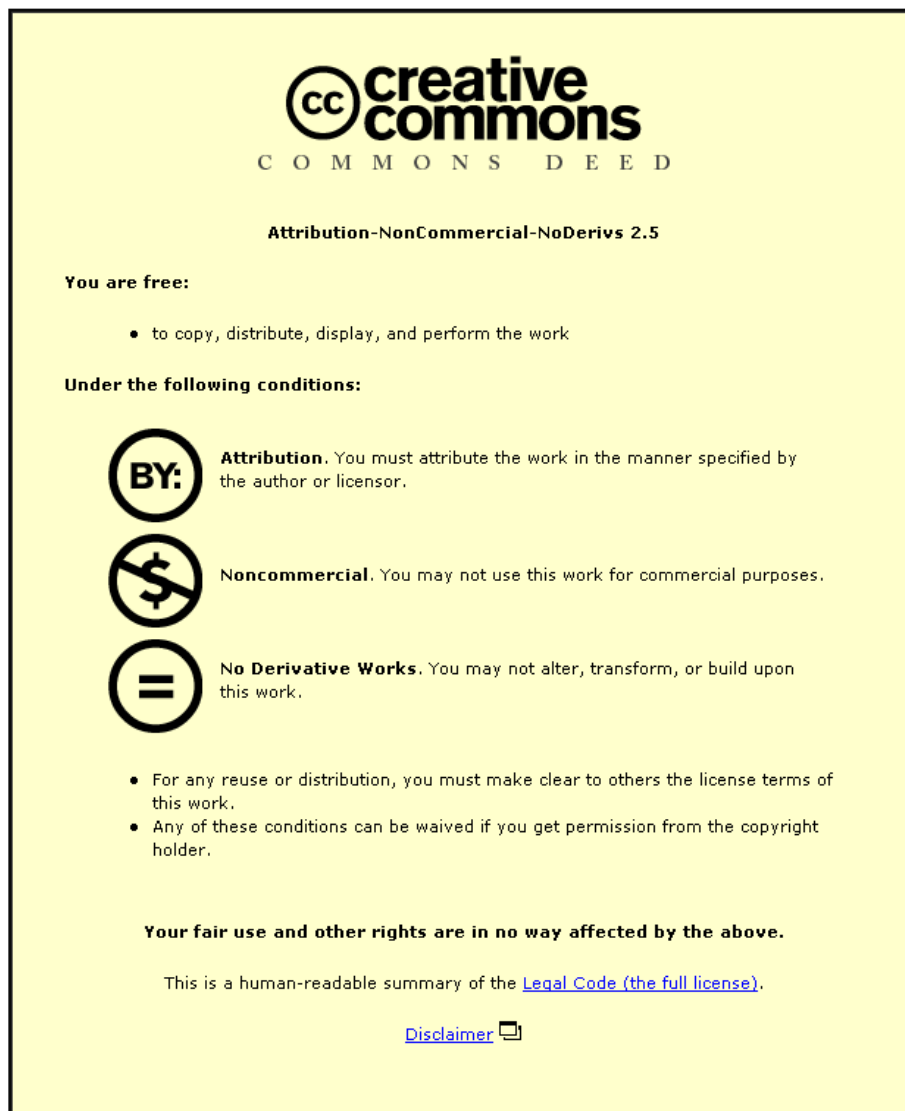


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SOME ASPECTS OF THE COLD COMPACTION

BEHAVIOUR OF METAL POWDERS

by

Peter John James M.Met B.Met M.I.M.

Doctoral Thesis

Submitted in partial fulfilment of the requirements for
the award of Doctor of Philosophy of the
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SYNOPSIS

An analytical appraisal has been made of the cold compaction behaviour of a series of metal powders of different geometry, crystallographic and metallurgical structure, variable size and size distribution. The powders were pressed isostatically to eliminate die-wall friction effects arising from the more common method of die compaction used industrially.

Studies were carried out on sieved fractions of the powders and included an appraisal of different mechanisms of compaction that were found to operate over specific ranges of pressure. This appraisal included a re-assessment of the Hausner friction index whereby a modified version based upon the extrapolated density value at zero pressure derived from the Konopicky-Shapiro equation of state was found to apply more satisfactorily.

It was found that the stages of compaction could be interpreted better from the Konopicky-Shapiro equation of state compared to that of Kawakita. This was supported by studies of the work-hardening behaviour of the powders. Micro-hardness values were measured on loose powder particles and at inter-particle boundary regions in the pressed compacts, following resin impregnation and metallographic preparation. By applying the Meyer work-hardening index and relating a quantitative assessment of particle flattening from scanning

electron microscopical studies to compacting pressure, the response to work-hardening of each powder type was accurately assessed.

