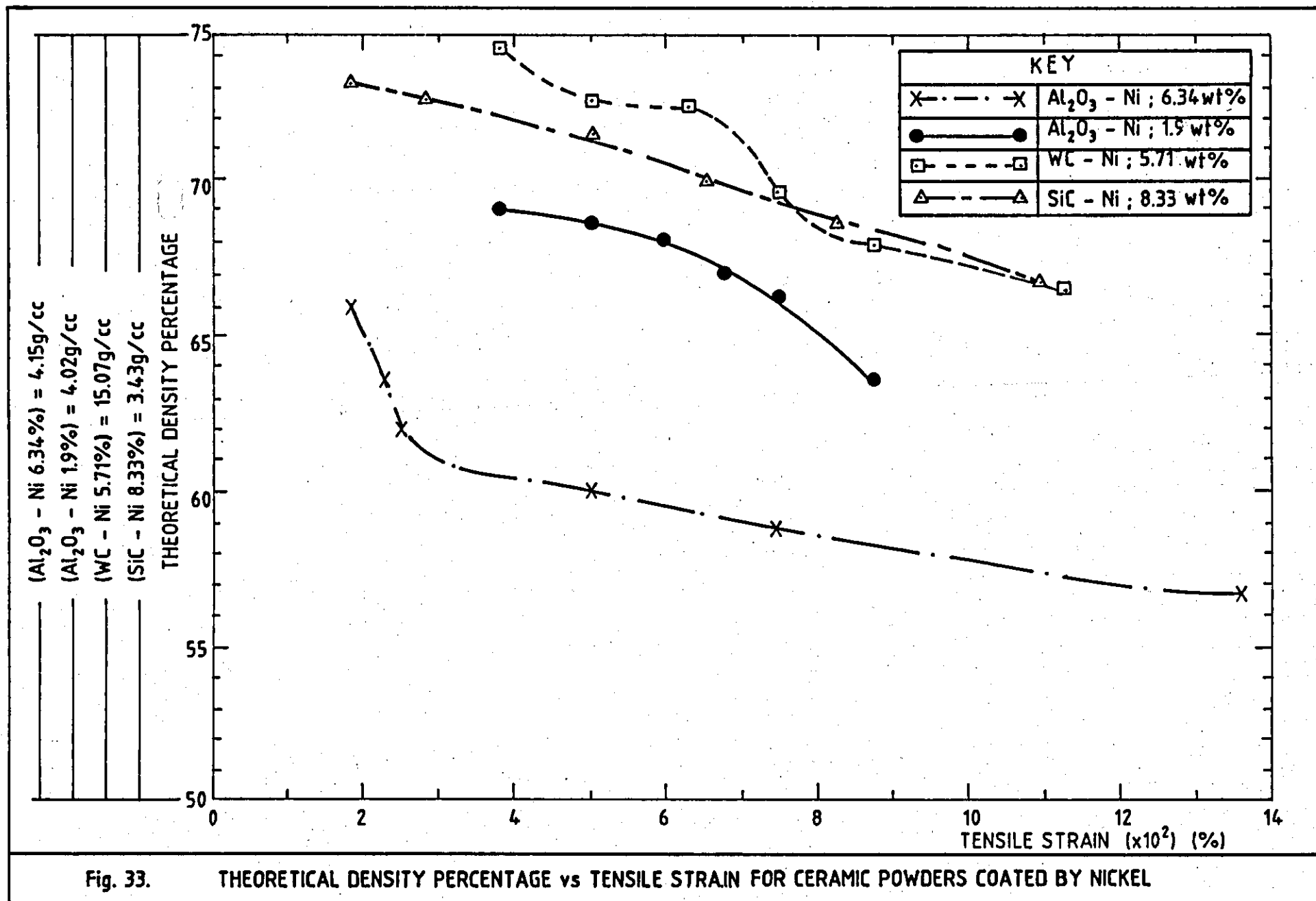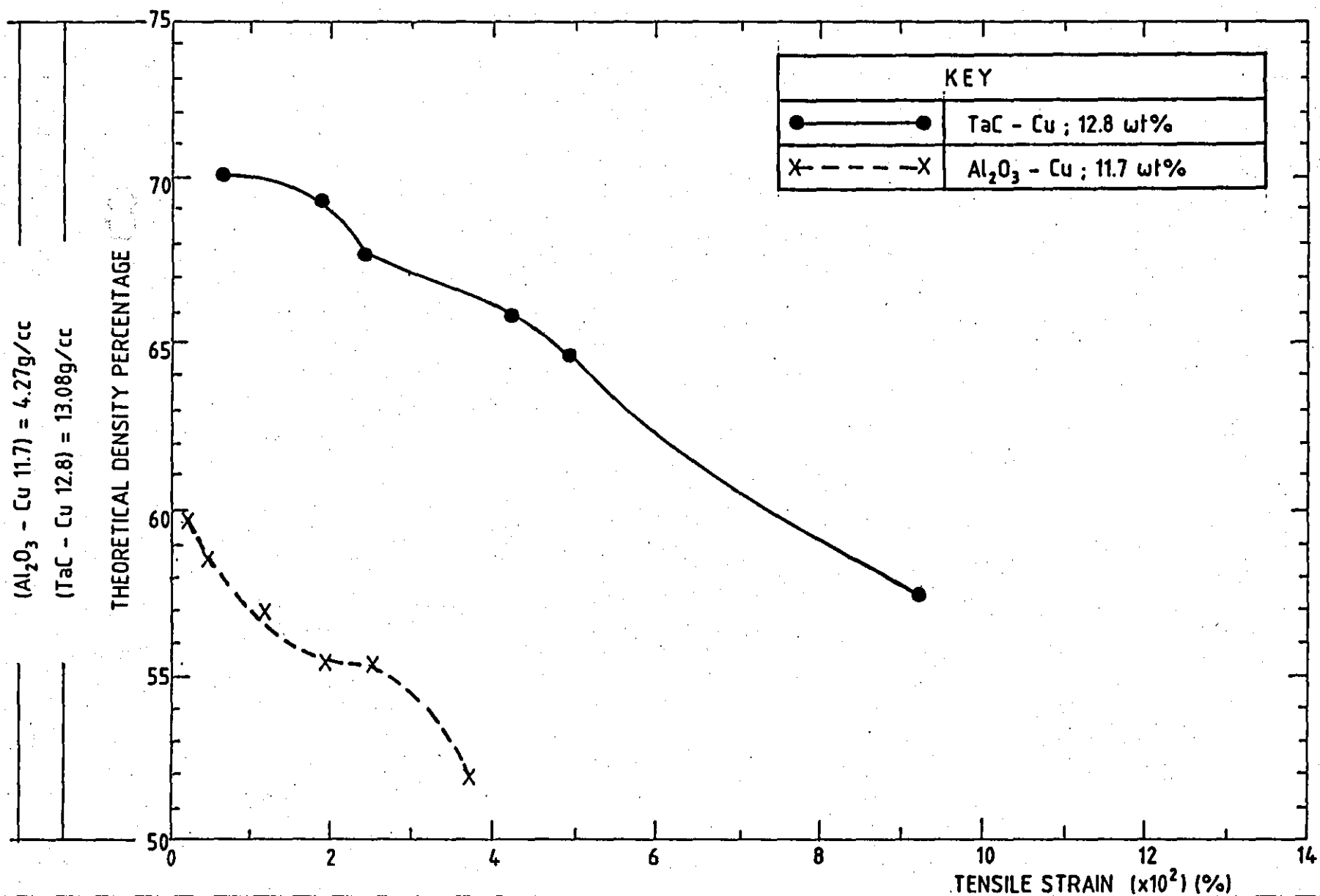
YOUNGS MODULUS OF ELASTICITY (MN/m²)
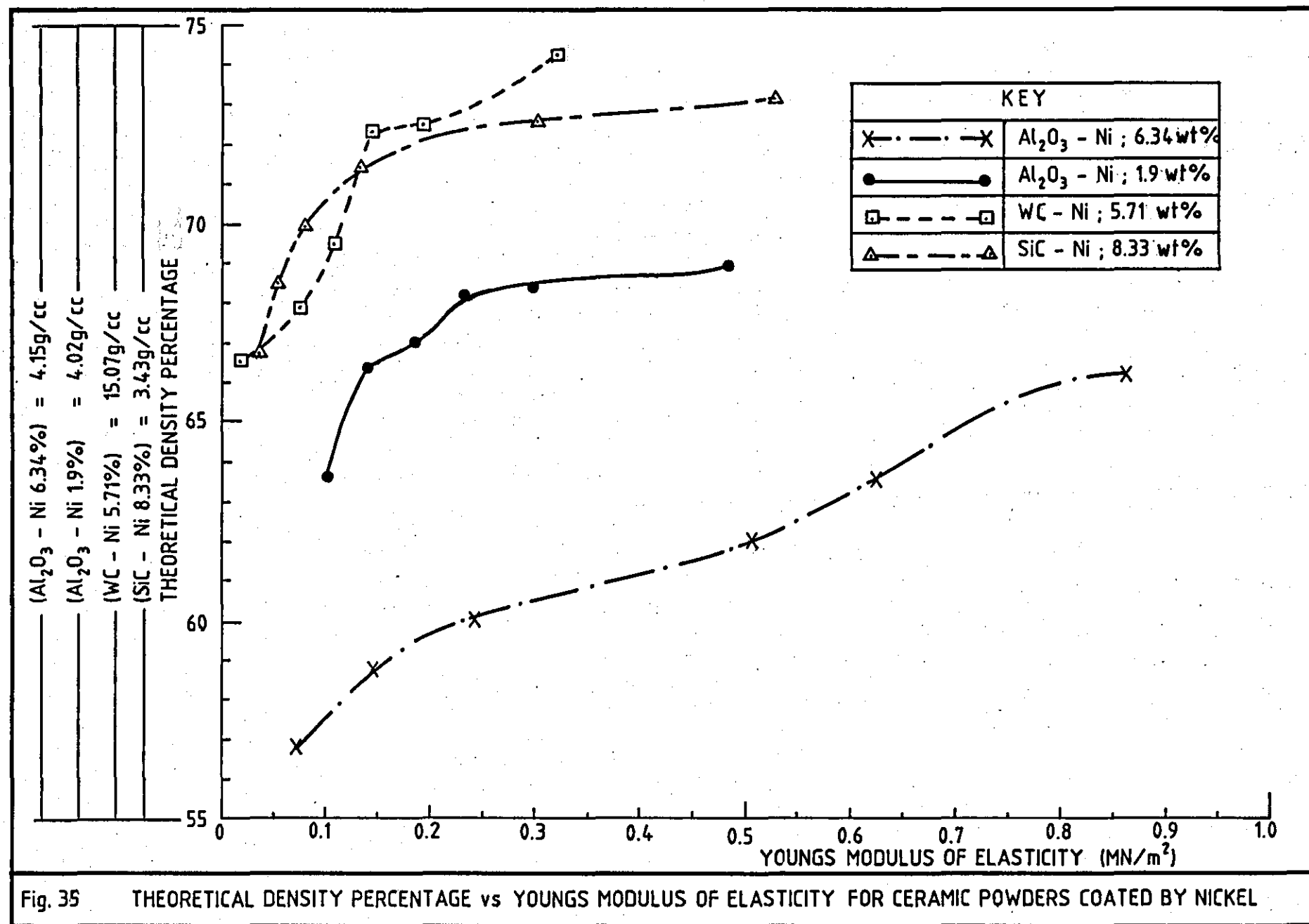
Fig. 35   THEORETICAL DENSITY PERCENTAGE vs YOUNGS MODULUS OF ELASTICITY FOR CERAMIC POWDERS COATED BY NICKEL
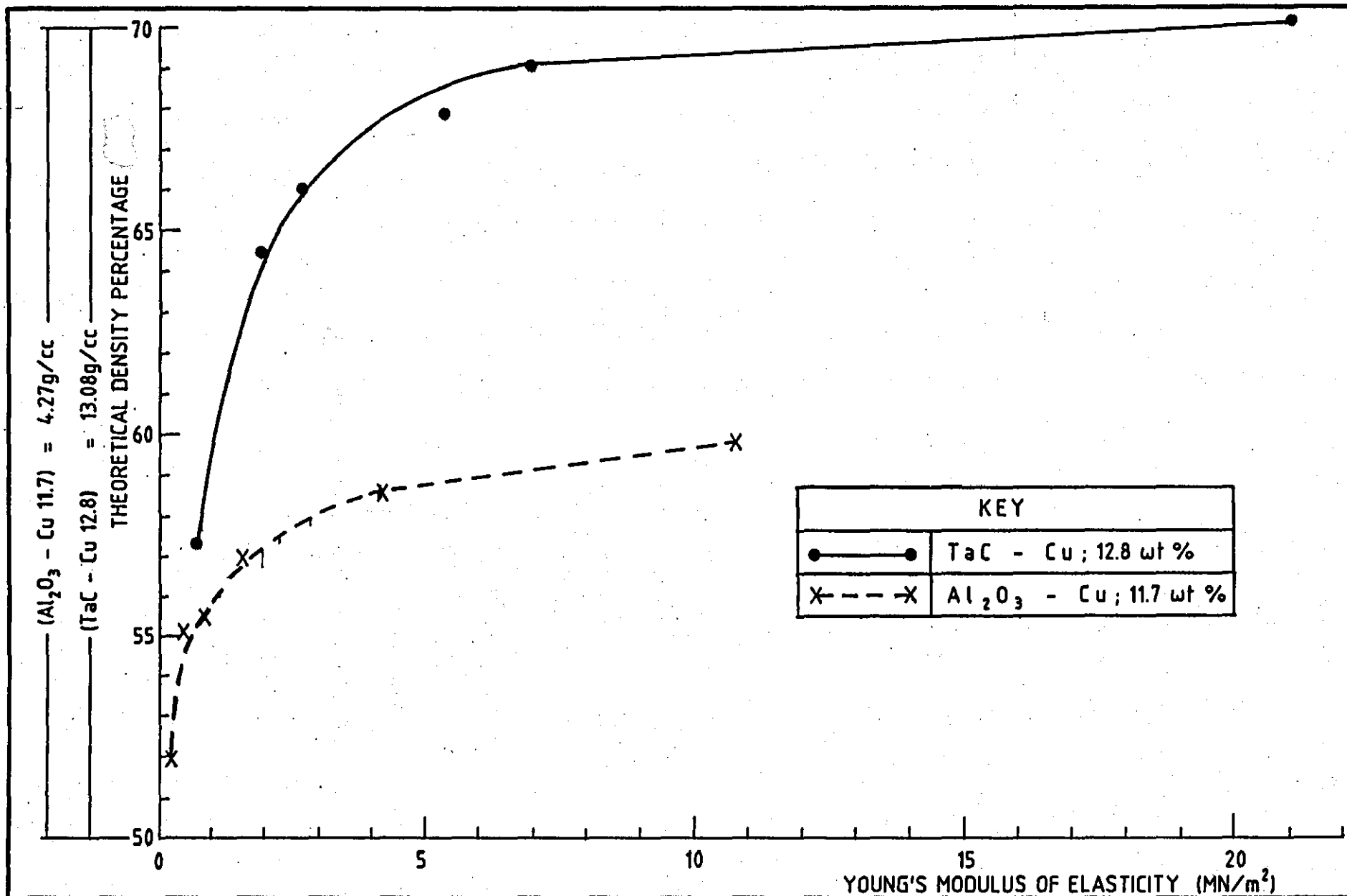
Fig. 36   THEORETICAL DENSITY PERCENTAGE vs YOUNGS MODULUS OF ELASTICITY FOR CERAMIC POWDERS COATED BY COPPER

FIGURE 37

Optical Micrograph of Mannesman WPL 200 Iron Powder,
Sectioned and Polished (a) X20; (b) X100
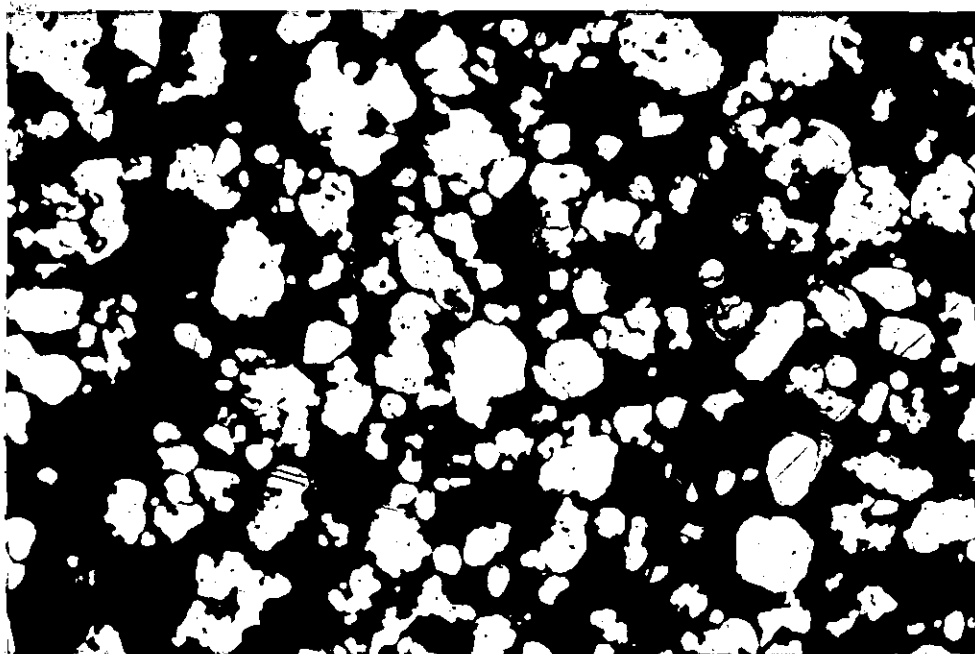
(a)
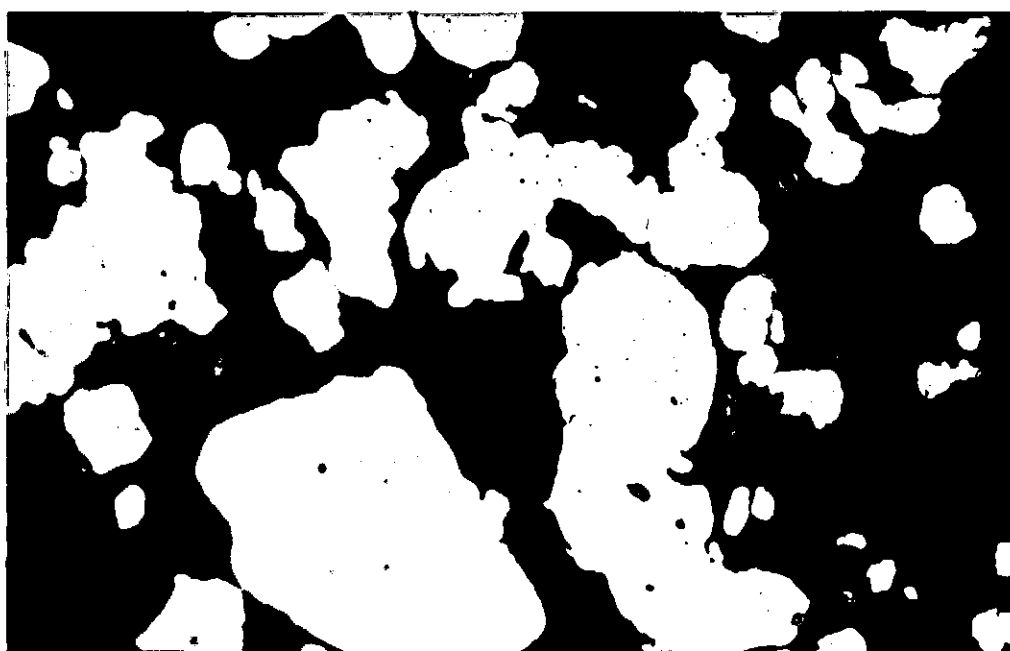
(b)

FIGURE 38

a) Optical micrographs of Mannesman WPL 200 iron
    powder (-150 +125 μm) coated with 7.1 Cu by
    displacement method in fluidised bed (X10)

b) Optical micrographs of Mannesman WPL 200 iron
    powder (-150 +125 μm) coated with 4.3 Cu-2.1 Sn by
    displacement method in bath (X10)

(a)

(b)