Two other areas of compaction were investigated involving die-compaction. The first area investigated the tensile strength behaviour of a series of fractions of die-compacted zinc powder. Strength was found to increase with compacting pressure and to be related to particle size. An inversion in strength at intermediate compacting pressures was attributed to a complex interaction of stress-relaxation; work-hardening and involvement of surface bonding forces. The second area investigated the use of non-metallic glass-ceramic inserts for die-compaction. Punch and ejection forces were monitored on a cam-operated press during the die-compaction of lubricated and non-lubricated iron powder. The results indicated that possibilities may lie in further developments of non-metallic inserts as inexpensive die materials for powder compaction.

INDEX

Page No

1	INTRODUCTION	1
2	LITERATURE REVIEW	4
2.1	Fundamentals	4
2.1.1	Material Types	4
2.1.2	Particle Packing	5
2.1.3	Interparticle Friction	5
2.1.4	Transmission of Forces	5
2.1.5	Bonding between Particles	6
2.1.6	The Mechanisms of Compaction	6
2.1.7	Other features affecting Consolidation	7
2.2	Isostatic Pressing	8
3	SCOPE OF INVESTIGATION	8
4	EXPERIMENTAL PROCEDURE	11
4.1	Metal Powders	11
(a)	Nickel	12
(b)	Copper	12
(c)	Aluminium	12
(d)	Stainless Steel	12
(e)	Sponge Iron	13
(f)	Zinc	13
4.2	Isostatic Pressing Equipment	13
4.2.1	Monobloc Die-Set	13
4.2.2	Pressure Transmission Medium for Monobloc Die-Set	14
4.2.3	Wet-Bag Tooling	15
4.2.4	Compound Die-Set	16
4.2.5	Pressure Transmission Medium for Compound Die-Set	17
4.2.6	Calibration of Compound Die-Set	18
4.2.7	Calibration of Monobloc Die-Set	19

	<u>Page No.</u>
4.3 Die Compaction Equipment	20
4.3.1 Dog-Bone Die-Set	20
4.3.2 Glass Ceramic Die-Inserts	21
4.4 Powder Measurements and Characterisation	22
4.4.1 Particle Sieving	22
4.4.2 Flow Rate	23
4.4.3 Apparent Density	23
4.4.4 Tap Density	23
4.4.5 Particle Shape and Surface Area	24
4.4.6 Particle Microhardness	25
4.5 Compact Measurements	27
4.5.1 Preparation	27
4.5.2 De-airing Procedure	28
4.5.3 Green Density Measurements	28
4.5.4 Fracture Surface Examination	30
4.5.5 Procedure for Metallographic Examination	31
4.5.6 Metallographic Examination	32
4.5.7 Work Hardening Assessment	33
4.5.8 Pore Size Distribution	33
4.5.9 Tensile Strength	34
5 RESULTS AND ANALYSES	34
5.1 Particle Characterisation	34
5.1.1 Flow Rate	34
5.1.2 Apparent Density	36
5.1.3 Tap Density	37
5.1.4 Surface Area	38
5.1.5 Meyer Hardness	39
5.2 Pressure-Density Relationships	40

	<u>Page No</u>
5.3 Isostatic Compaction Studies	44
5.3.1 Effect of Particle Size	44
5.3.2 Effect of Particle Geometry	45
5.3.3 Effect of De-Airing	46
5.3.4 Applicability of Kawakita Equation of State	48
5.3.5 Applicability of Konopicky-Shapiro Equation of State	49
5.3.6 Interpretation of Constants in the Kawakita and Konopicky-Shapiro Equations of State	49
5.3.7 Pore Closure	56
5.3.8 Fracture Surface Assessment	57
5.3.9 Metallographic Examination	61
5.3.10 Particle Work-Hardening	62
5.4 Die Compaction Studies	64
5.4.1 Green Tensile Strength	64
5.4.2 Glass-Ceramic Die Inserts	70
6 DISCUSSION OF RESULTS	72
6.1 Introduction	72
6.2 Particle Characteristics	73
6.3 Isostatic Compaction Studies	75
6.3.1 Effect of Particle Characteristics	75
6.3.2 Effect of De-Airing	76
6.3.3 Applicability of Equations of State	76
6.3.4 Densification Mechanisms	77
(a) The stage of transitional restacking	78
(b) The stage of local flow	79
(c) The stage of bulk compression	81
6.4 Die Compaction Studies	82
6.4.1 Tensile Strength	82
6.4.2 Glass-Ceramic Die Inserts	83