FIGURE 39

a) Optical micrographs of Fe-7.1 Cu wt% particles polished and sectioned, plated by displacement method in fluidised bed (X50)

b) Optical micrograph of Fe-3.5 Cu-1.3 Sn wt% particles polished and sectioned, plated by displacement method in fluidised bed (X50)
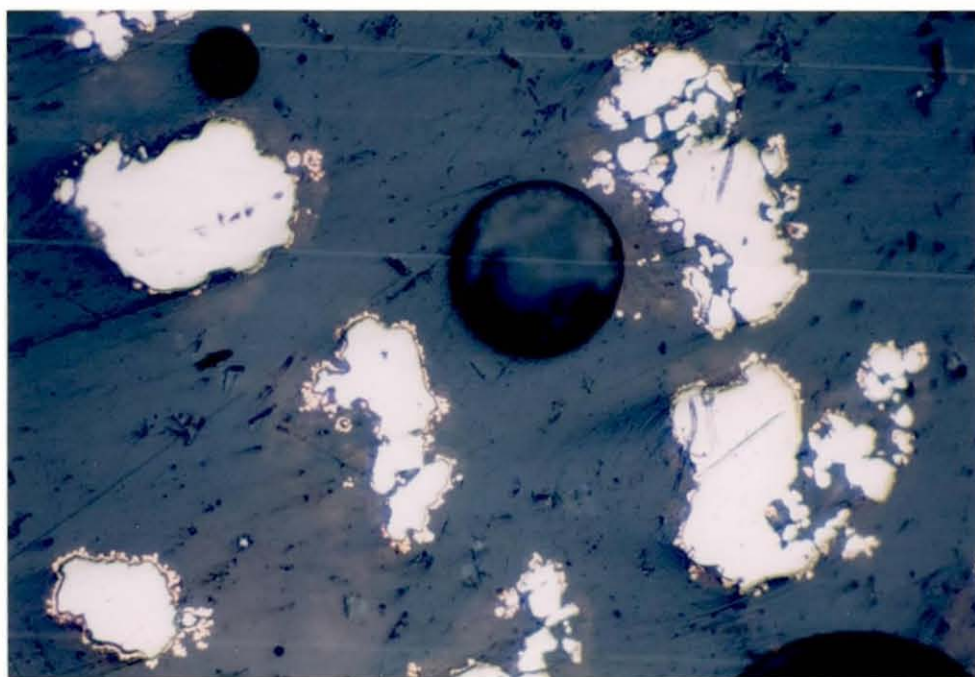
(a)
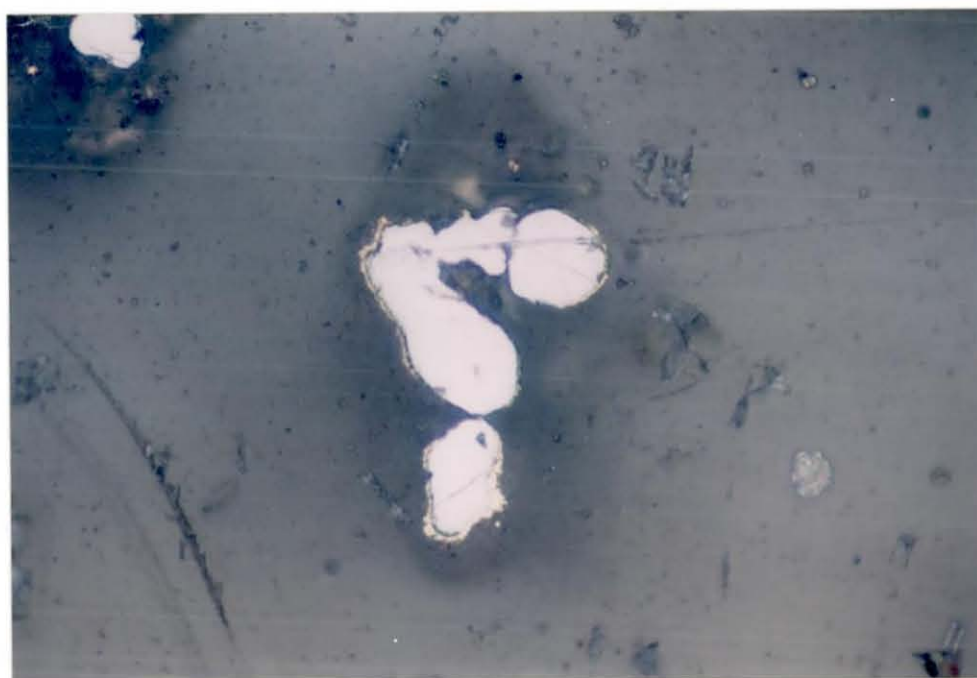
(b)

FIGURE 40

SEM Photograph of Mannesman WPL 200 Iron Powder
a) X100
b) X500
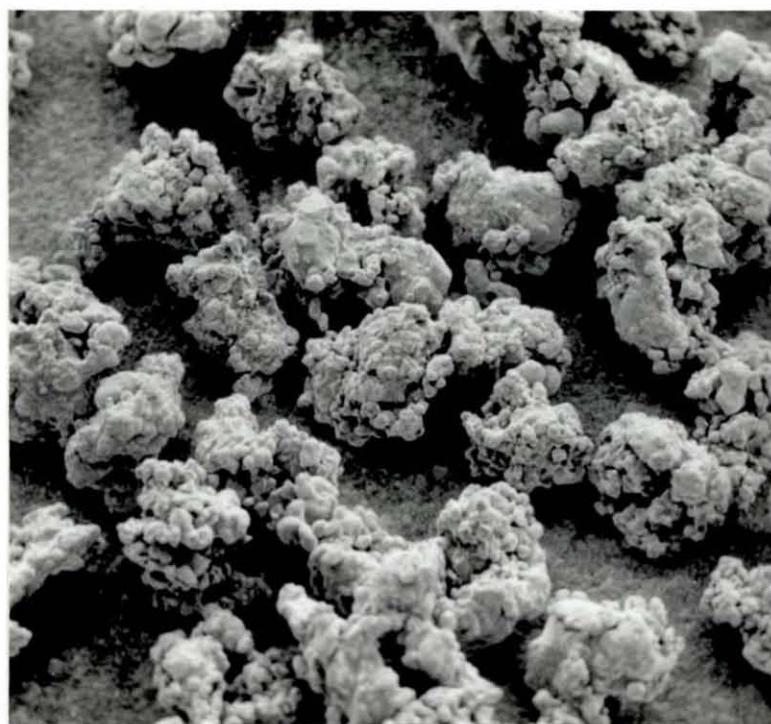
(a)
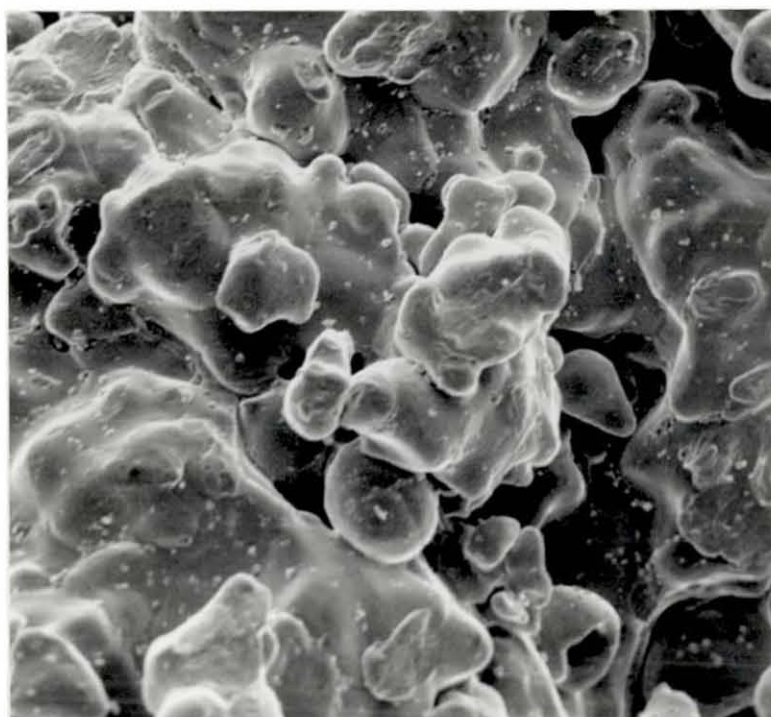
(b)

FIGURE 41

SEM Photograph of Mannesman WPL 200 Iron Powder Coated
with Copper by Displacement Method in Bath

a) Fe-1.5 Cu wt% (X100)

b) Fe-1.5 Cu wt% (X500)

c) Fe-1.5 Cu wt% (X1000)

(a)
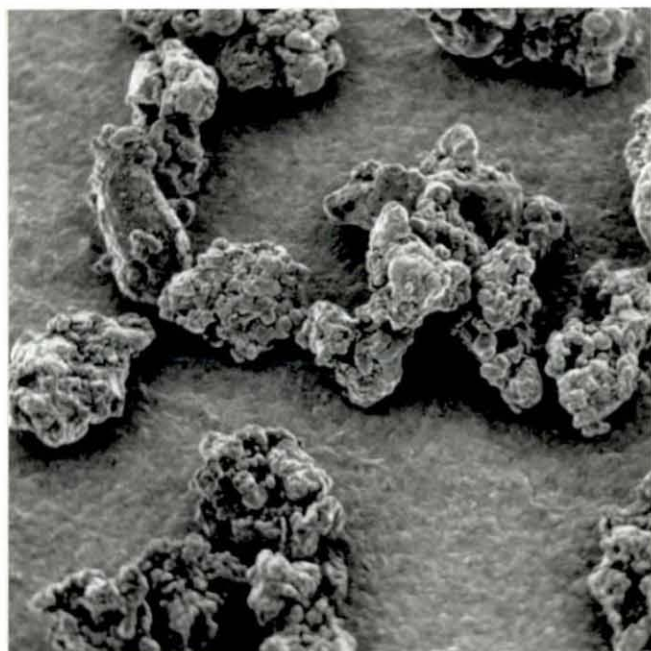
(b)
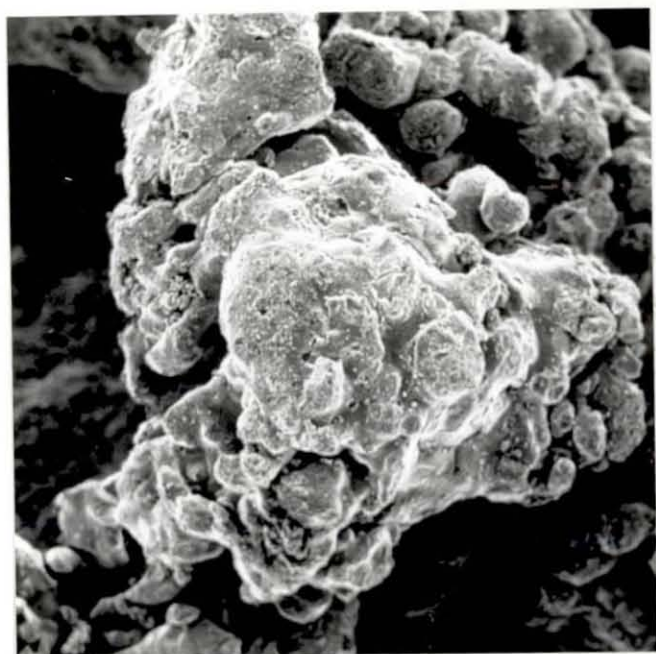
(c)

FIGURE 42

SEM photographs of Mannesman WPL 200 iron powder coated
with copper by displacement method in fluidised bed

a)   Fe - 7.1 Cu wt%   (X100)
b)   Fe - 7.1 Cu wt%   (X500)
c)   Fe - 7.1 Cu wt% (X10000)

(a)
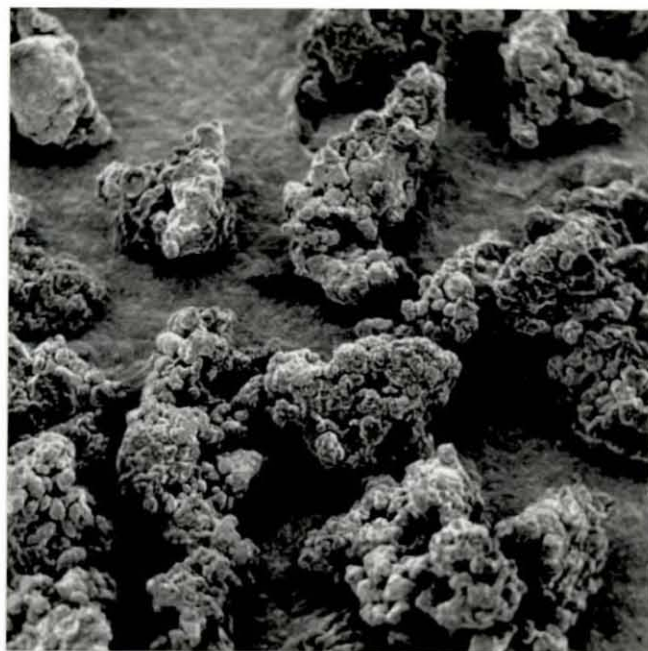
(b)
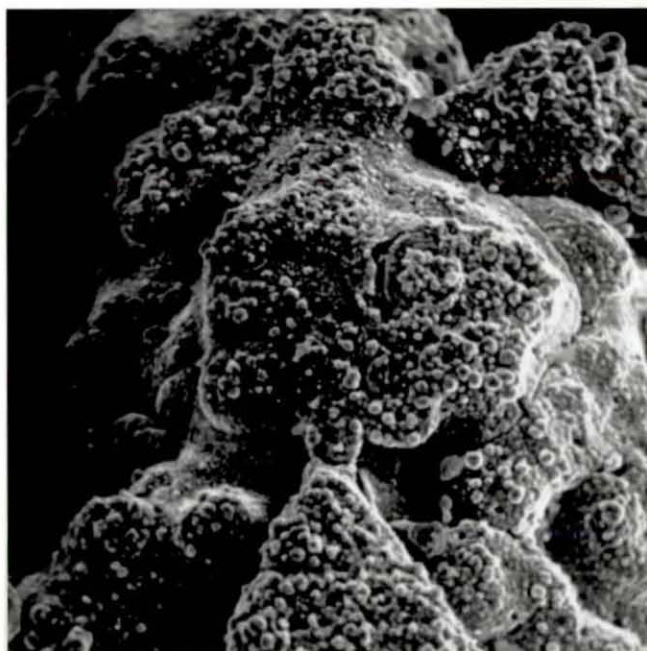
(c)
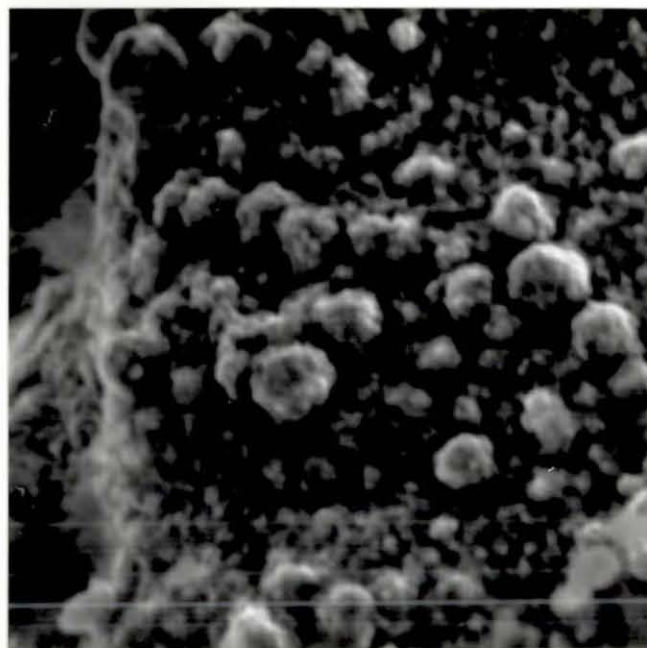
FIGURE 43

SEM photographs of Mannesman WPL 200 iron powder coated
with copper by displacement method in fluidised bed

a)   Fe - 8.6 Cu wt%   (X100)
b)   Fe - 8.6 Cu wt%   (X500)
c)   Fe - 8.6 Cu wt% (X10000)

(a)
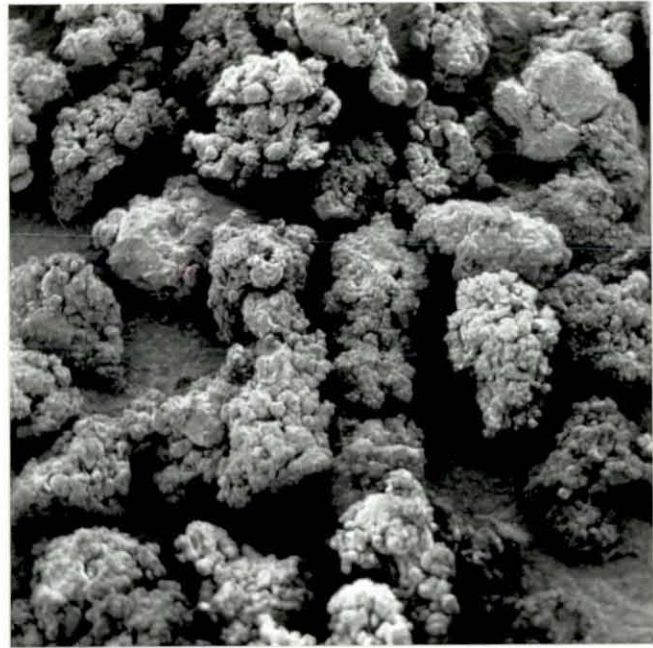
(b)
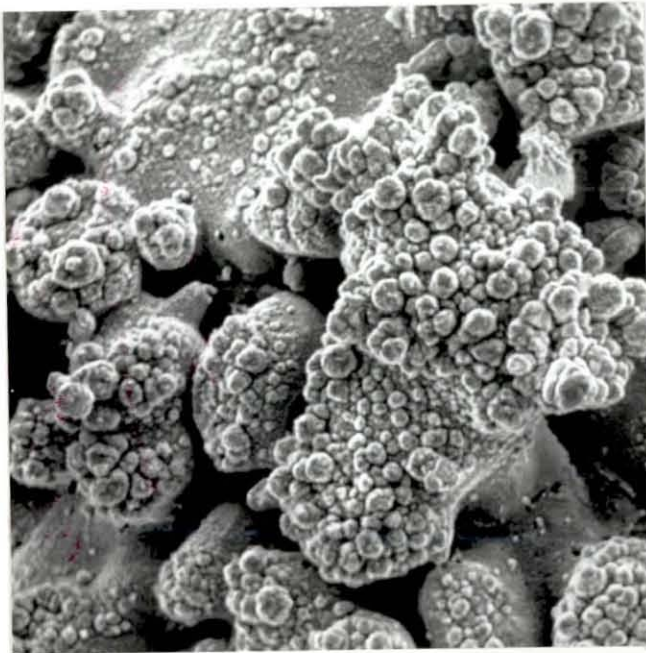
(c)
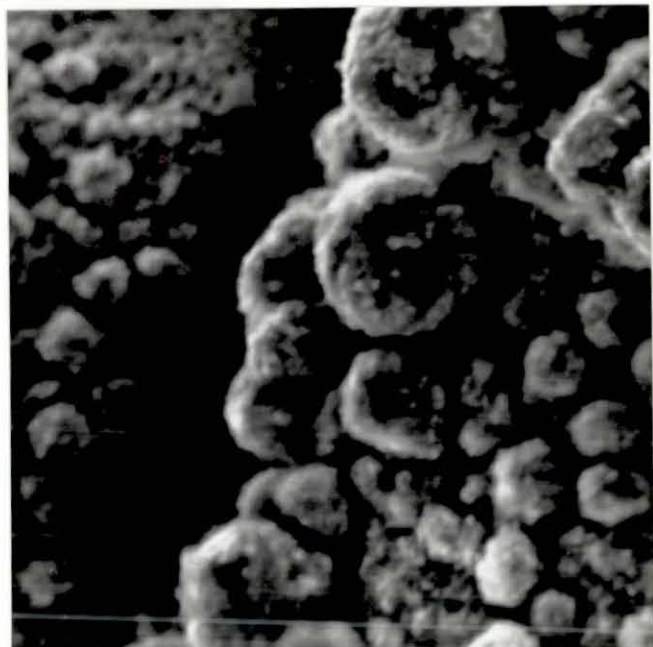
FIGURE 44

SEM photographs of Mannesman WPL 200 iron powder coated with copper by displacement method in fluidised bed

a)   Fe - 9.8 Cu wt% (X100)
b)   Fe - 9.8 Cu wt% (X500)
c)   Fe - 9.8 Cu wt% (X10000)

(a)
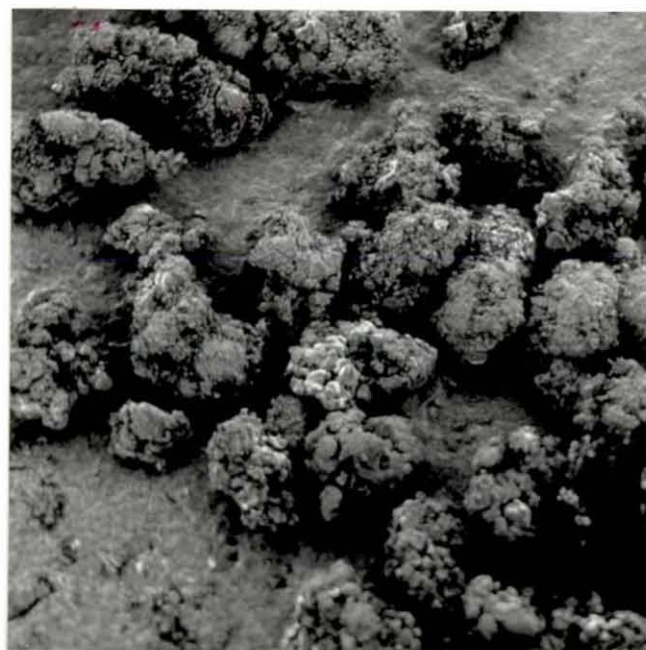
(b)

(c)
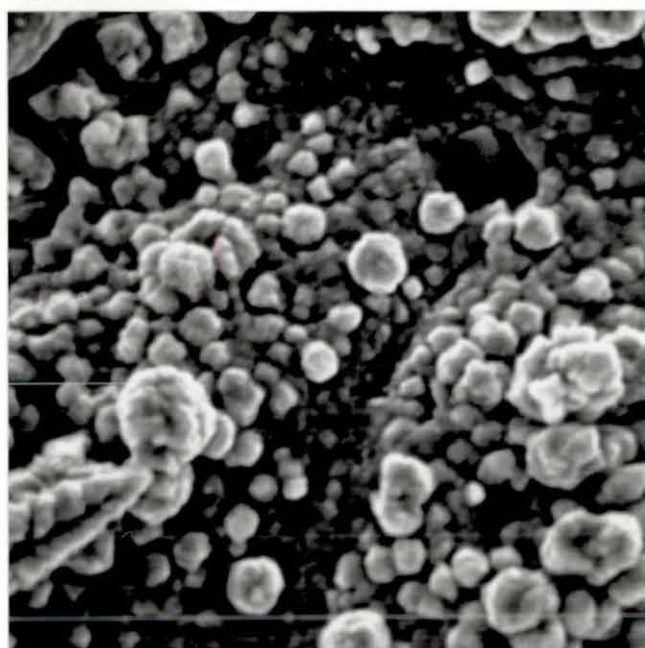
FIGURE 45

SEM photographs of Mannesman WPL 200 iron powder coated
with copper-tin alloy by displacement method in bath

a) Fe - 0.7 Cu - 0.2 Sn wt%   (X100)
b) Fe - 0.7 Cu - 0.2 Sn wt% (X10000)