	<u>Page No</u>
7 CONCLUSIONS	85
7.1 Particle Characterisation	85
7.2 Isostatic Compaction	86
7.3 Die Compaction	90
8 SUGGESTIONS FOR FURTHER WORK	92
9 REFERENCES	95 - 98
TABLES	
FIGURES	
APPENDICES	(i) - (viii)

1. INTRODUCTION

The work described in this thesis is complementary to that already published by the author¹⁻¹⁰ and details the philosophy, experimental approach and techniques carried out in connection with the cold compaction of metal powders. The origins of the work began almost ten years ago when the author was introduced to the powder metallurgy (P M) process, which among other issues, is still largely concerned with the production of metal sinterings of high precision and close tolerances, directly from metal powders. During the middle 1960's the process was enjoying active and commercial development with annual growth rates of the order of 15-20%. However towards the end of the 1960s, over-capitalisation in equipment, based to some extent on un-sound optimistic forecasts in growth rates had led to a situation where these growth rates over the early 1970's were not realised. This was believed due to increasing competition from other material areas: the increasing use of plastics and improved technologies in more established metal fabrication areas e.g. die-casting. Since the P M process is very capital intensive full utilisation is necessary to provide for healthy development.

The process, basically is quite simple for the production of parts in great quantity. Metal powder, which may be made by the sinterings firm, though more usually, purchased from an outside specialist powder producer, is compacted to a given "green" density by die or isostatic means. The part is then sintered to a slightly higher density in special controlled-atmosphere

furnaces and subjected, if necessary, to a number of post-sintering operations e.g. coining, sizing, steam treatment etc. The process has been well-documented both theoretically and on more practical grounds by a number of references¹¹⁻¹⁵ on the subject.

Of the two basic processes involved, i.e. compaction and sintering, the latter appears to have received more attention in both the more classical references and in published papers, than that for compaction. While diffusion theory, applicable to agglomeration at temperature, including the considerations of neck growth at particle - particle junctions, the effects of furnace atmosphere and control during sintering have been well documented, powder behaviour and mechanisms of densification during compaction have not been so well-defined. Since the price of metal powder is a vital economic factor in the P M process the methods of making it are similarly competitive and varied. Thus, of the different powder manufacturing techniques: atomisation, electrolytic, chemical reduction, crushing and milling - the resultant shape characteristics, metallurgical structure and properties of the powder particles should have a profound influence upon densification in the green state. This, in turn, will influence the final sintered density level and mechanical and physical properties of the pressed component.

By the end of the 1960's it was becoming apparent that certain improvements and developments in the P M process were necessary for healthy growth, in order to respond to the competitive influences of other processes elsewhere.

Basically two areas emerged: that of increasing density, the other being to increase the size of the compacted part. The former feature largely relates to the final mechanical properties, i.e. strength, hardness and toughness all of which increase with density. It has been toughness, particularly the value of impact strength, which traditionally in P M components has always been low. The improvement in fracture toughness of P M parts has always been a desirable aspect and, to a first degree, can be achieved by simply pressing to higher pressures. This can be done with suitable presses of higher capacity. Whereas ten years or so ago press sizes were rated at maximum capacities of 100 tons, now mechanical presses of up to 500 tons force, and up to several thousand tons force for hydraulic equipment are available. These in turn allow for the pressing of much larger parts.

These trends and attitudes in the P M industry thus provided motivation for a detailed survey of the various factors that influence the compaction of metal powders. It was intended that following such a survey, some possible guidelines could be established with respect to such variables as powder geometry, distribution and metallurgical structure in order to optimise upon a given green density for specific pressing conditions. The work was initiated when the author was in the Metallurgy and Materials Section of the Department of Chemistry and developed within the Department of Materials Technology to the present date of submission. No research grants or funds were available to support this investigation except those limited by Departmental sources.

2. LITERATURE REVIEW

2.1 Fundamentals

A comprehensive literature survey²⁻⁴ reviewing fundamental aspects concerning the consolidation of powders, has been made and is submitted separately. The purpose of this review was to:

- (i) survey those areas which are considered basic to the agglomeration of powders under pressure;
- (ii) rationalise the behaviour of the powder during compaction.

The review considered a number of important topics relevant to agglomeration in general which are summarised as follows:

2.1.1 Materials Types

Here behavioural patterns were compared due to differences in:

- (a) mechanical properties e.g. hardness, plastic behaviour and rates of work-hardening;
- (b) chemical bonding between particles;
- (c) surface effects e.g. friction, adhesion, adsorbed layers;
- (d) geometric factors, e.g. size, shape, surface area and distribution of the powder particles;
- (e) the use of additives or lubricants to the powder;
- (f) mode of compaction e.g. whether pressure is applied unidirectionally or hydrostatically.