(a)
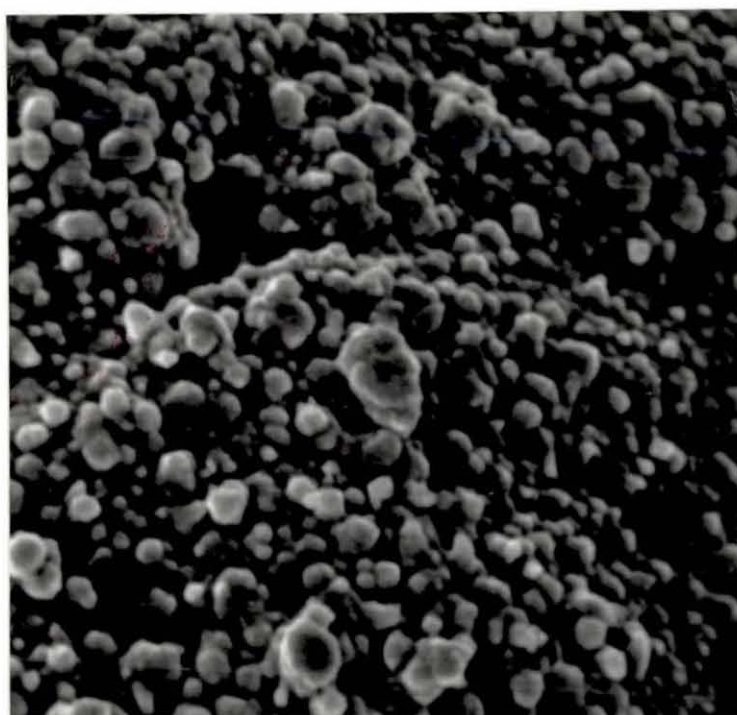
(b)

FIGURE 46

SEM photographs of Mannesman WPL 200 iron powder coated
with copper-tin alloy by displacement method in bath

a)   Fe - 3.5 Cu - 1.3 Sn wt%   (X100)
b)   Fe - 3.5 Cu - 1.3 Sn wt%   (X500)
c)   Fe - 3.5 Cu - 1.3 Sn wt% (X10000)

(a)
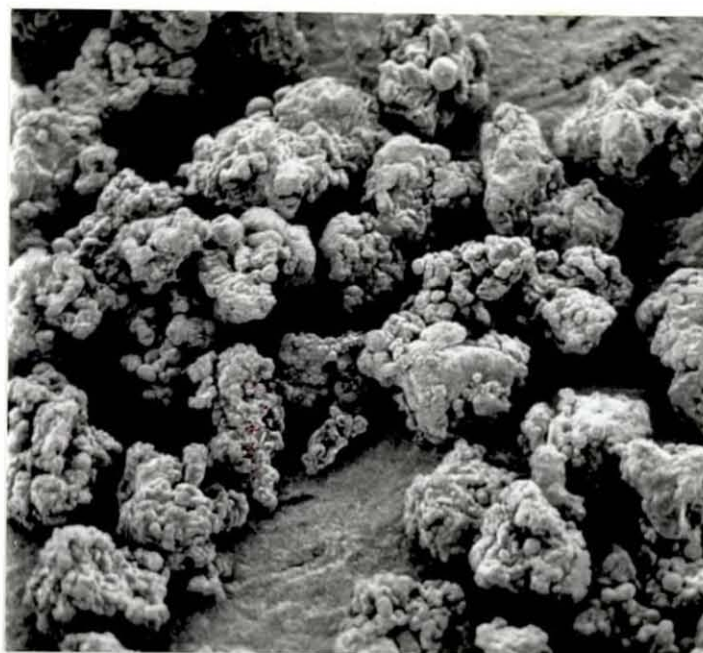
(b)
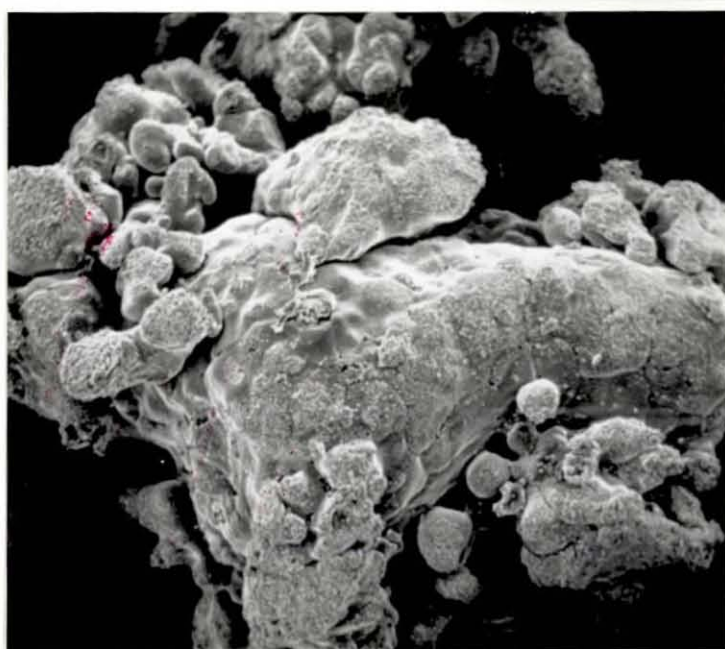
(c)
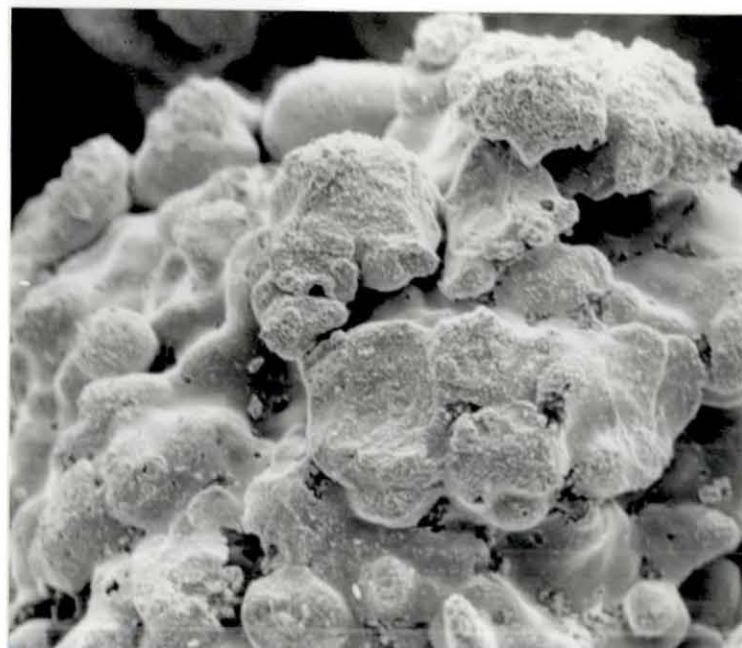
FIGURE 47

SEM photographs of Mannesman WPL 200 iron powder coated
with copper-tin alloy by displacement method in bath

a)   Fe - 4.3 Cu - 2.1 Sn wt%   (X100)
b)   Fe - 4.3 Cu - 2.1 Sn wt%   (X500)
c)   Fe - 4.3 Cu - 2.1 Sn wt% (X10000)

(a)
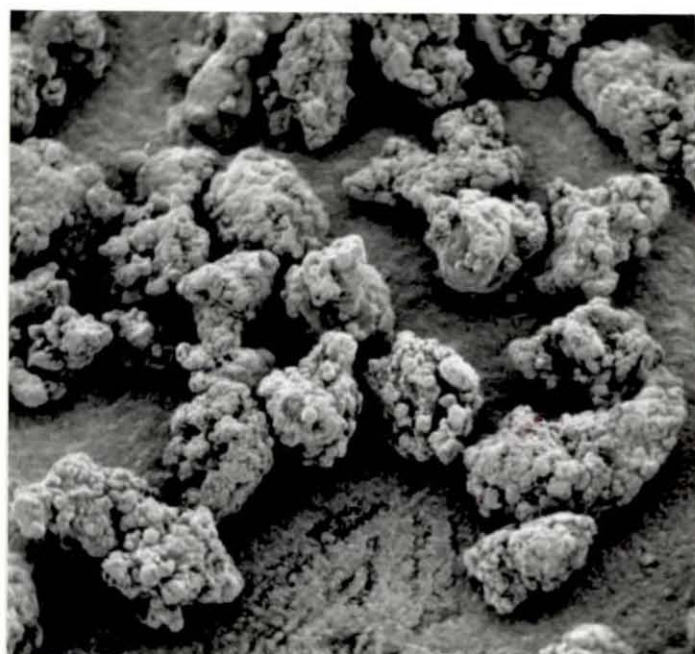
(b)
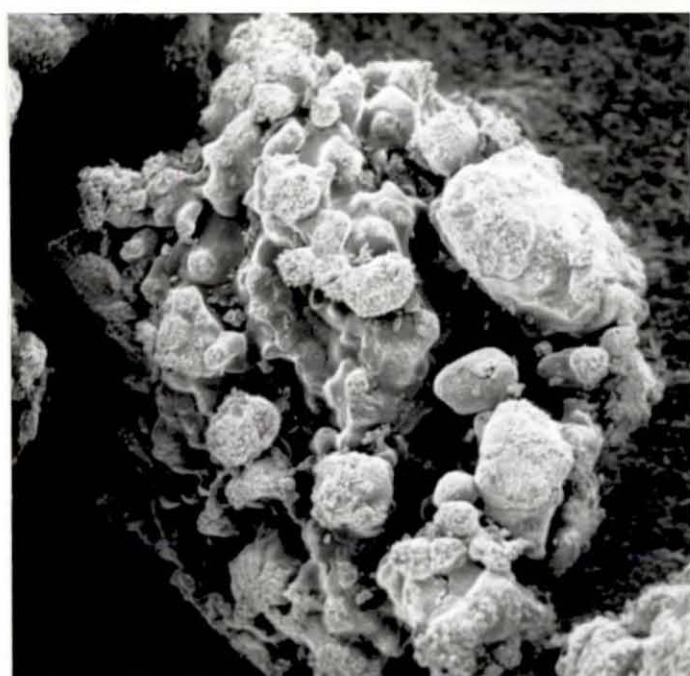
(c)
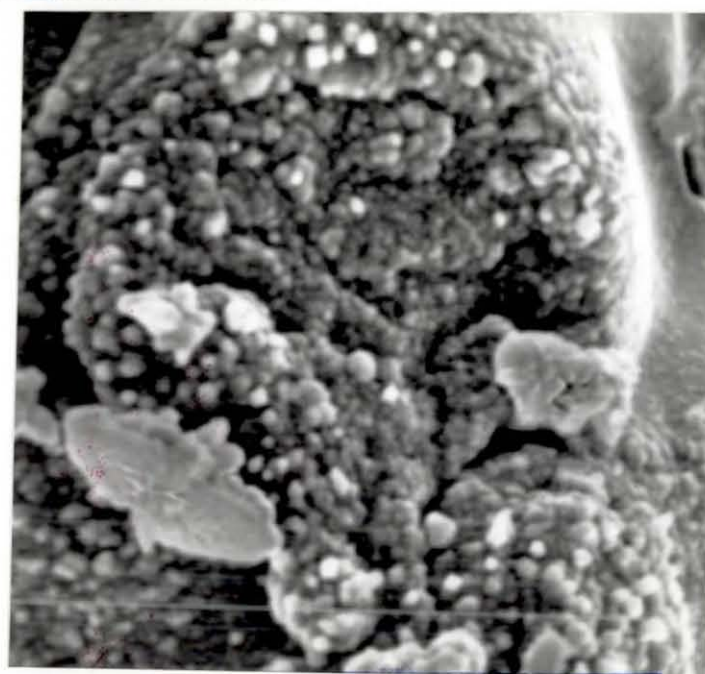
FIGURE 48

Optical micrograph of a pressed compact made from iron
powder coated with 9.8 wt% Cu by displacement technique
in fluidised bed system (X100)
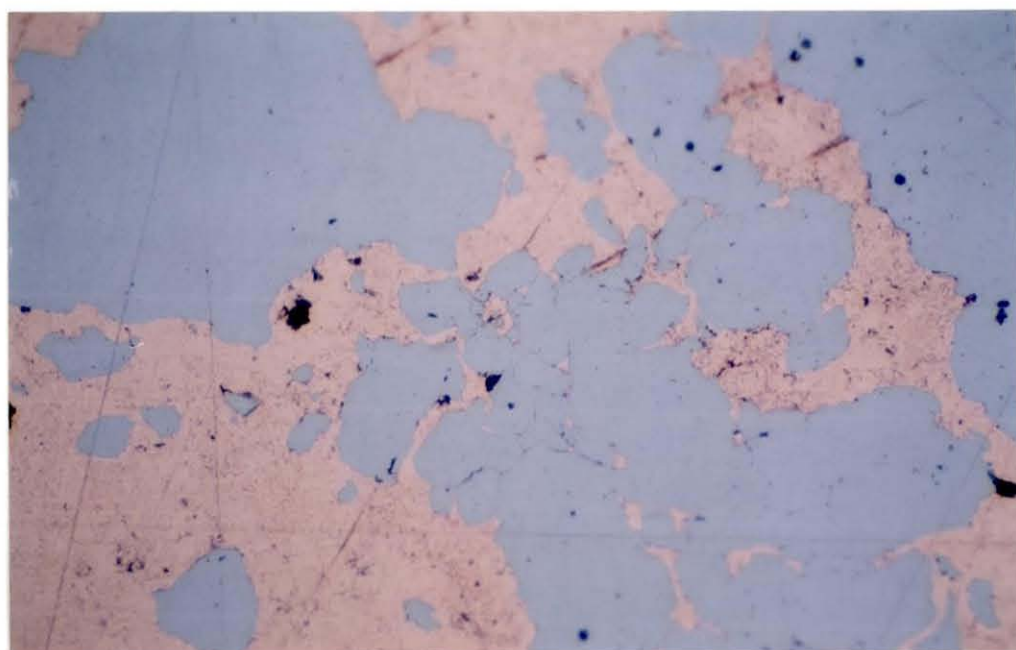
FIGURE 49

Optical micrograph of an Fe-0.7 Cu-0.2 Sn wt% pressed
compact made from iron powder coated with Cu-Sn alloy
by displacement technique in bath
a)   X50
b)   X100
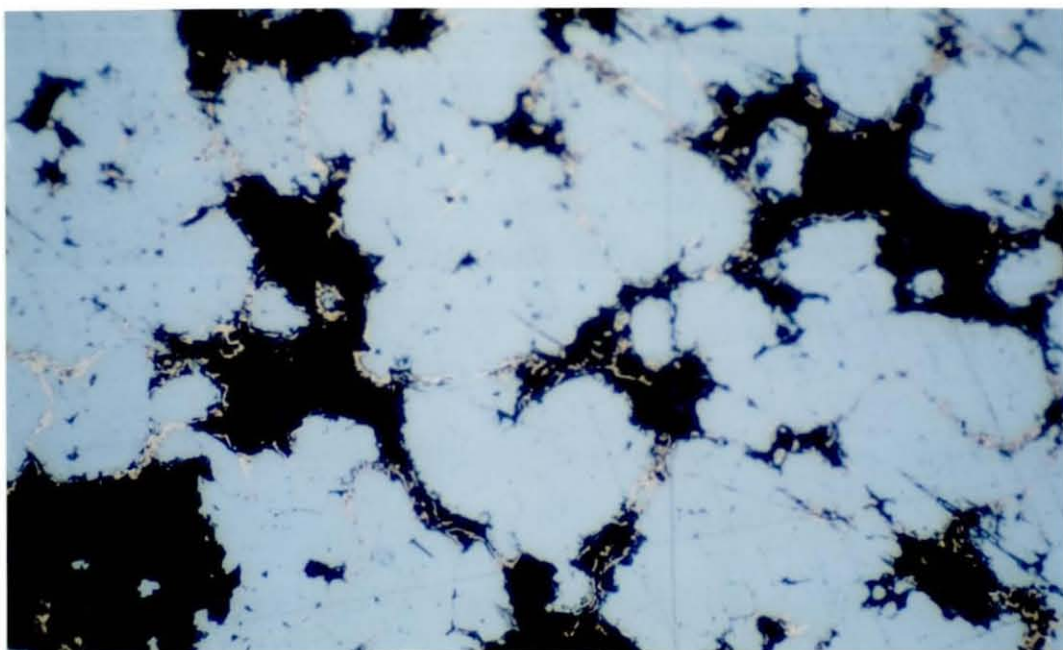
(a)
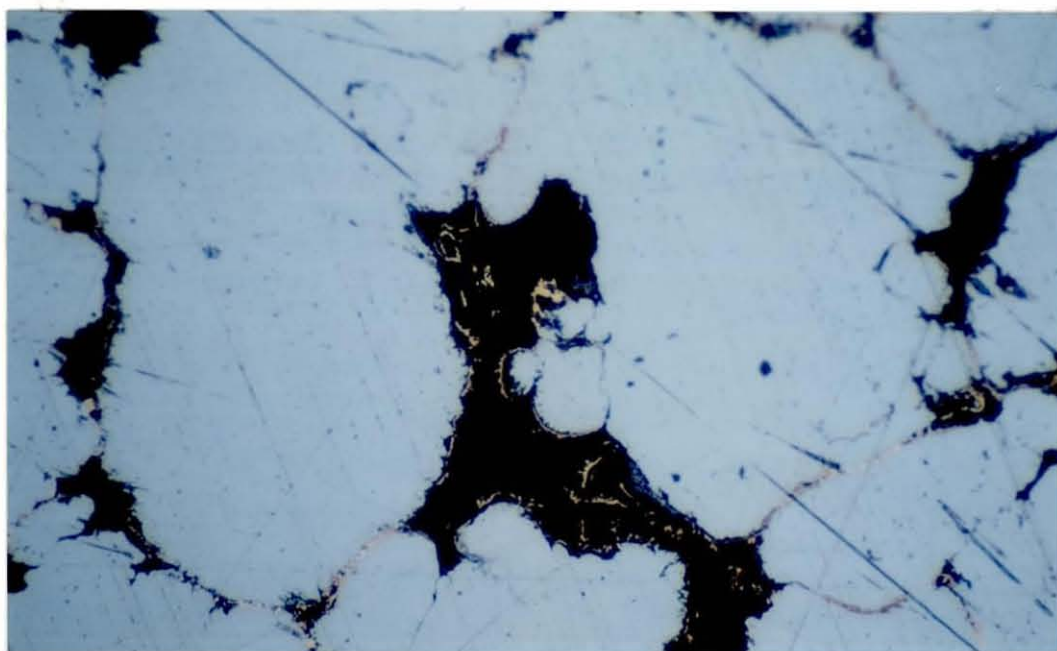
(b)

FIGURE 50

Failure surface of unsintered iron powder compact
tested for the indirect (diametrical) tensile strength
(X2000), ρ = 7.09 g/cm^3

FIGURE 51

Failure surfaces of unsintered iron based compacts
tested for the indirect (diametrical) tensile strength,
plated by displacement method (X2000)

    a) Fe - 1.5 Cu wt%, ρ = 7.13 g/cm^3
    b) Fe - 7.1 Cu wt%, ρ = 7.13 g/cm^3
    c) Fe - 8.6 Cu wt%, ρ = 7.35 g/cm^3
    d) Fe - 9.8 Cu wt%, ρ = 7.38 g/cm^3
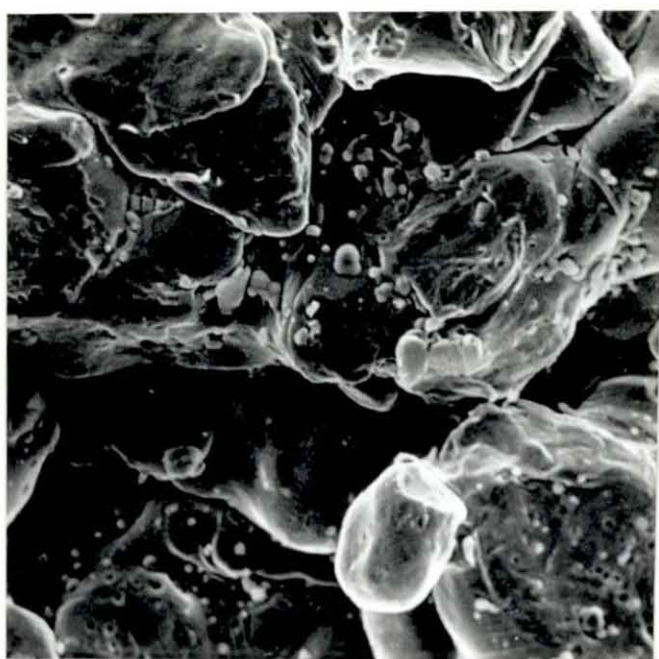
FIG 50.
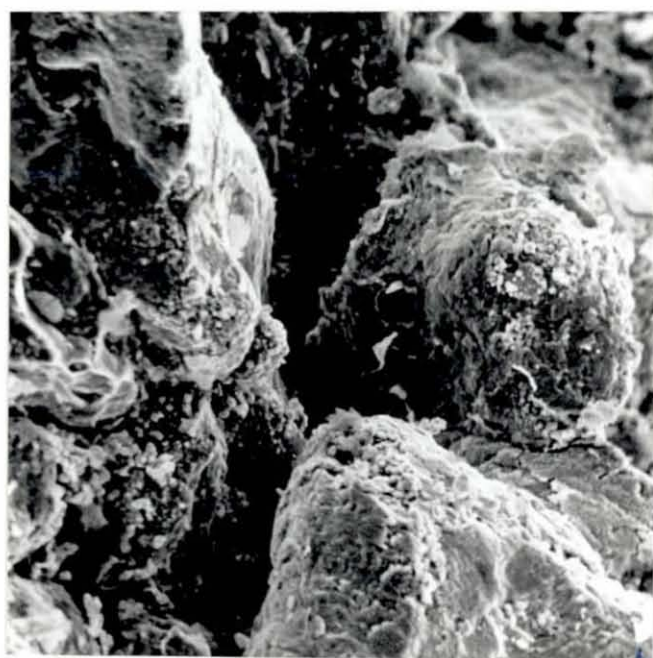
(a)

(b)
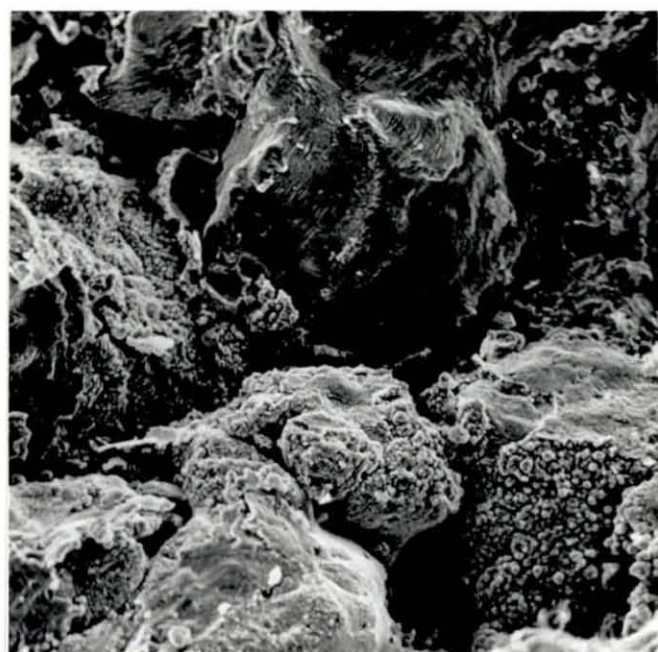
(c)
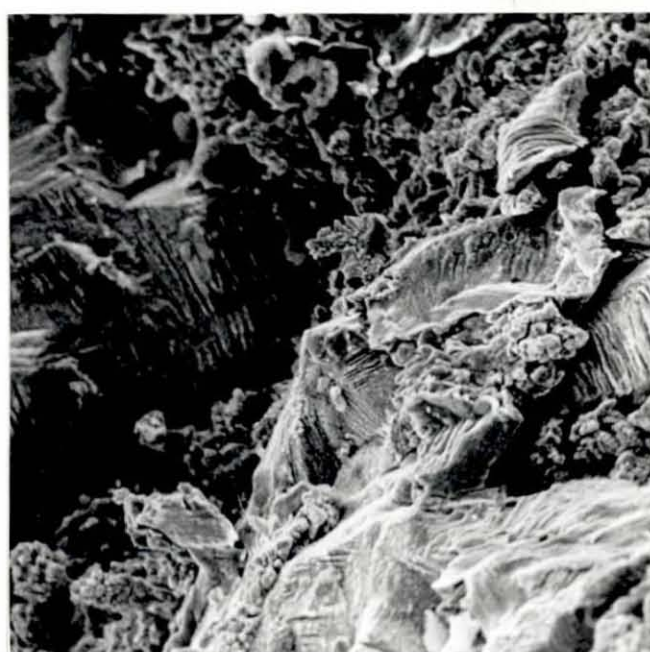
(d)
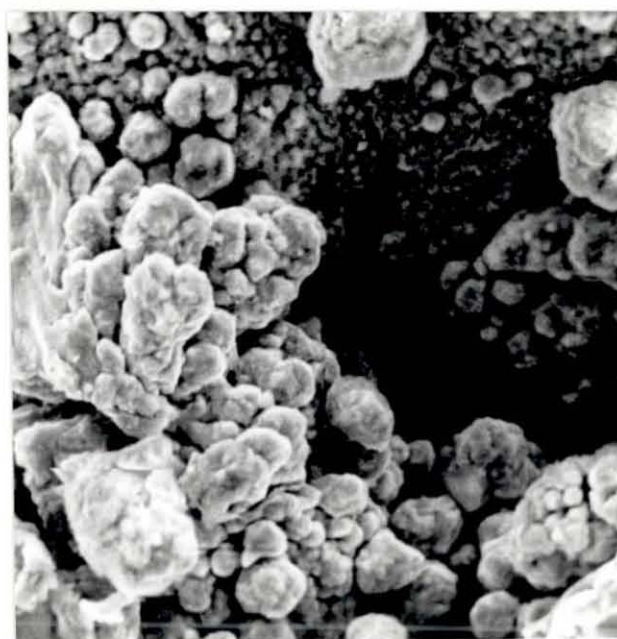
FIGURE 52

Failure surfaces of unsintered iron based compacts
tested for the indirect (diametrical) tensile strength,
plated by displacement method (X2000)

a) Fe - 0.7 Cu - 0.25 Sn wt%, ρ = 6.98 g/cm^3
b) Fe - 3.5 Cu - 1.3 Sn wt%, ρ = 7.03 g/cm^3
c) Fe - 4.3 Cu - 2.1 Sn wt%, ρ = 7.04 g/cm^3

(a)

(b)
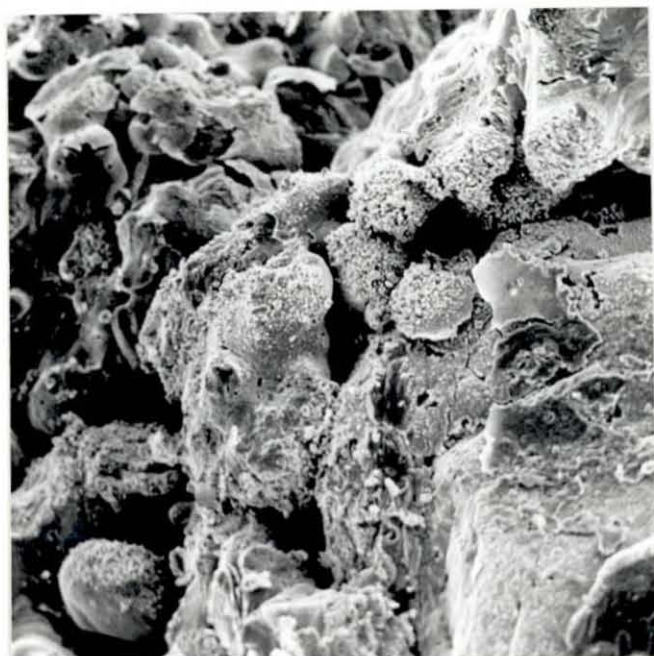
(c)

FIGURE 53

a) Optical micrograph of an Fe-9.8 wt% Cu sintered
    compact made from iron powder coated with Cu by
    displacement technique (X50).
    Sintering temperature: 800°C for 9 min

b) Optical micrograph of an Fe-0.7 wt% Cu-0.2 wt% Sn
    sintered compact made from iron powder coated with
    Cu-Sn alloy by displacement method (X50).
    Sintering temperature: 800°C for 9 min
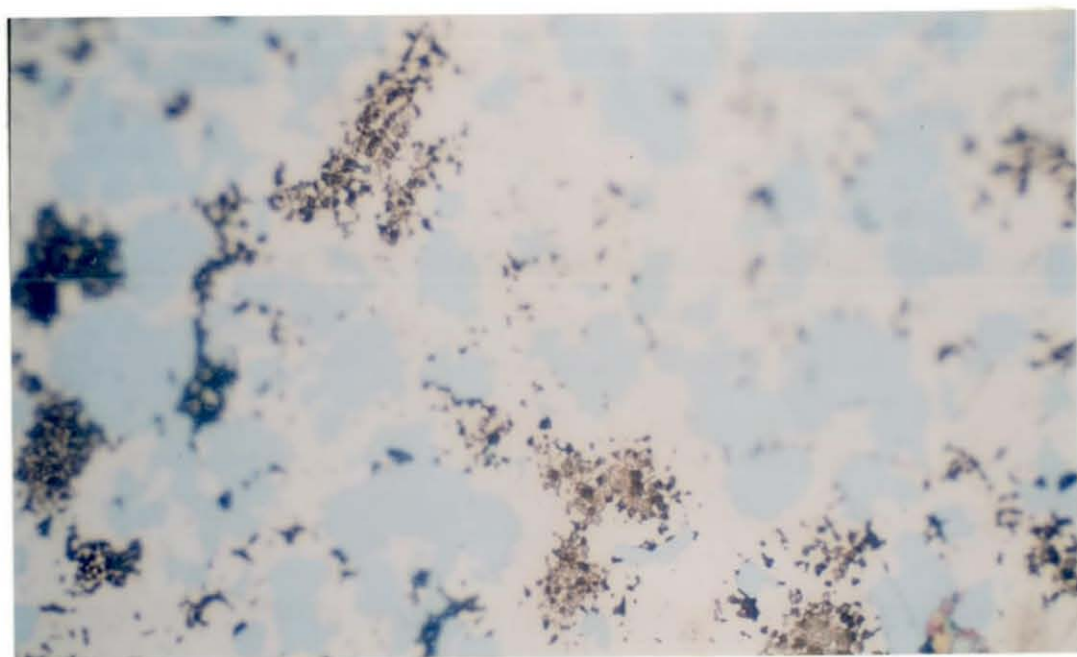
(a)

(b)

FIGURE 54

a)   Electron probe microanalysis (EPMA) trace for an Fe-9.8 Cu sintered compact made from Mannesman WPL 200 iron powder coated with copper by the displacement plating method, sintered at $800^\circ C$ for 9 mins

Note:		Background (c/s)	Standard (c/s)	Sample Count Rate (c/s)
	Fe	223.0	7283.2	10^4
	Cu	422.1	100312.3	10^3

b)   Electron probe microanalysis (EPMA) trace for an Fe-4.3 Cu - 2.1 Sn sintered compact made from Mannesman WPL 200 iron powder coated with Cu-Sn by the displacement plating method, sintered at 800° for 9 mins

Note:		Background (c/s)	Standard (c/s)	Sample Count Rate (c/s)
	Fe	241.2	9224.2	10^4
	Cu	419.2	102310.3	10^4

c)   Electron probe microanalysis (EPMA) trace for an Fe-4.3 Cu-2.1 Sn sintered compact made from Mannesman WPL 200 iron powder coated with Cu-Sn by the displacement plating method, sintered at $800^\circ C$ for 9 mins

Note:		Background (c/s)	Standard (c/s)	Sample Count Rate (c/s)
	Fe	234.5	8234.9	10^4
	Sn	74.4	6531.1	3×10^2

Fig. 54 (a)

for H 25-1

Fe

Cu

Fig. 54 (b)

Cu

Fe

Fig. 54 (c)

Fe

Sn
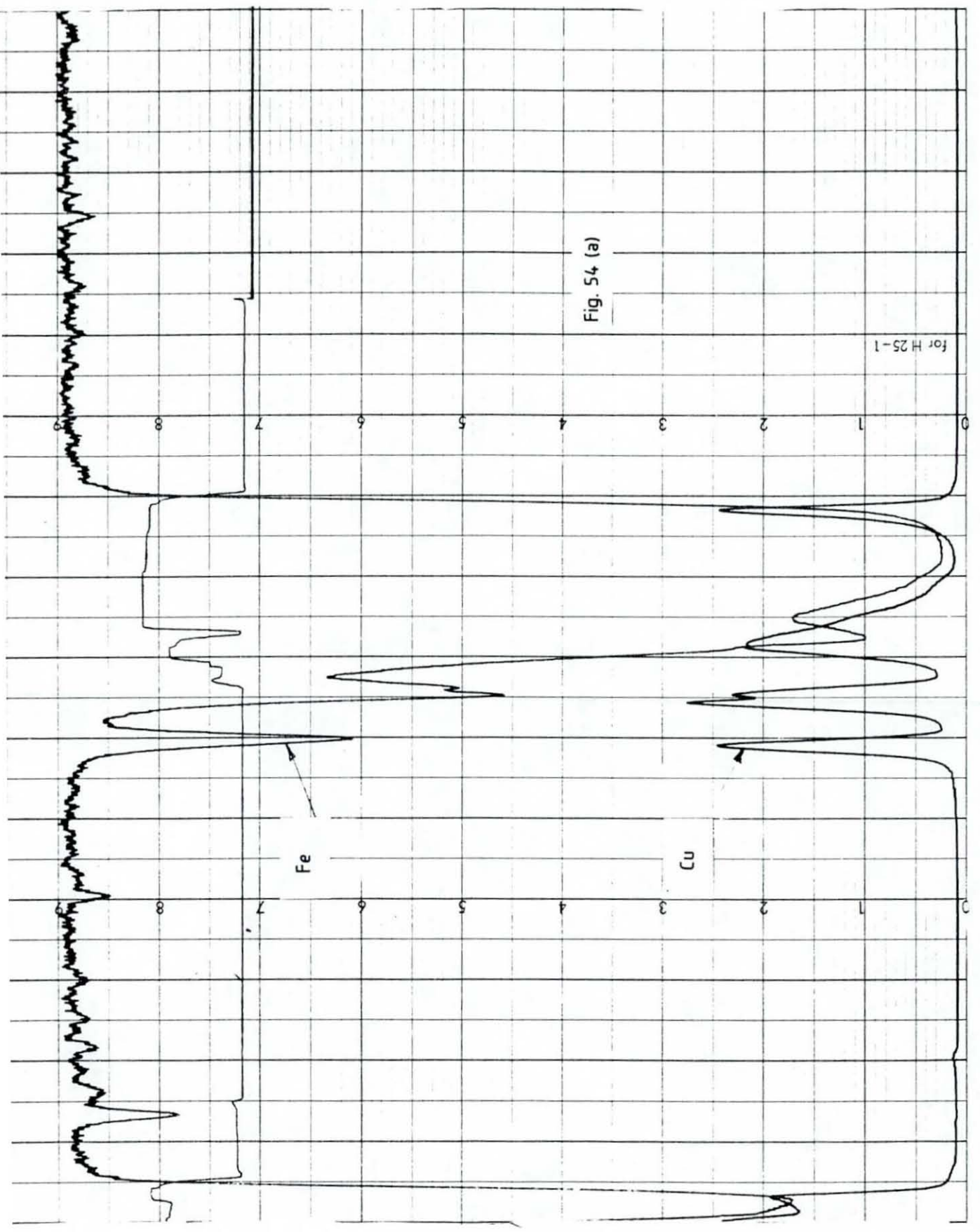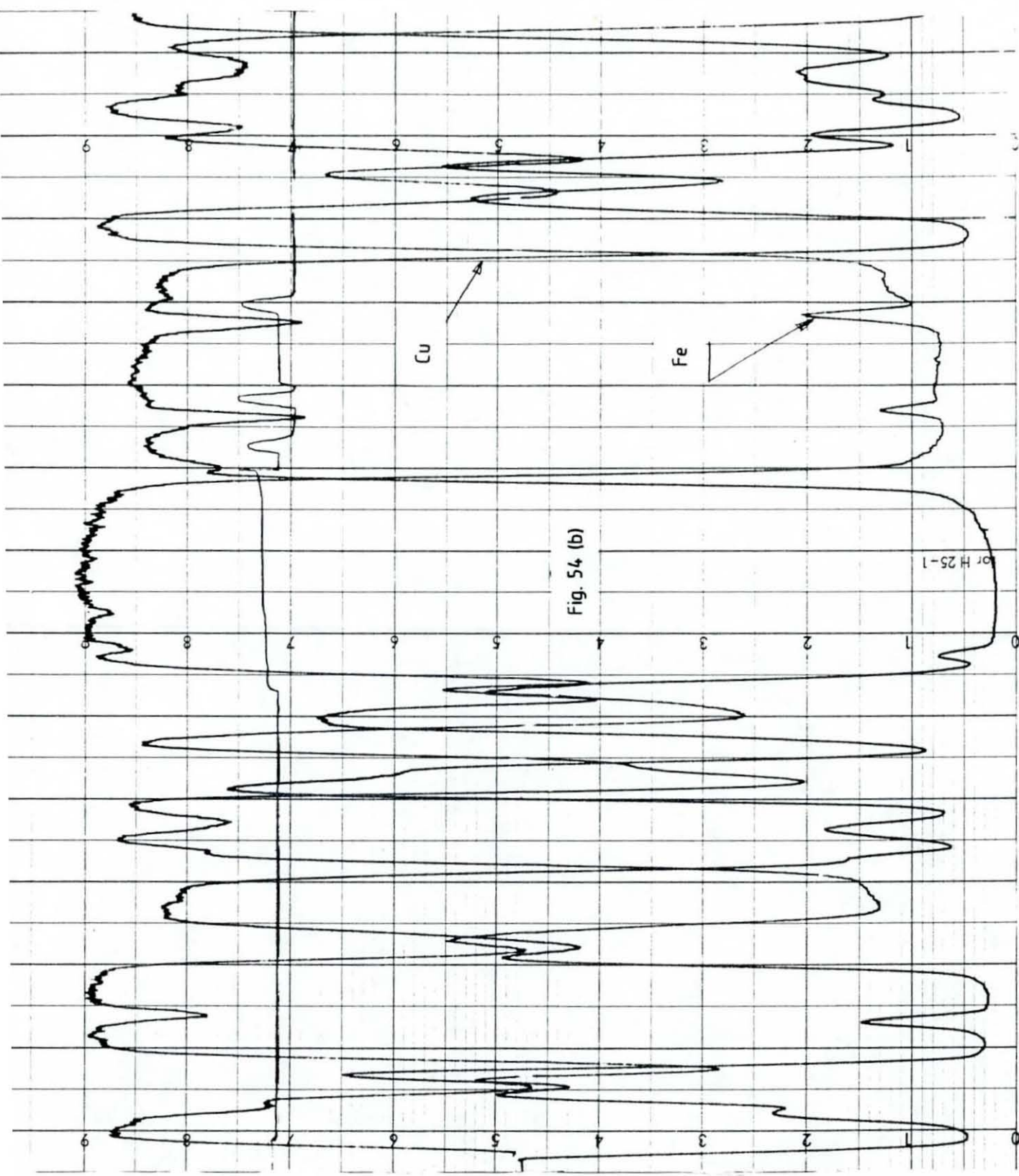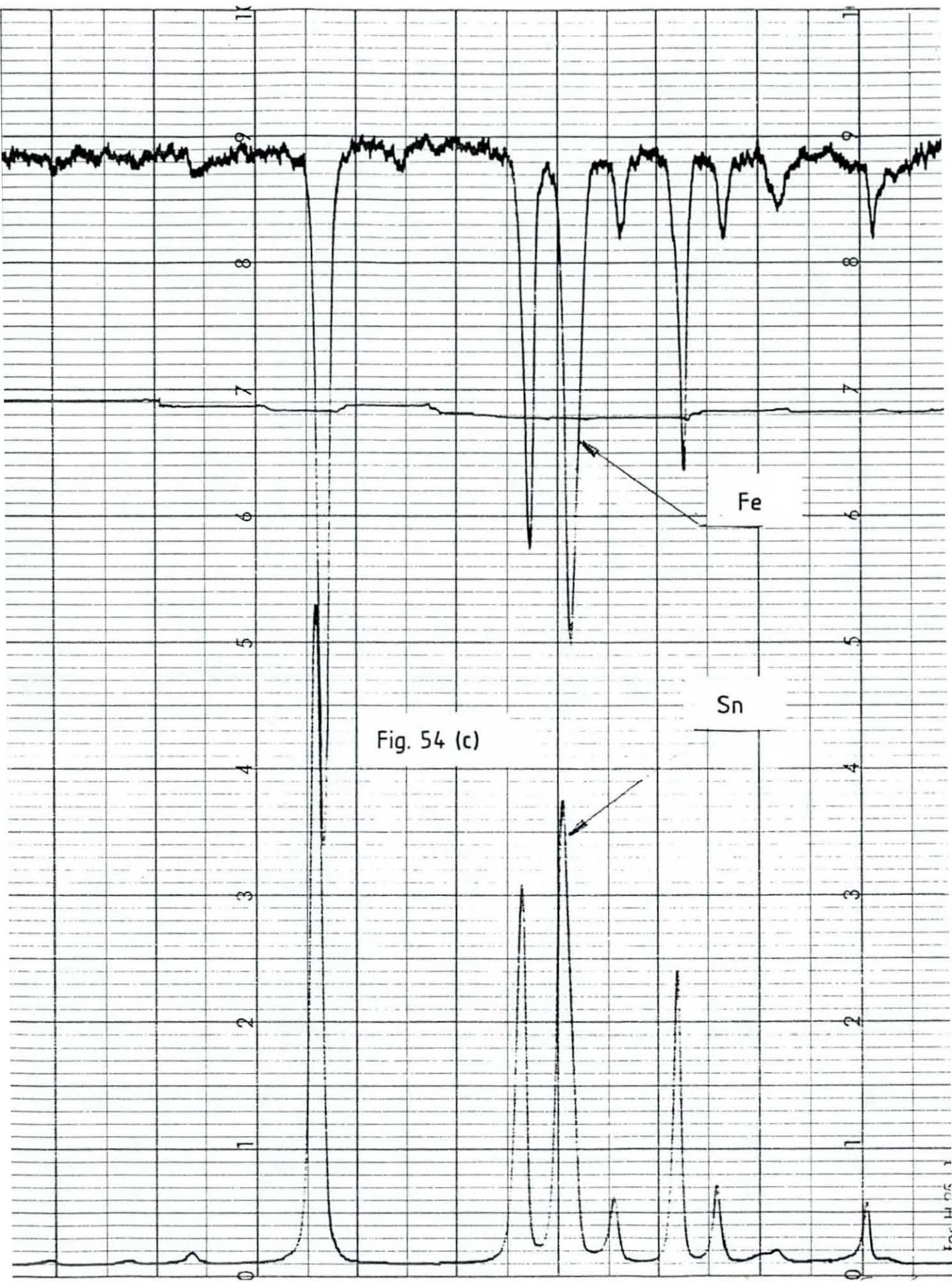
FIGURE 55

Photograph of Al_2O_3 powder (45 μm), coated by
electroless method

a) with 11.7 Cu wt% (X25)
b) with 6.34 Ni wt% (X25)

(a)
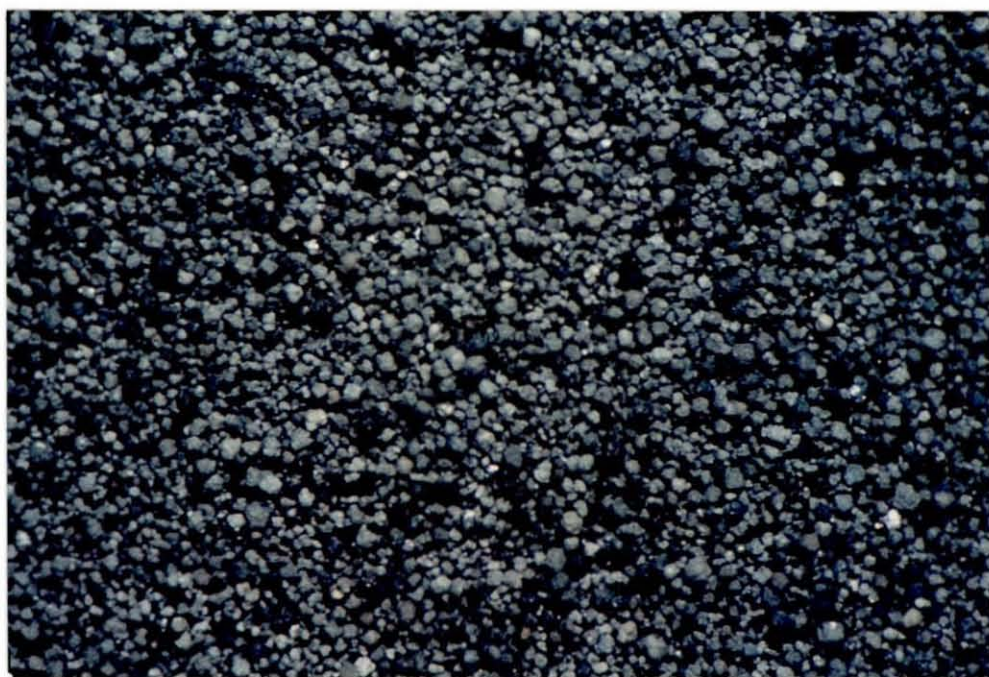
(b)

FIGURE 56

Photograph of SiC powder (250 μm) coated with 3.5 wt%
Ni by electroless method (X10)

FIGURE 57

Photograph of WC powder (45-150 μm) coated by
electroless method

    a)   WC - 12.72 wt% Co (X10)
    b)   WC - 8.74 wt% Cu (X10)
    c)   WC - 5.71 wt% Ni (X10)

fig 56.

fig 57 :   (a)

(b)
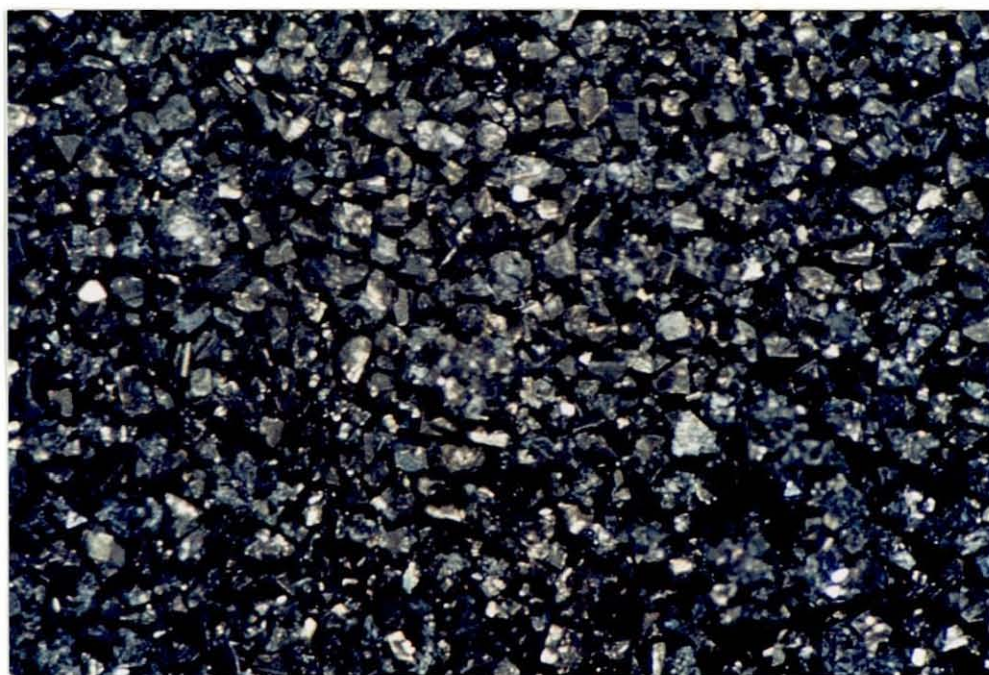
(c)

FIGURE 58

Photographs of SiC powder (29 μm) coated by electroless method

a) SiC - 62.4 wt% Cu (X10)
b) SiC - 8.33 wt% Ni (X5)

(a)
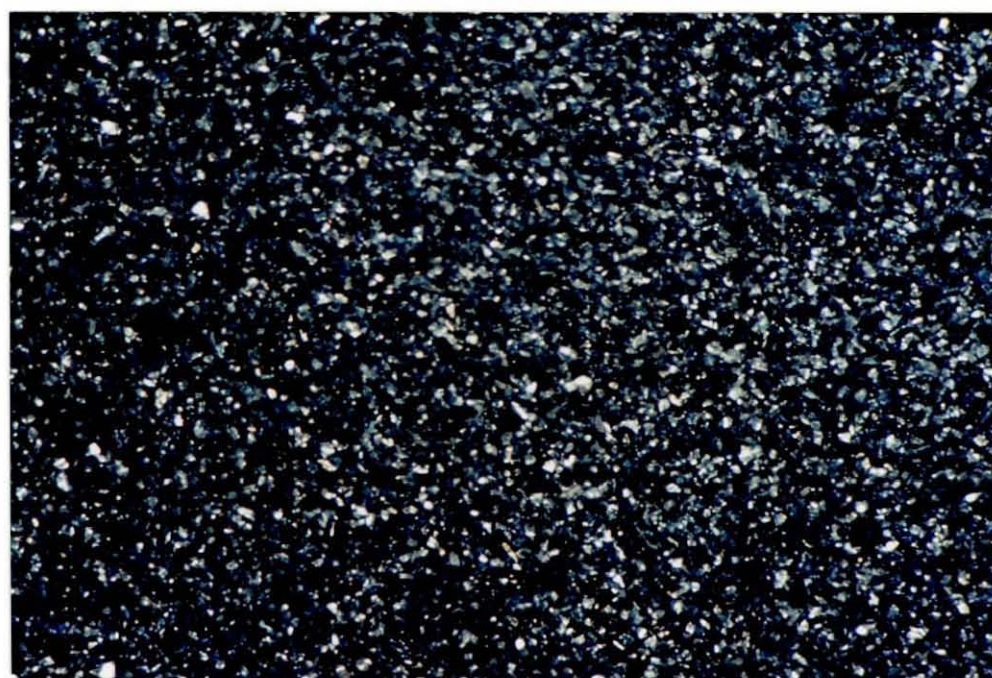
(b)

FIGURE 59

Photographs of TaC (45 μm) powder coated by electroless
method

a)   TaC - 12.8 wt% Cu (X35)
b)   TaC - 4.8 wt% Ni (X35)

(a)

(b)

FIGURE 60

Photographs of polished and sectioned surfaces of
alumina (45 μm) particles coated with copper (11.7 wt%)
by electroless method

a) X50
b) X100
c) X100

(a)
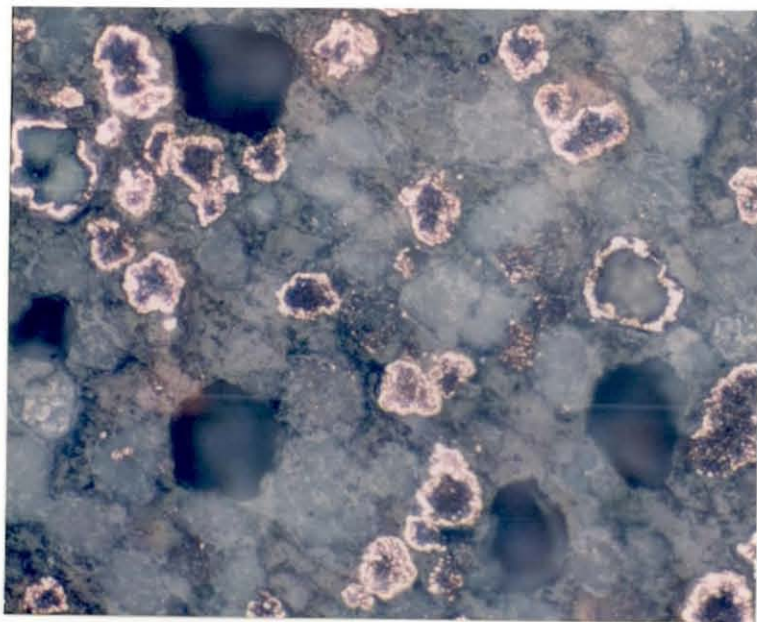
(b)
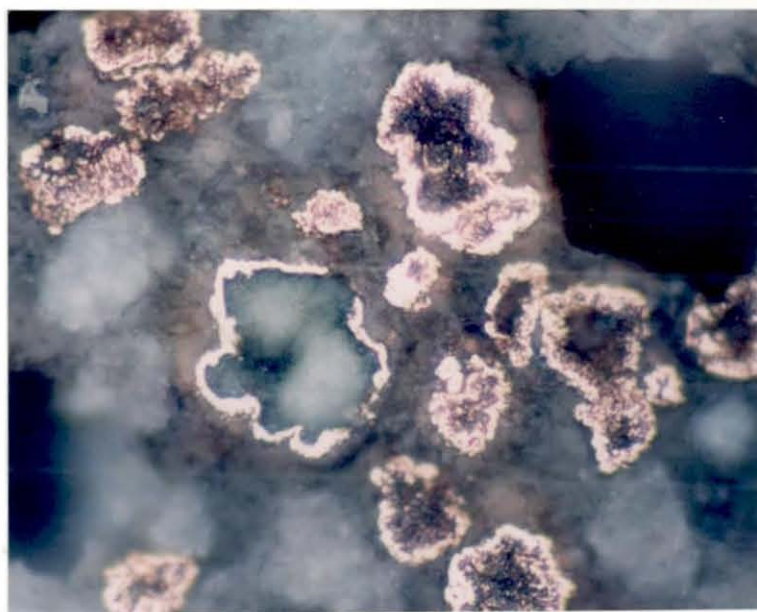
(c)
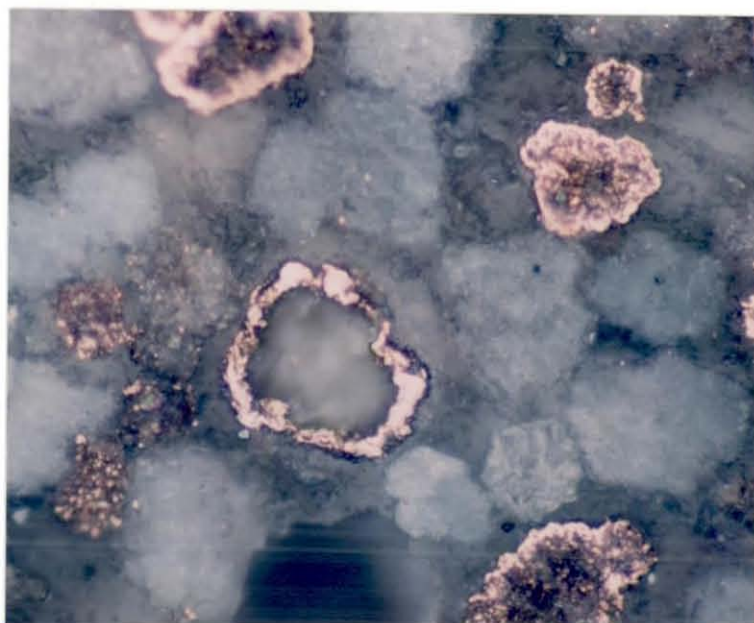
FIGURE 61

Photograph of polished and sectioned surface of WC (45-
150 μm) particles coated with 8.74 wt% Cu by
electroless method (X50)
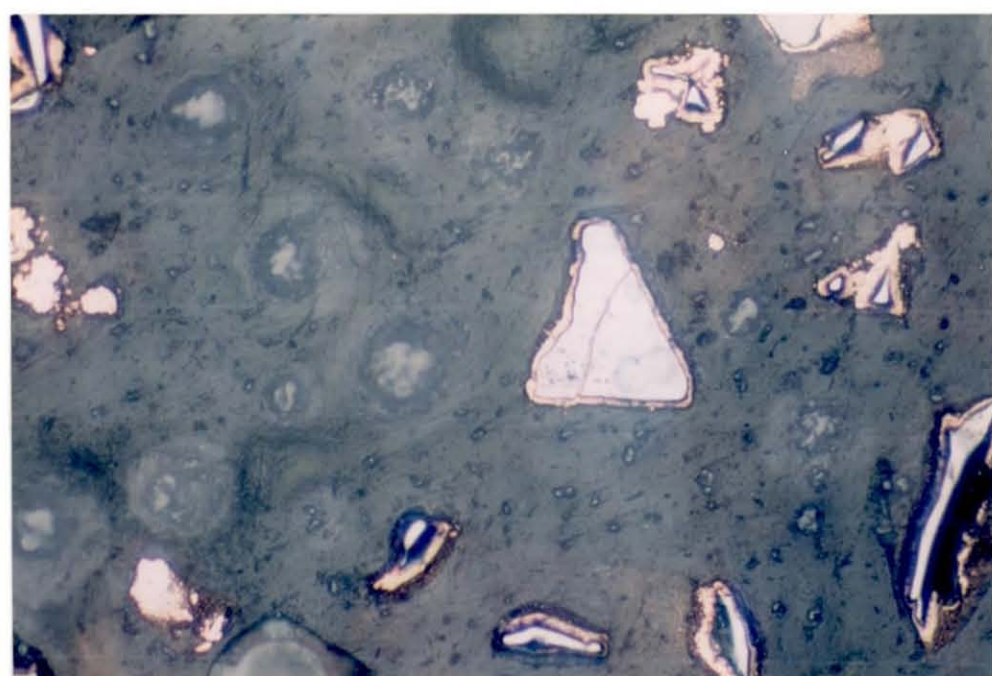
FIGURE 62

Photographs of polished and sectioned surfaces of SiC
particles coated with Cu by electroless method

a)   SiC   (29 μm) - 35.0 wt% Cu (X100)
b)   SiC   (29 μm) - 62.4 wt% Cu (X100)
c)   SiC (250 μm) - 14.3 wt% Cu   (X50)

(a)
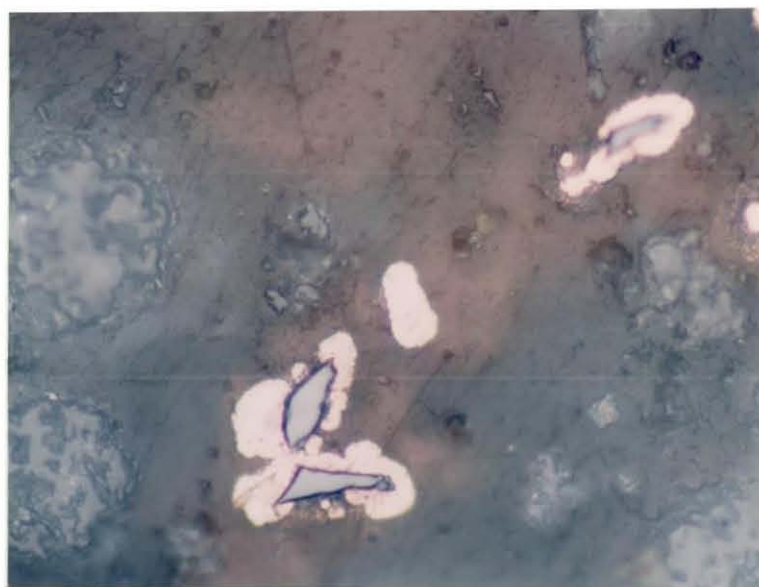
(b)

(c)
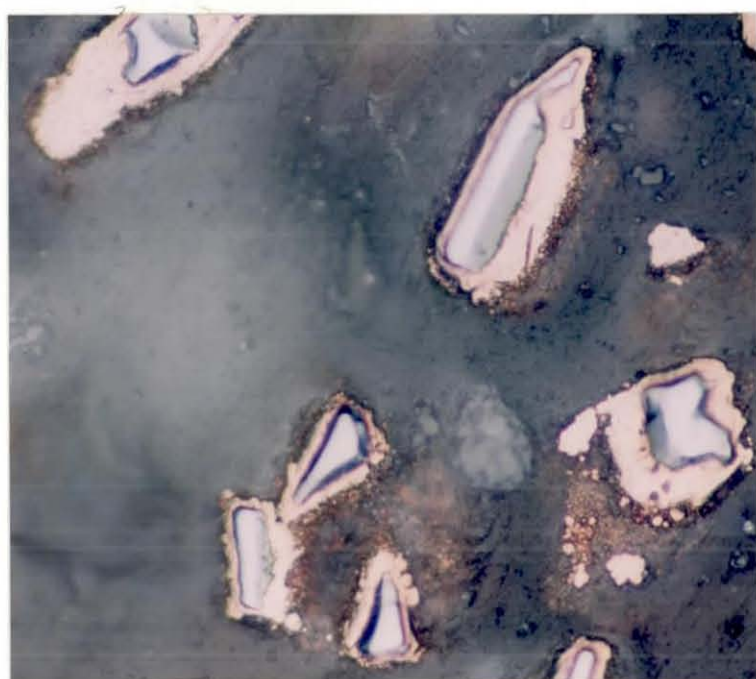
FIGURE 63

Photograph of TaC particles coated with 12.8 wt% Cu
slightly polished (X80)
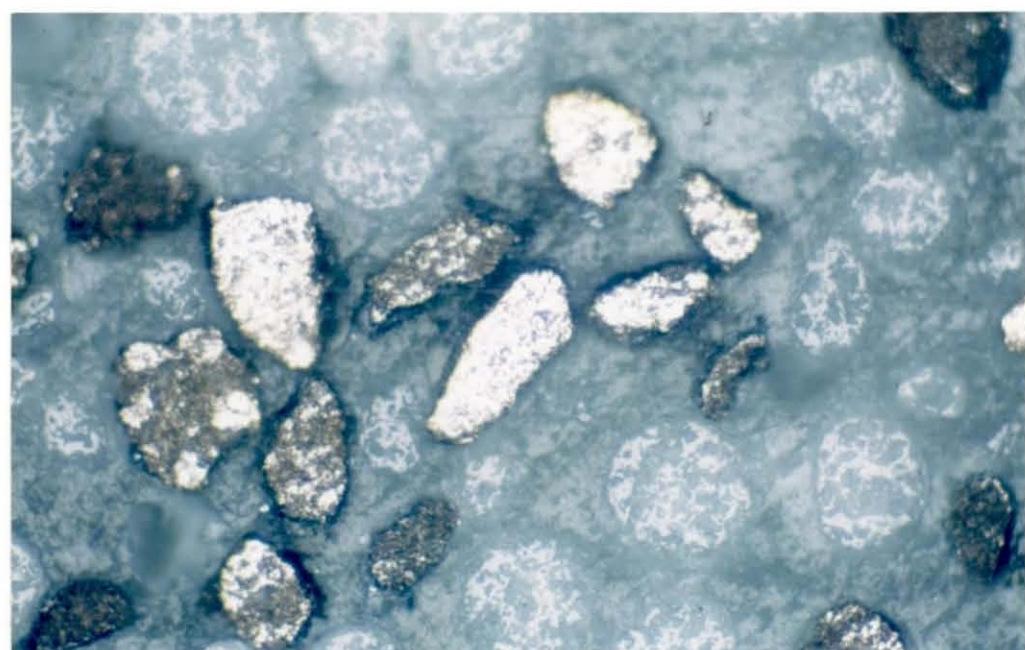
FIGURE 64

Photograph of polished and sectioned surface of Al_2O_3 (45 µm) particles coated with 6.34 wt% Ni, by electroless method

a) X80
b) X100
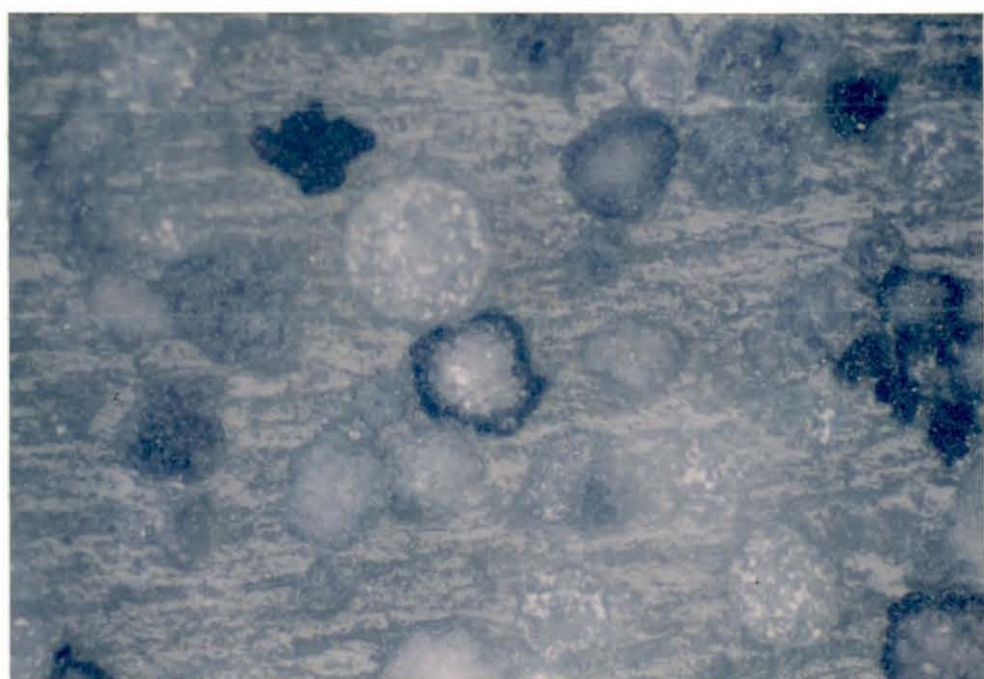
(a)
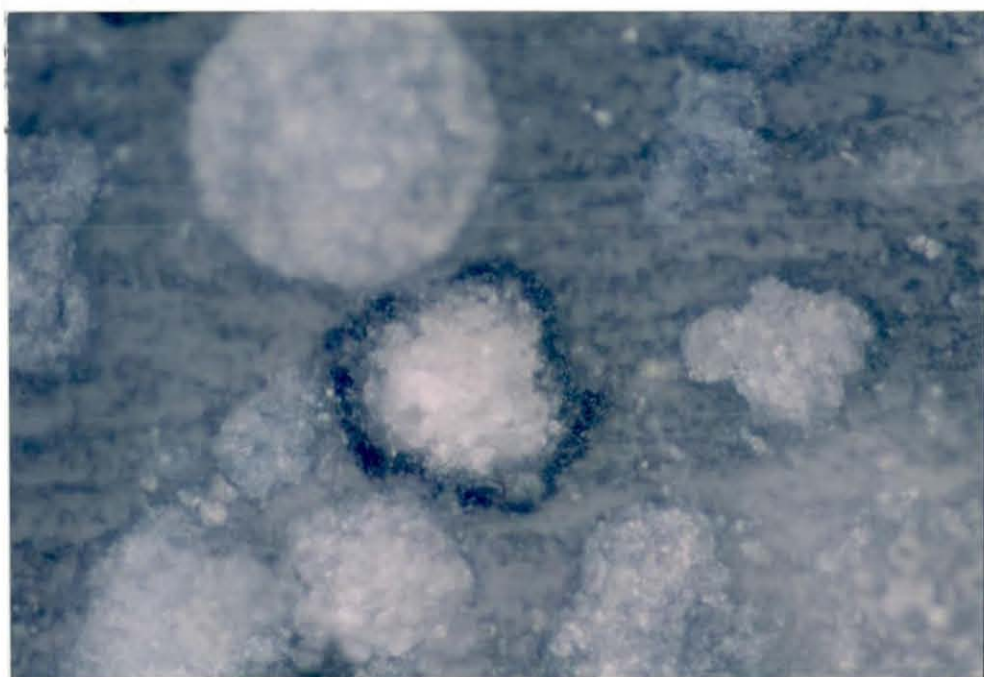
(b)

FIGURE 65

Photographs of polished and sectioned surfaces of SiC
particles coated with Ni by electroless method

a) SiC (250 µm) - 3.5 wt% Ni (X50)
b) SiC (400 µm) - 7.4 wt% Ni (X40)
c) SiC (400 µm) - 7.4 wt% Ni (X100)
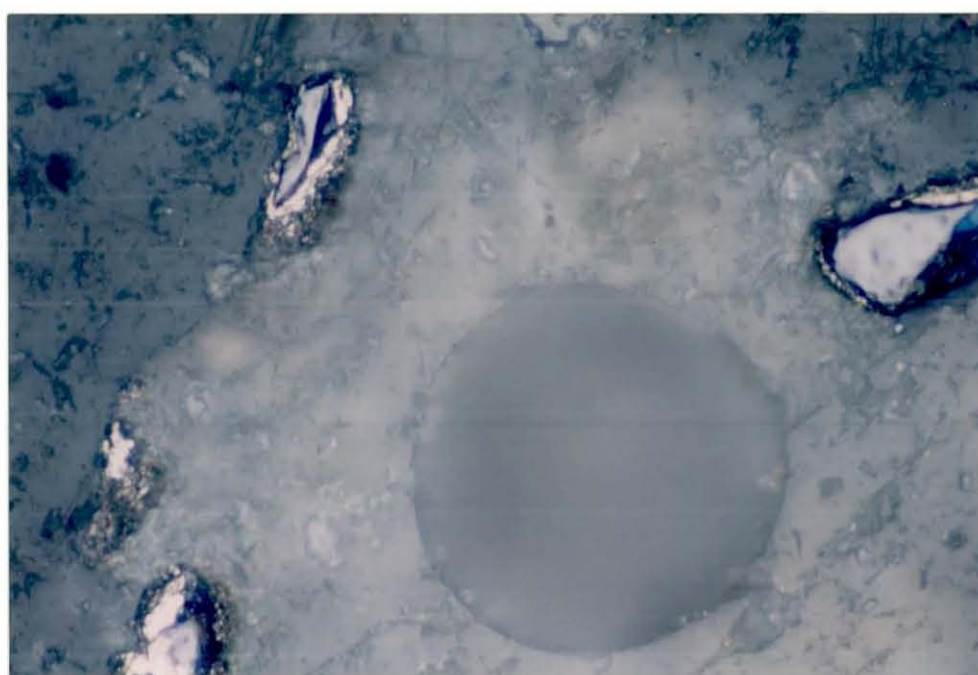
(a)
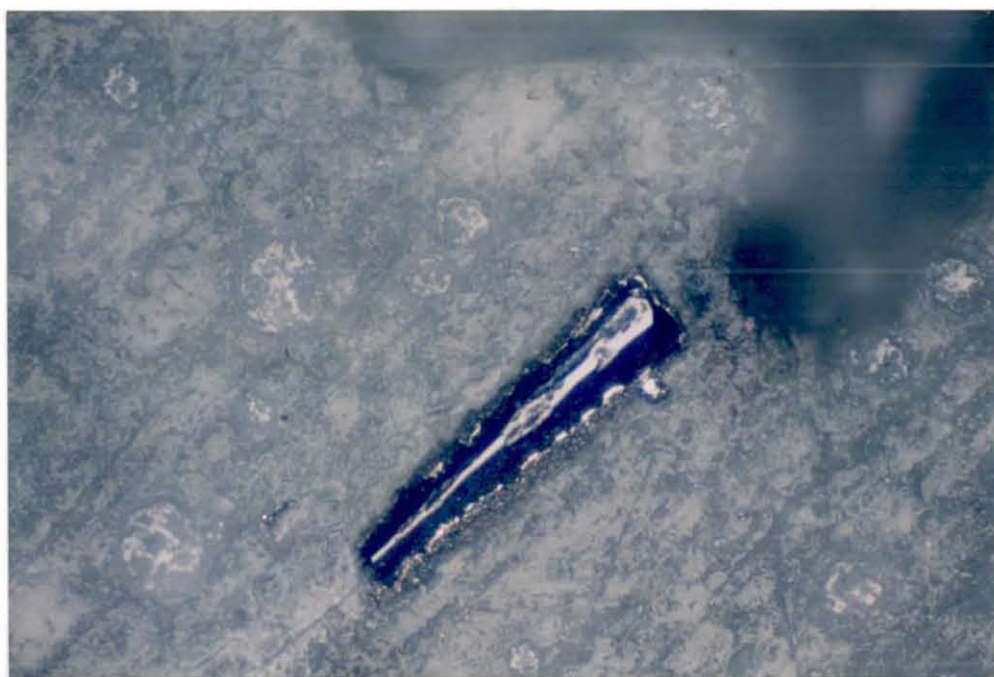
(b)
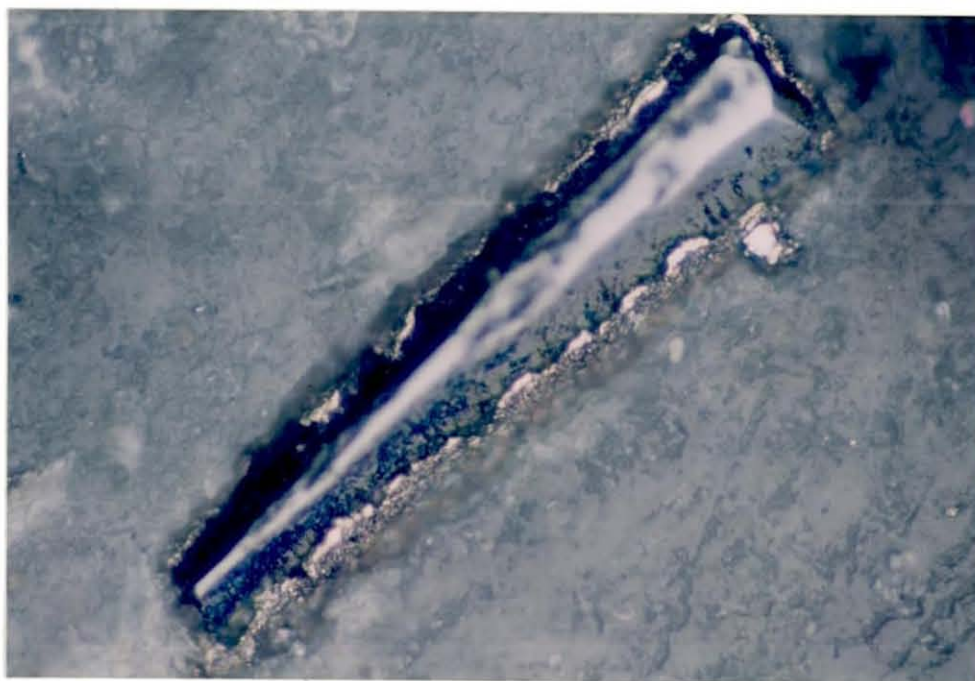
(c)

FIGURE 66

Photographs of polished and sectioned surfaces of WC (45-150 μm) particles coated with 5.71 wt% Ni, by electroless method (X50)

FIGURE 67

SEM Photographs of SiC (29 µm) powder

a)   X160
b)   X800

(a)

(b)

FIGURE 68

SEM Photographs of SiC (250 μm) powder

a) X160
b) X900

(a)
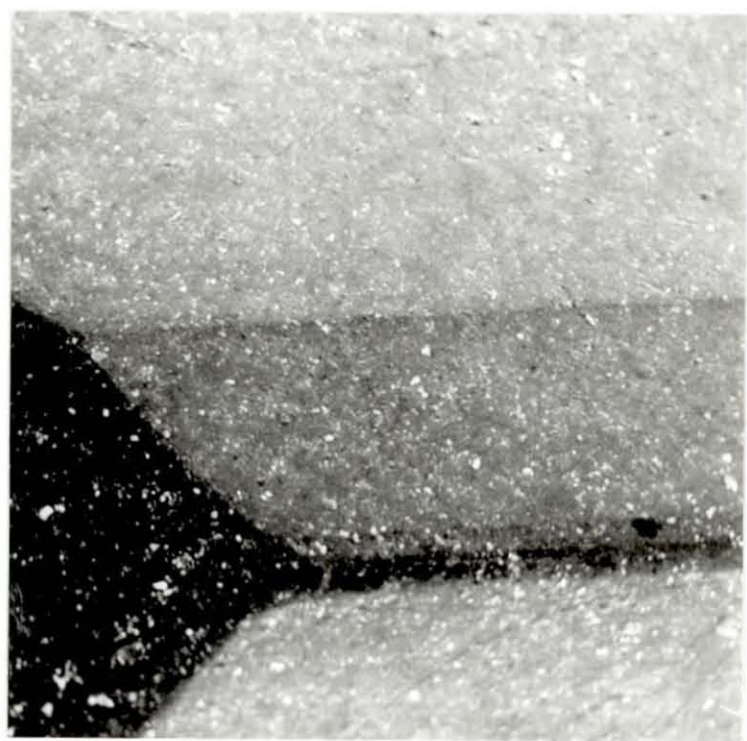
(b)

FIGURE 69

SEM Photographs of SiC (400 µm) powder

a) X160
b) X800

(a)
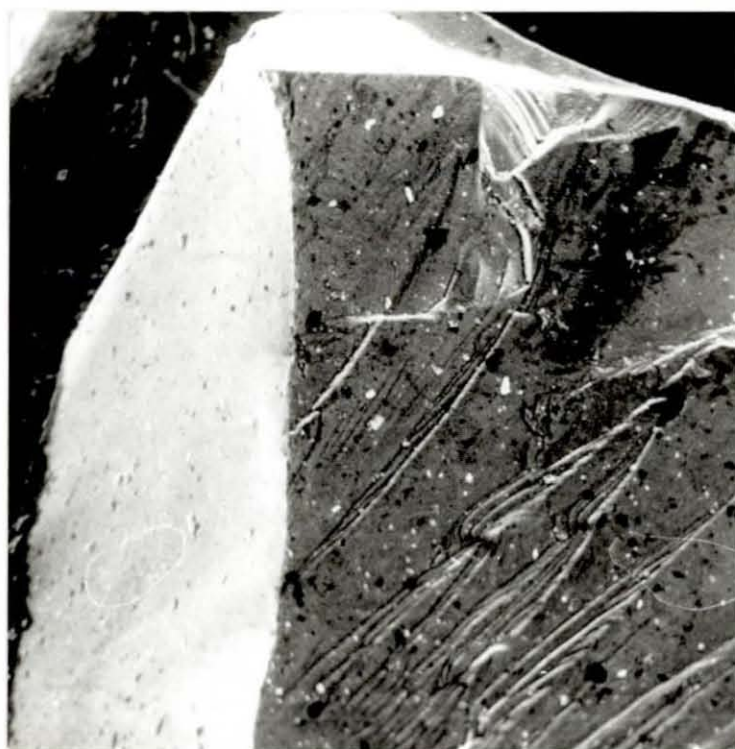
(b)

FIGURE 70

SEM Photographs of Al_2O_3 (45 μm) powder

a) X160
b) X1600

(a)
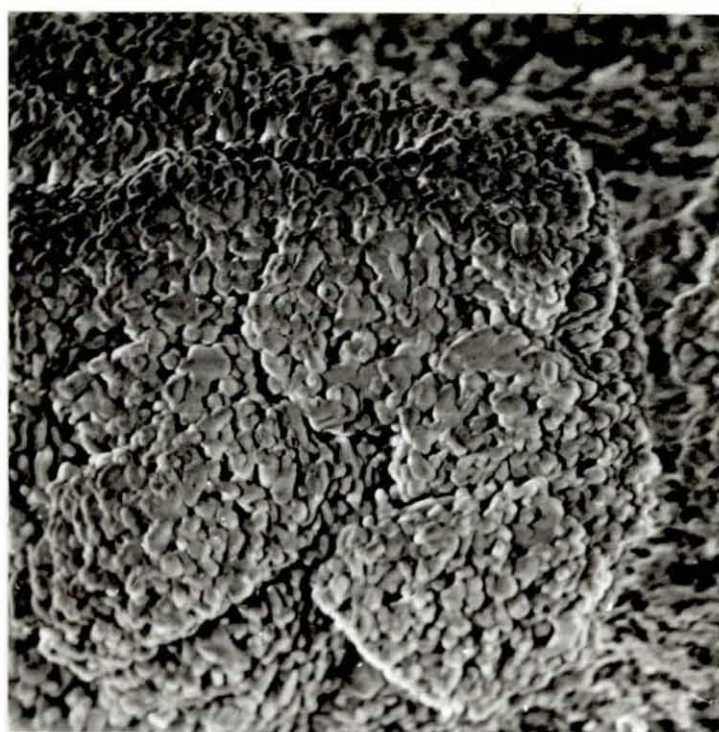
(b)

FIGURE 71

SEM Photographs of WC (45-150 μm) powder

a)   X160
b)   X800

(a)
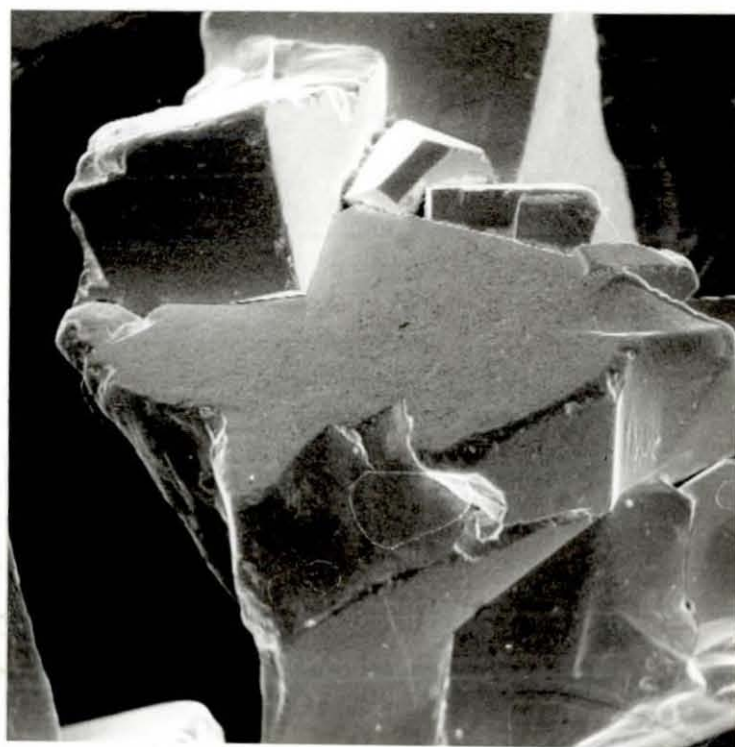
(b)

FIGURE 72

SEM Photographs of TaC (45 µm) powder

a)   X400
b)   X2000
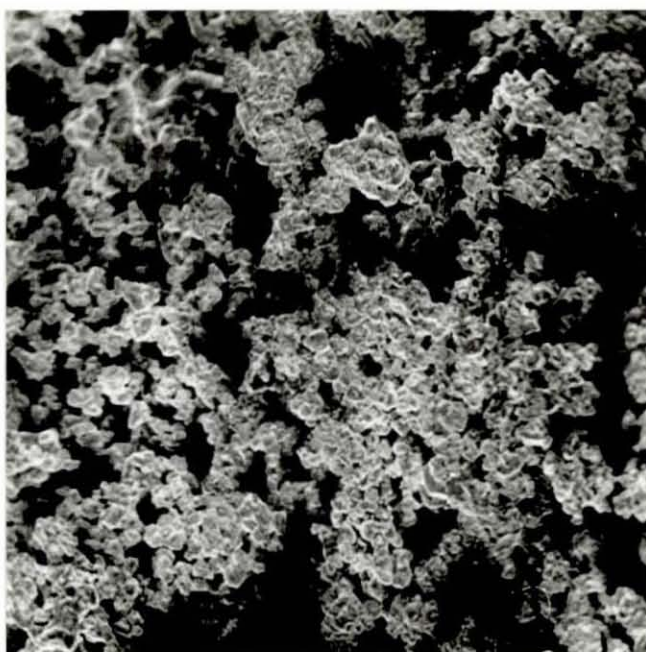
(a)
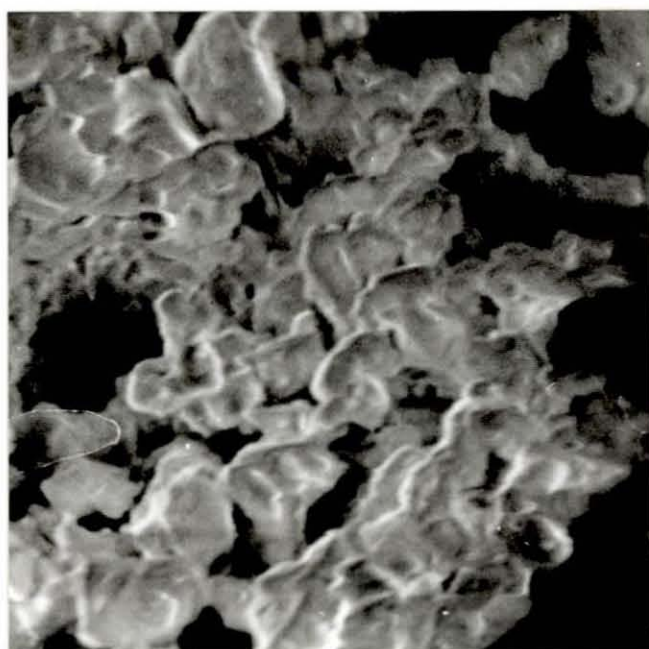
(b)

FIGURE 73

SEM photographs of Cu plated Al_2O_3 (45 μm) particles
obtained by single plating using electroless method.
(Cu content: 3.4 wt%). Al_2O_3 powder: 5g; Temperature
30°C; quantity of solution: 150 ml; Time: 15 min;
agitation: manually.

a) X180
b) X900
c) X4500

(a)

(b)

(c)
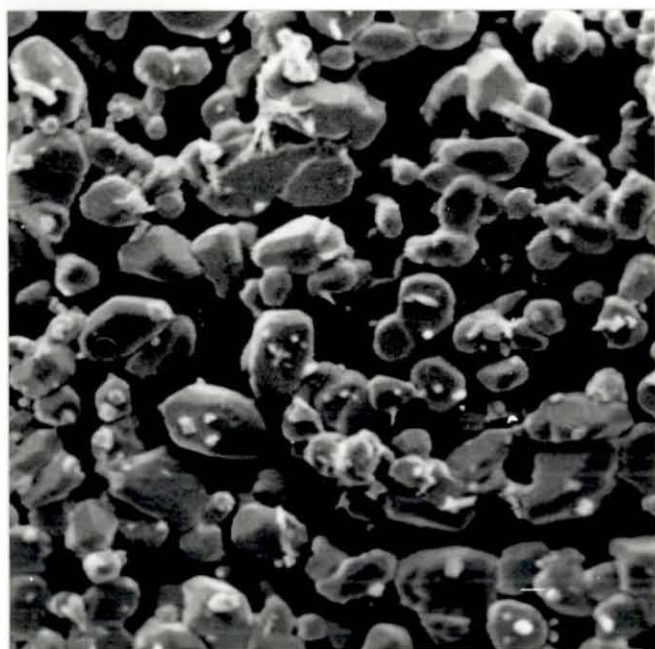
FIGURE 74

SEM photographs of Cu plated Al_2O_3 (45 µm) particles obtained by multiple plating using electroless method. (Cu content: 7.1 wt%). Al_2O_3 powder: 5g; Temperature 30°C; Quantity of Cu solution and plating time per cycle: 150 ml, 5 min; Number of cycles: 3.

a)   X900
b)   X4500
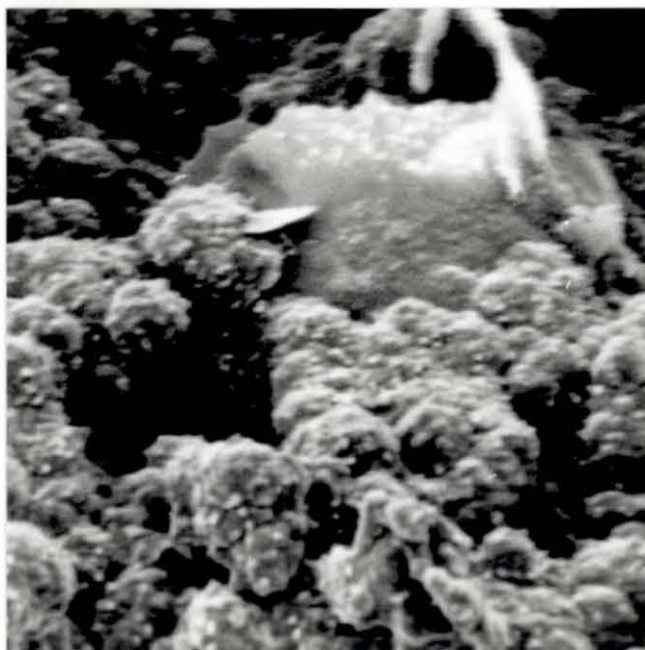
(a)
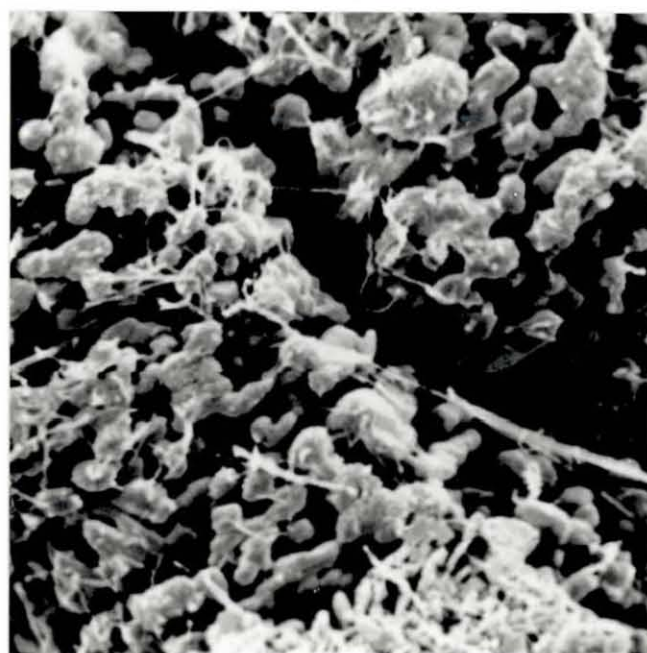
(b)

FIGURE 75

SEM photographs of Cu plated Al_2O_3 particles obtained by multiple plating using electroless method. (Cu content: 11.7 wt%). Al_2O_3 powder: 5g; Temperature 30°C; Quantity of Cu solution and plating time per cycle: 150 ml, 5 min; Number of cycles: 5.

    a)   X180
    b)   X900
    c)   X4500

(a)
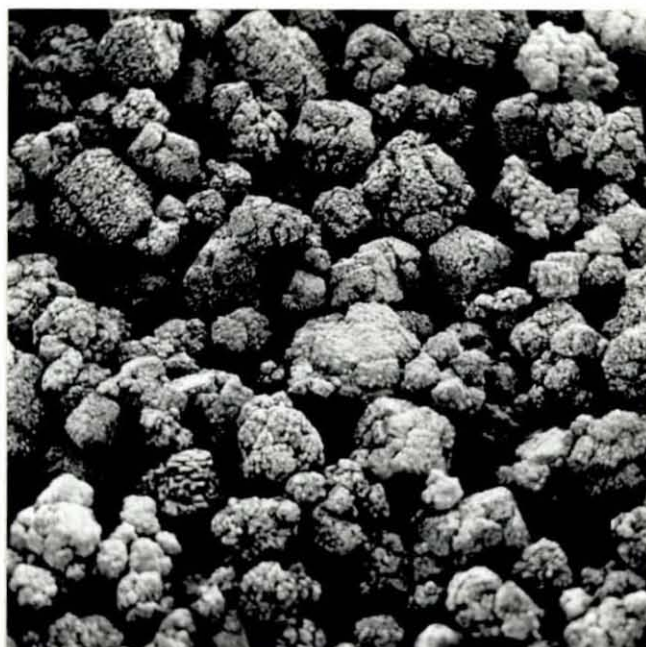
(b)

(c)
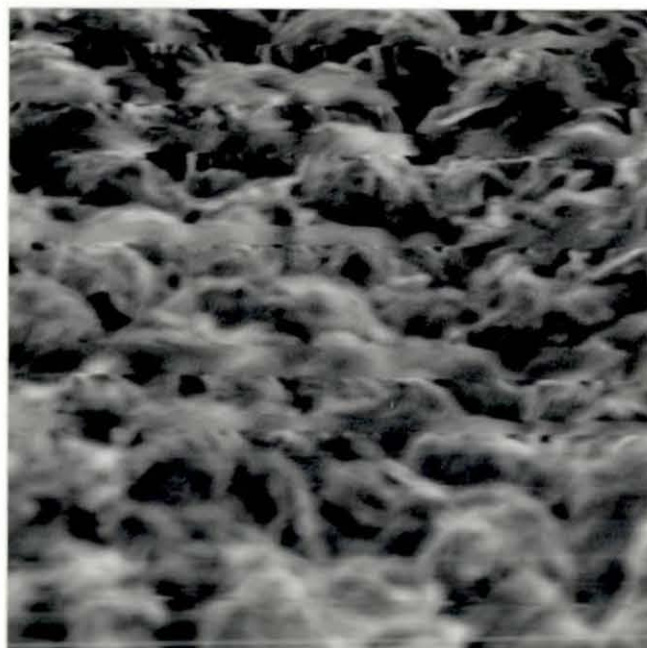
FIGURE 76

SEM photographs of Cu plated TaC particles obtained by multiple plating using electroless method. (Cu content: 12.8 wt%). TaC powder: 5g; Solution: 50 ml; Temperature: $30^{\circ}C$; Time: 5 min; Number of cycles: 9.

a)   X200
b)   X900
c)   X4500

(a)

(b)
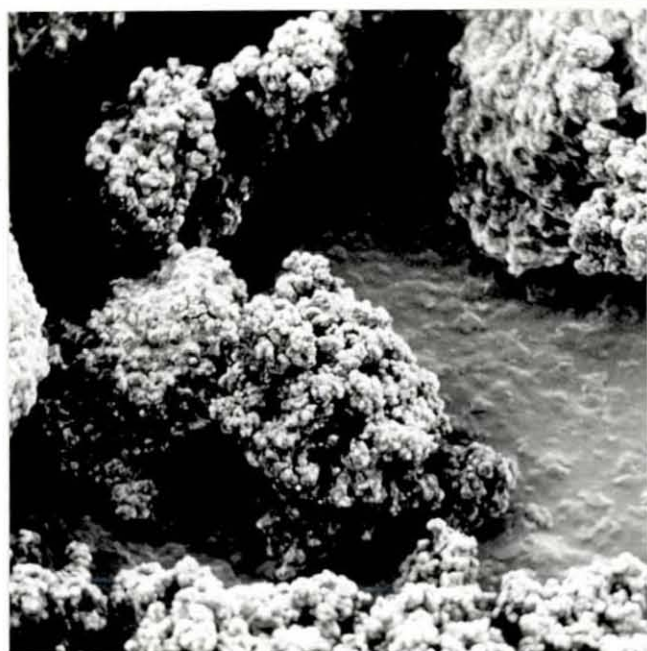
(c)

FIGURE 77

SEM photographs of Cu plated WC (45-150 µm) particles
obtained by single plating using electroless method.
(Cu content: 8.74 wt%). WC powder: 10g; Solution:
200 ml; Temperature: 30°C; Time: 5 min.

a) X180
b) X900
c) X4500

(a)

(b)
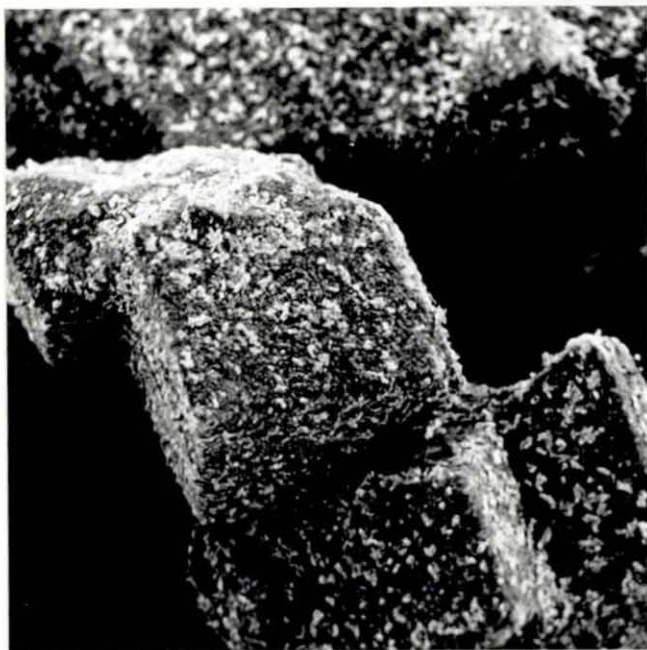
(c)
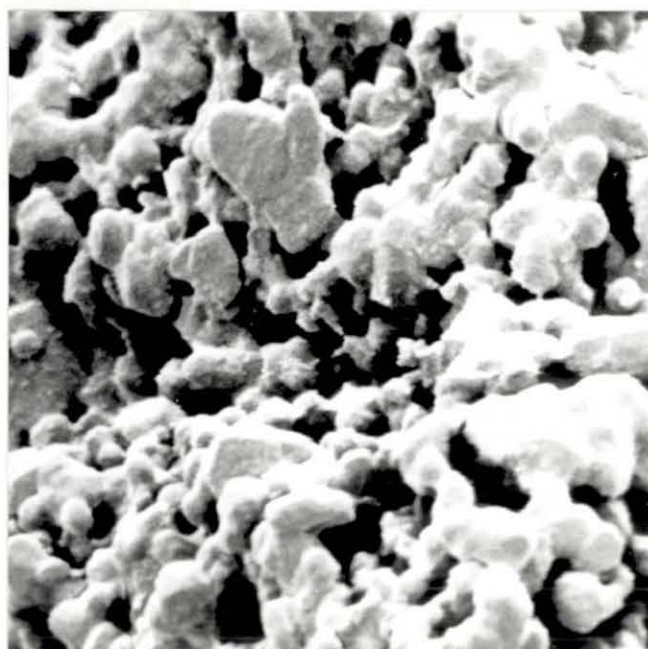
FIGURE 78

SEM photographs of Cu plated WC (45-150 µm) particles obtained by multiple plating using electroless method. (Cu content: 35 wt%); WC: 10g; Solution: 200 ml; Temperature: 30°C; Time: 5 min; Number of cycles: 3.

a) X180
b) X900
c) X4500

(a)

(b)
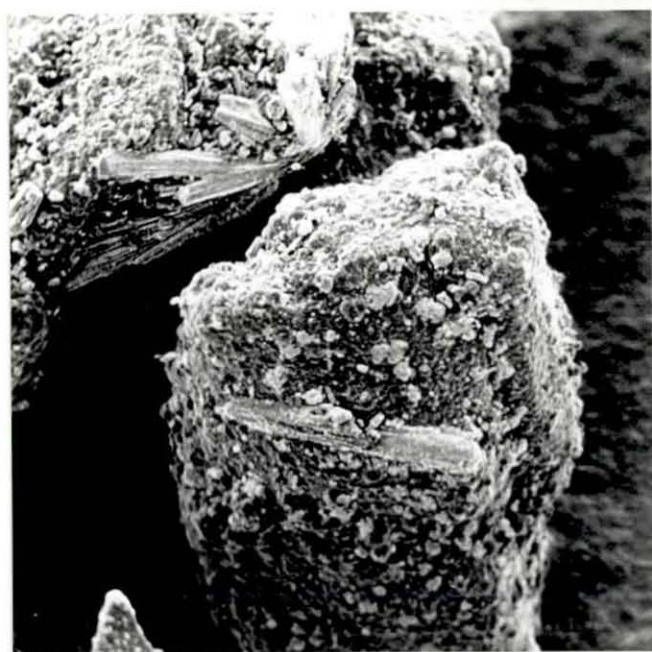
(c)
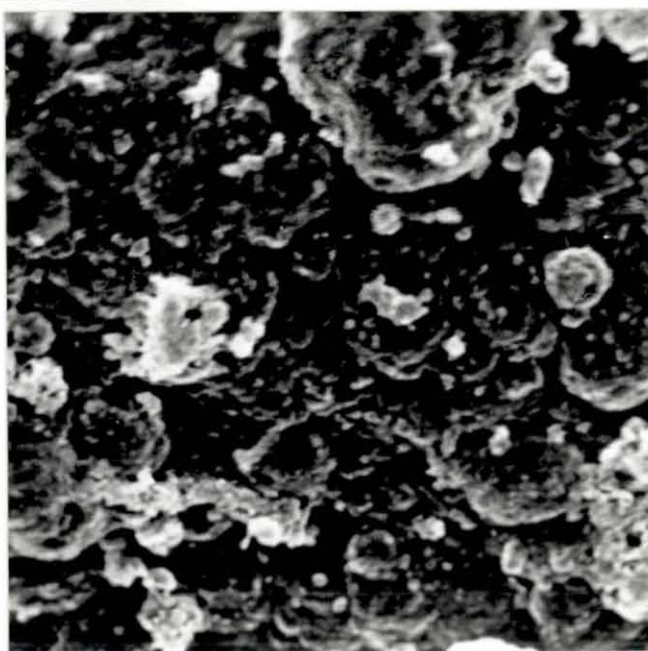
FIGURE 79

SEM photographs of Cu plated SiC (29 μm) particles
obtained by single plating using electroless method.
(Cu content: 0.7 wt%);   SiC powder: 5g;   Solution:
     50 ml;   Temperature: 30°C;   Time: 3 min.

a)   X180
b)   X900
c)   X4500

(a)

(b)

(c)
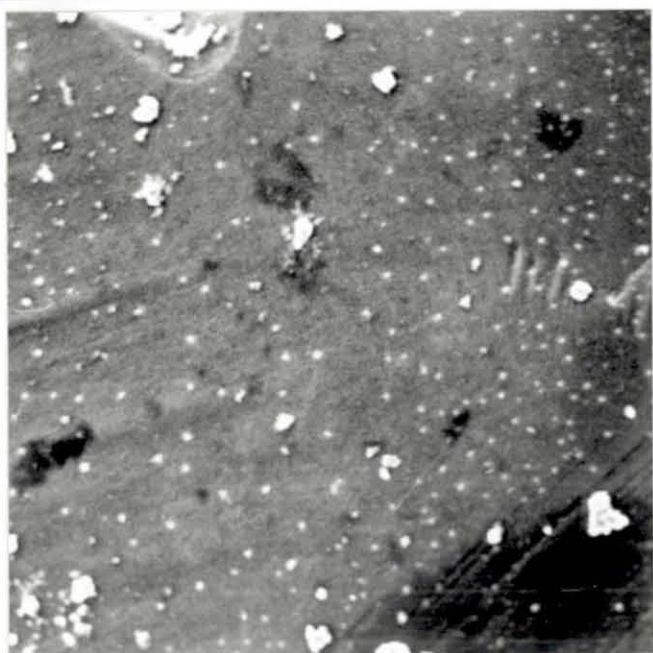
FIGURE 80

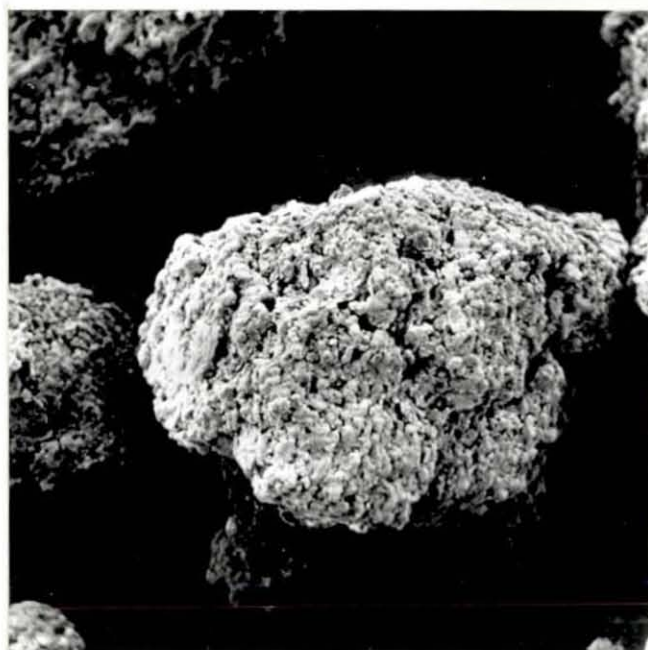SEM photographs of Cu plated SiC (29 μm) particles
obtained by multiple plating using electroless method.
(Cu content: 8.1 wt%); SiC powder: 5g; Solution: 50
ml; Temperature: 30°C; Time: 5 min; Number of
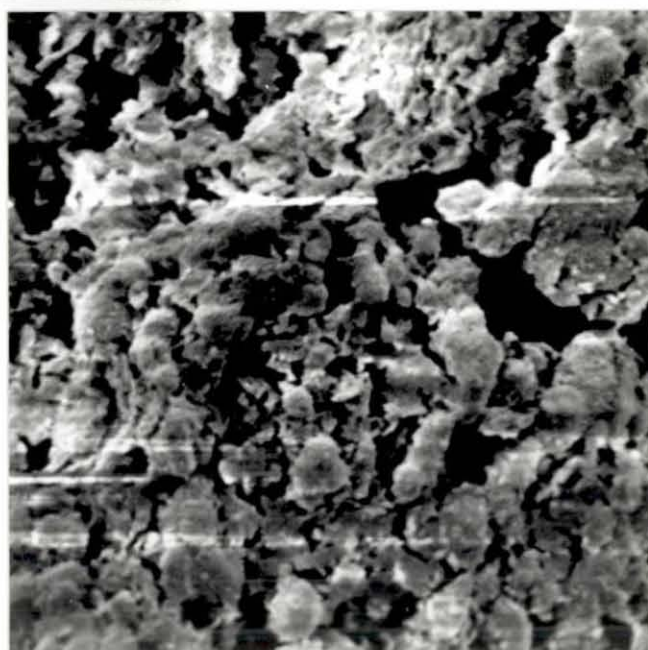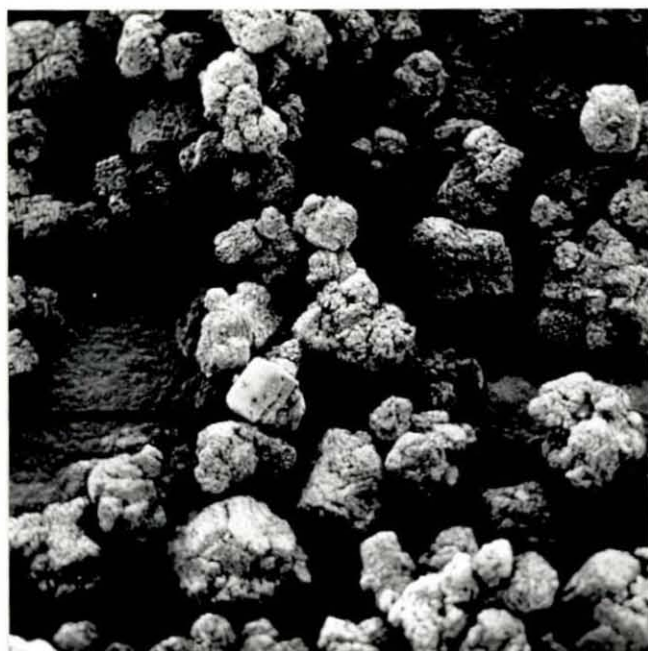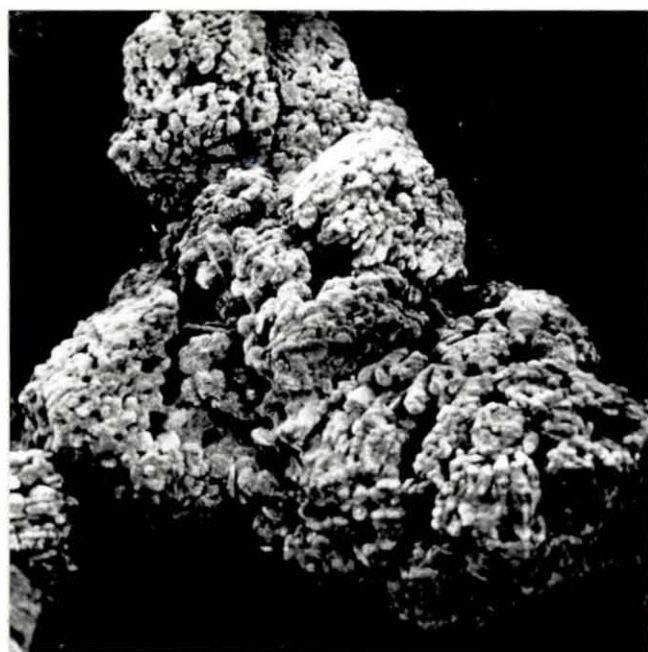cycles: 5.

a)   X180
b)   X900
c) X4500

(a)

(b)

(c)
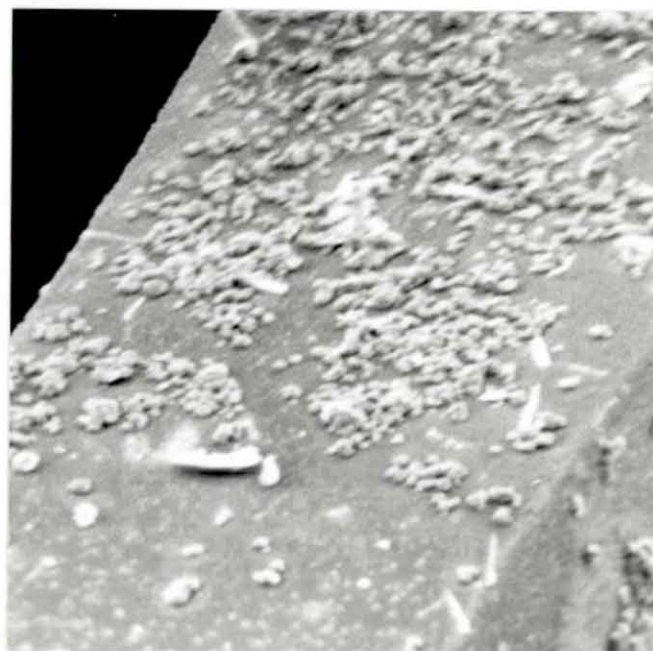
FIGURE 81

SEM photographs of Cu plated SiC (29 µm) particles
obtained by multiple plating using electroless method.
(Cu content: 62.4 wt%); SiC powder: 5g;   Solution:
100 ml;   Temperature: 30°C;   Time: 5 min;   Number of
cycles: 9.

a)   X90
b)   X450
c)   X4500

(a)

(b)
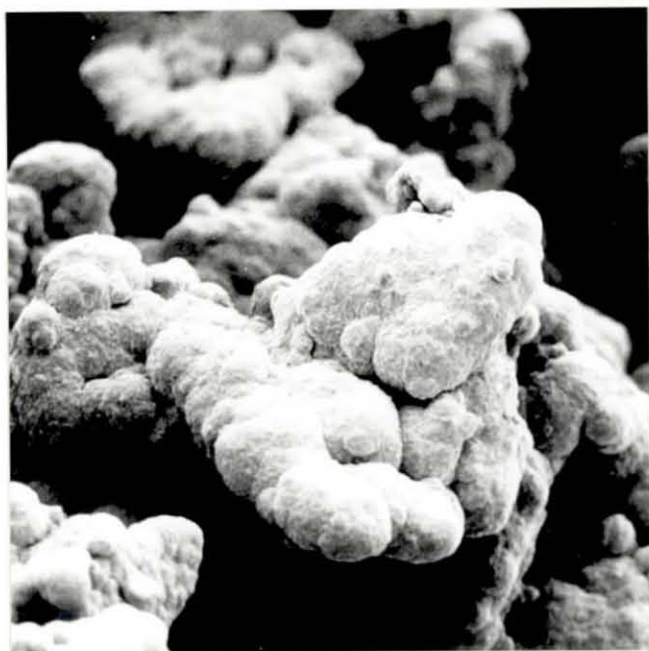
(c)
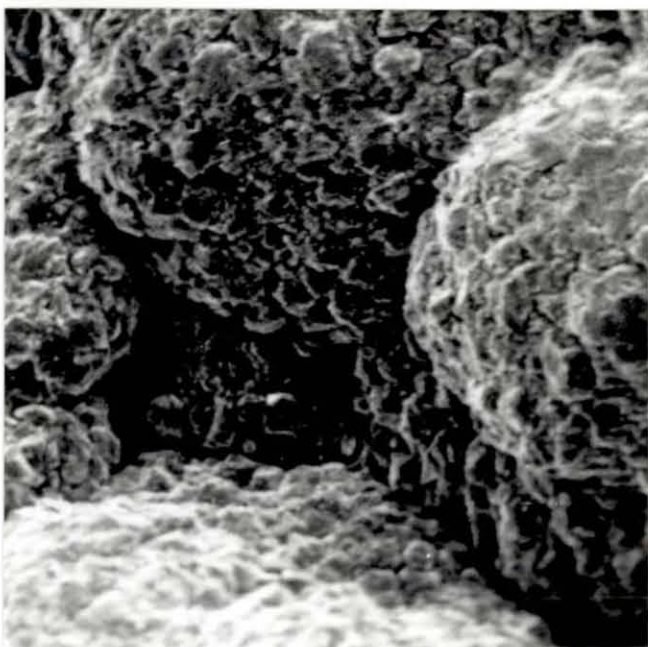
FIGURE 82

SEM photographs of Cu plated SiC (250 µm) particles
obtained using electroless method. (Cu content: 4.3
wt%); SiC powder: 5g; Solution: 300 ml;
Temperature: 30°C; Time: 20 min.

a)   X120
b)   X500
c)   X4500

(a)

(b)
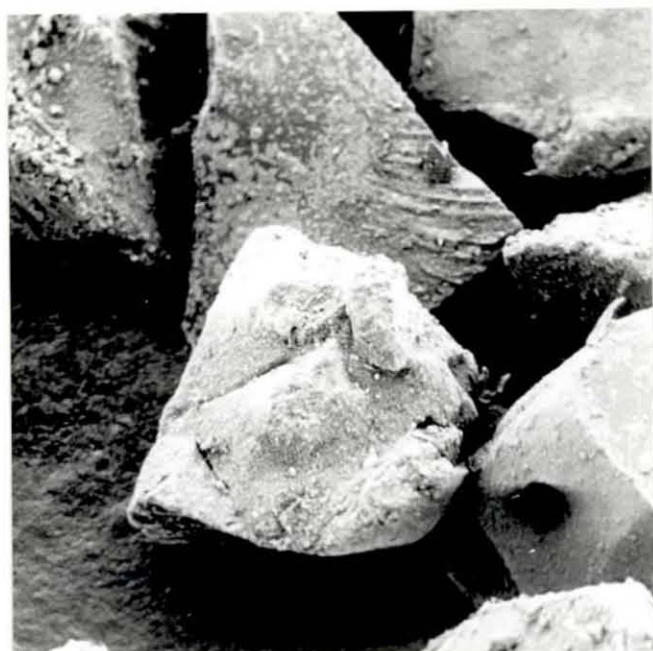
(c)
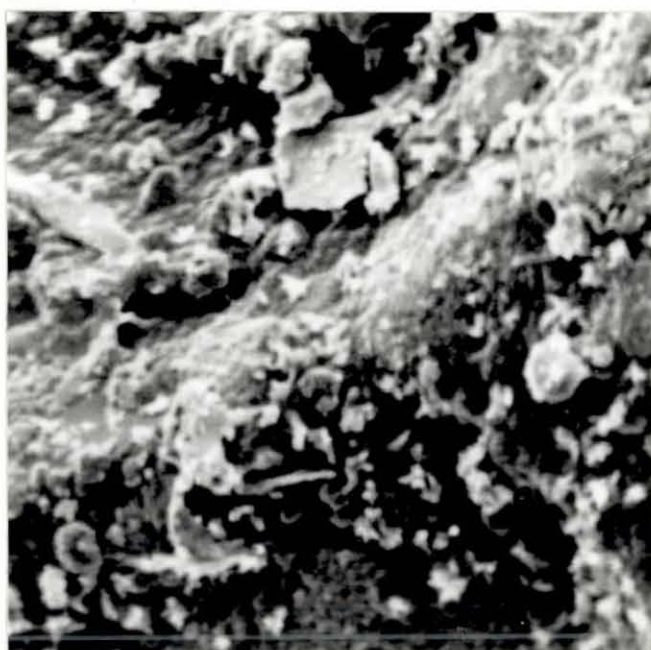
FIGURE 83

SEM photographs of Cu plated SiC (400 µm) particles
obtained using electroless method. (Cu content: 5.3
wt%); SiC powder: 5g;   Solution: 200 ml;
Temperature: 30°C; Time: 30 min.
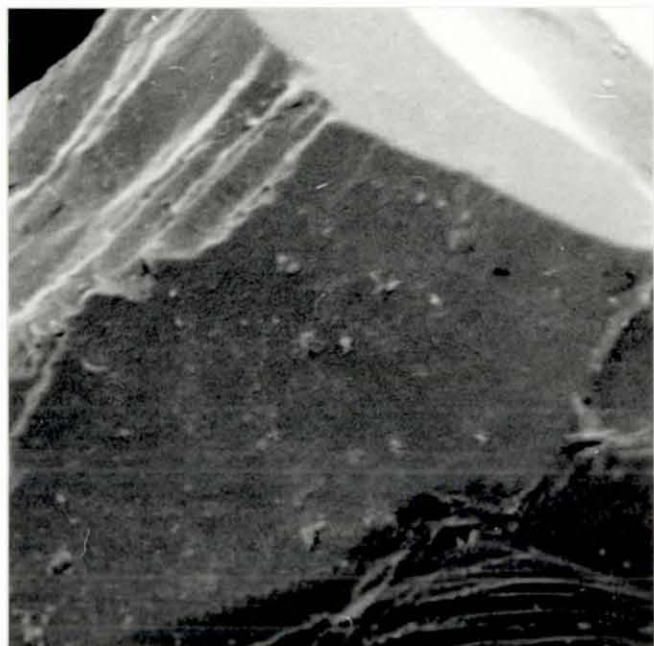
a)   X90
b)   X500
c) X4500

(a)

(b)
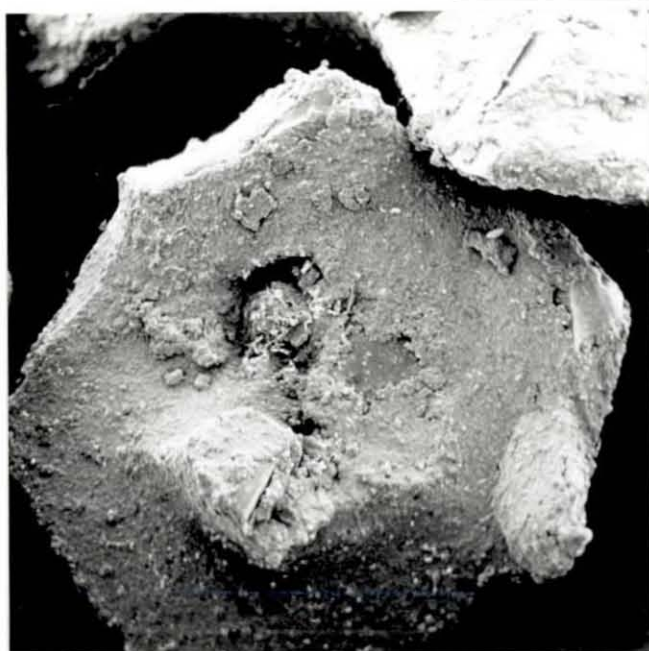
(c)

FIGURE 84

SEM photographs of Ni plated Al_2O_3 (45 μm) particles obtained by single plating using electroless method. (Ni content: 1.9 wt%); Al_2O_3 powder: 5g; Temperature: 40°C; Quantity of Ni Solution: 100 ml; Time: 15 min; Agitation: manually.
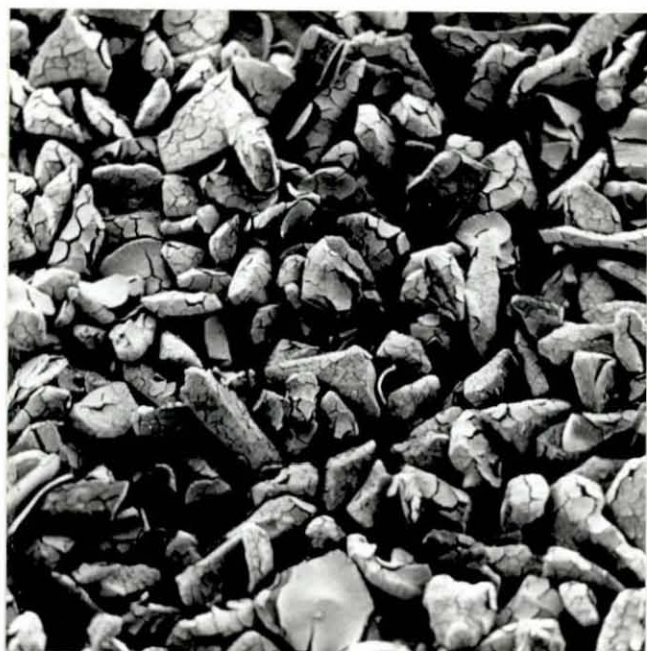
a) X180
b) X900
c) X4500

(a)
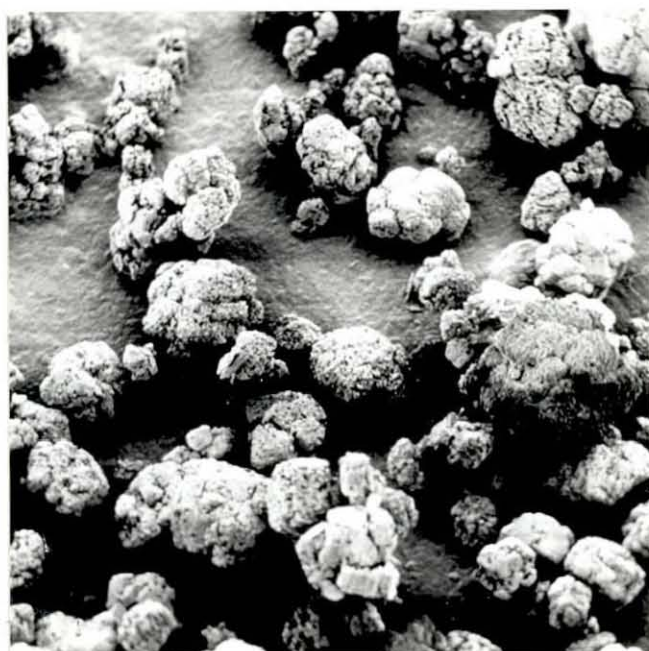
(b)

(c)

FIGURE 85

SEM photographs of Ni plated Al_2O_3 (45 µm) particles obtained by multiple plating using electroless method. (Ni content: 3.1 wt%); Al_2O_3 powder: 5g; Temperature: 40°C; Quantity of Ni Solution and plating time per cycle: 50 ml, 5 min; Number of cycles: 3.

a) X180
b) X900
c) X4500

(a)
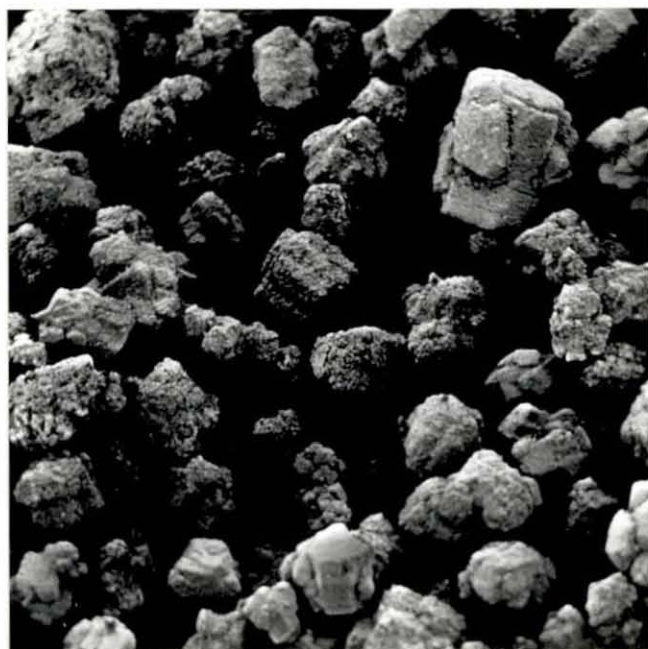
(b)

(c)

FIGURE 86

SEM photographs of Ni plated Al_2O_3 (45 μm) particles obtained by multiple plating using electroless method. (Ni content: 6.34 wt%); Al_2O_3 powder: 5g; Temperature: 40°C; Quantity of Ni Solution and plating time per cycle: 50 ml, 5 min; Number of cycles: 5.

a) X180
b) X900

(a)

(b)

FIGURE 87

SEM photographs of Ni plated SiC (29 µm) particles obtained by single plating using electroless method. (Ni content: 1.3 wt%); SiC powder: 5g; Solution: 100 ml; Temperature: 40°C; Time: 15 min.
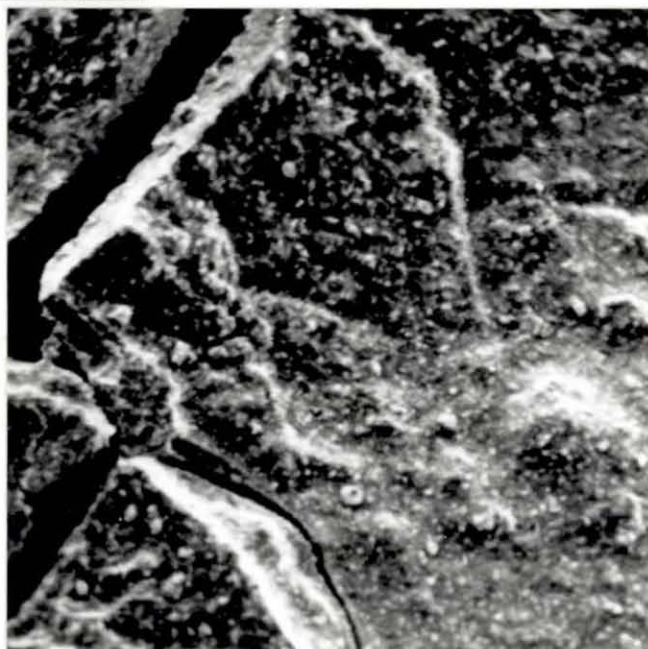
a) X180
b) X900
c) X4500

(a)

(b)

(c)

FIGURE 88

SEM photographs of Ni plated SiC (29 μm) particles
obtained by multiple plating using electroless method.
(Ni content: 8.33 wt%); SiC powder: 5g; Solution:
100 ml; Temperature: 40°C; Time: 5 min; Number of
cycles: 6.

a) X180

b) X900

c) X4500

(a)

(b)

(c)

FIGURE 89

SEM photographs of Ni plated SiC (250 μm) particles
obtained by multiple plating using electroless method.
(Ni content: 3.5 wt%); SiC powder: 5g; Solution:
100 ml; Temperature: 40°C; Time: 5 min; Number of
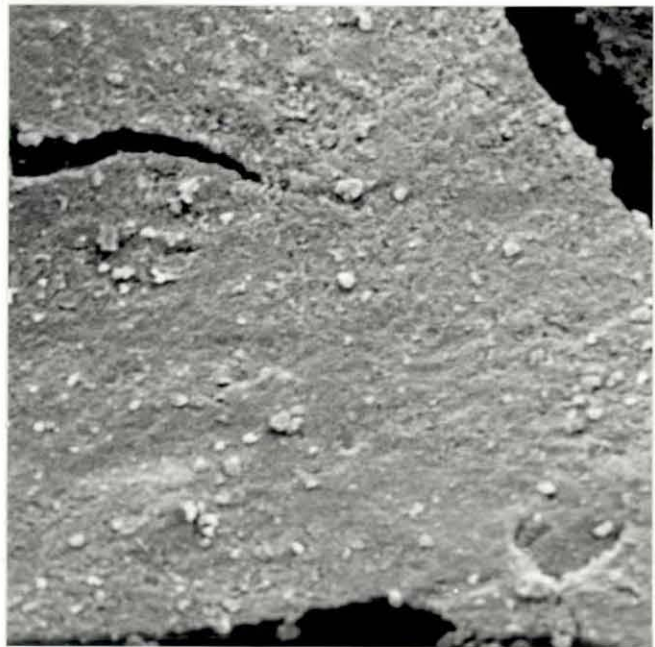cycles: 2.

a)   X45
b)   X3000
c)   X4500

(a)

(b)

(c)

FIGURE 90

SEM photographs of Ni plated WC (45-150 µm) particles obtained by multiple plating using electroless method. (Ni content: 5.71 wt%); WC powder: 10g; Solution: 100 ml; Temperature: 40°C; Time: 20 min; Number of cycles: 4.
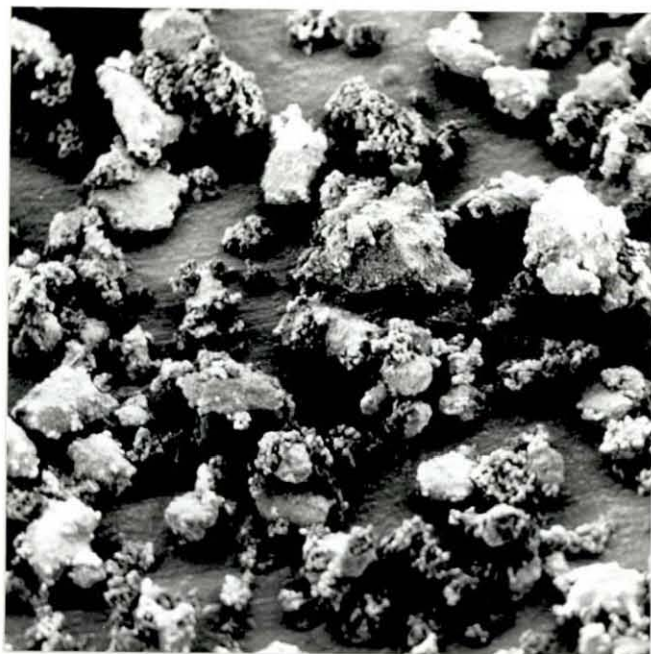
a)   X45
b)   X120
c)   X4500

(a)

(b)

(c)

FIGURE 91

SEM photographs of Ni plated TaC (45 μm) particles obtained using electroless method. (Ni content: 4.8 wt%); TaC powder: 10g; Solution: 250 ml; Temperature: 40°C; Time: 30 min.
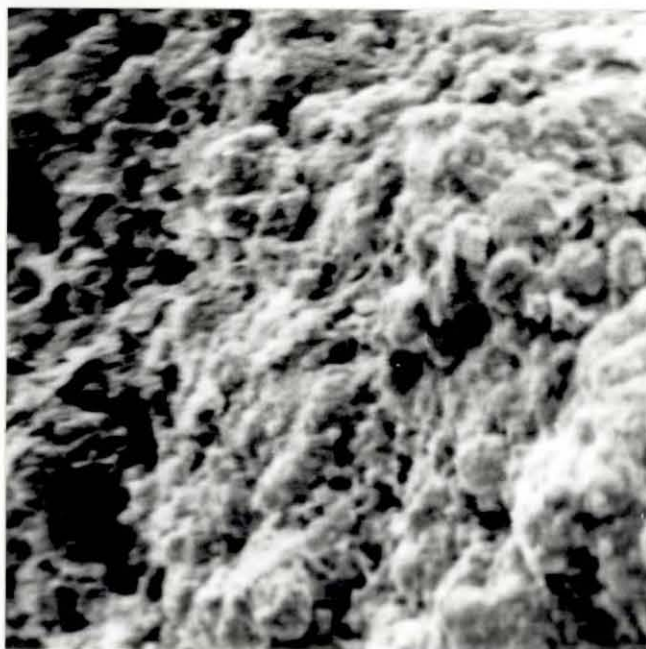
a) X200
b) X900
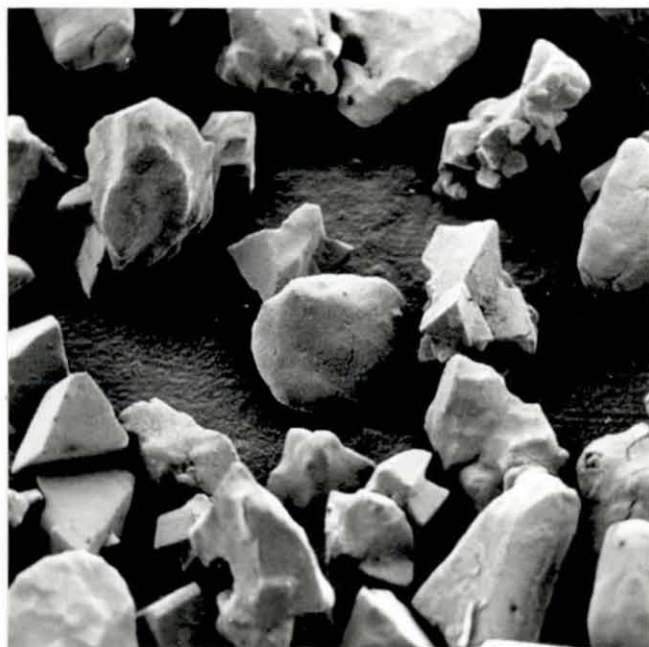c) X5000

(a)

(b)

(c)

FIGURE 92

SEM photographs of Co plated WC (45-150 μm) particles
obtained by multiple plating using electroless method.
(Co content: 12.71 wt%); WC powder: 15g; Solution:
200 ml; Temperature: 55°C; Time: 24 min;   Number
of cycles: 3.

a)   X180
b)   X800
c)   X5000

(a)

(b)

(c)