2.1.2 Particle Packing

Since initial or loose stacking of a powder depends on its physical characteristics and to a lesser extent on the features of the die geometry the various geometrical arrangements that a powder may assume, which will influence the degree of compressibility subsequent on compaction, were compared. These arrangements included systems involving :

- (a) mono-size spheres;
- (b) multi-size spheres;
- (c) irregularly shaped particles.

2.1.3 Interparticle Friction

This feature governs the free-flowing properties of the powder and is important in situations involving loose handling, i.e. powder transport, storage and filling of dies prior to compaction. The review considered approaches by a number of workers which included the Coulomb Yield Criterion and assessments of yield loci for various powders, along with other angles of internal friction and angles of repose. The Hausner Index which utilises the ratio of tap to apparent densities was also discussed and compared for various powders.

2.1.4 Transmission of Forces

The distribution of pressure within a powder body is now accepted to be relatively variable, depending on the shape of the pressed component and the method by which the overall pressure is applied. Accordingly various techniques were reviewed to establish the manner by which forces are transmitted through a powder mass.

2.1.5 Bonding between Particles

In addition to the various packing considerations influenced by particle geometry, the coherency of a powder is a further factor that influences its stability both under loose and densified conditions. Invariably powders have some form of contaminating film of oxide, grease or adsorbed gas restricting interparticle contact as opposed to the ideal situation where particles have chemically clean surfaces. Four interparticle bonding mechanisms were discussed which included:

- (a) the formation of solid bridges;
- (b) the effect of interfacial forces;
- (c) the effects of adhesive and cohesive forces;
- (d) the effect of intermolecular and electrostatic forces.

2.1.6 The Mechanisms of Compaction

An appreciation of the stages undergone during powder consolidation is important since this should lead to a better understanding of the individual mechanisms of compaction undergone by the powder particles themselves. This in turn should enable better correlations to be made between strength and porosity/structure both in the green state and, subsequently, in the sintered state. From the available literature, the following stages associated with powder densification were proposed:

- (a) slippage of the particles without excessive deformation - sometimes referred to as transitional restacking;
- (b) elastic compression at the particle-particle contact points;

- (c) plastic deformation at these points resulting in the development of contact areas;
- (d) growth of these areas through further plastic deformation and breakage - here fragmentation could occur;
- (e) gradual involvement of the particle when forces become great enough to cause massive deformation and, when the particulate nature has been entirely lost;
- (f) elastic compression of the mass as a whole.

Stages (e) and (f) could thus be regarded as a form of bulk compression where plastic compression of the mass, tending towards final elimination of pores and ultimate densification terminates with the subsequent elastic (recoverable) compression of the mass.

All the above stages are regarded to overlap to a certain degree and are influenced by the properties of the powder material, the particle size/size distribution and shape, lubrication, tooling and method of compaction. A more detailed analysis of the above stages is also submitted separately.⁹

2.1.7 Other features affecting Consolidation

Two other areas were reported which were considered relevant to powder consolidation. These included firstly, the effects of stresses within the powder particles and secondly the effects of internal gas pressures within the powder compact.

2.2 Isostatic Pressing

Within the scope of the investigation into powder densification described below, the decision was made to employ isostatic means for consolidating the powder. Accordingly, this technique was reviewed emphasising, both the principles of the process¹⁰ and as a production process¹ which featured such topics as the tooling and equipment, design and safety criteria, and features of the compact. The applicability of isostatic pressing was also highlighted in terms of materials and components in both articles which are submitted separately.

3. SCOPE OF INVESTIGATION

Initially the programme of work was broadly planned to include compacting a series of metal powders of different geometry, crystallographic and metallurgical structure, size and size distribution. It was considered that this would allow for accurate assessment of each of these parameters on powder densification which in turn would enable a better understanding to be obtained of ways by which more efficient densification of powders could be applied in practice.

In order that powder densification should not be affected nor limited by such variables as die-wall friction and powder-fill height, both of which can lead to non-uniformity of density distribution in the pressed compact¹, the method of isostatic compaction was chosen. This technique was utilised for the majority of compaction studies. However in some cases die-compaction techniques were utilised in the assessment of a

glass-ceramic as a die insert material,⁷ and for the production of tensile test-specimens for the determination of fracture strength of pressed compacts.

The survey²⁻⁴ of the literature concerning the area involving mechanisms of compaction indicated that this field has been studied only to a limited depth in which isolated areas concerning the behaviour of usually one specific powder have been reported. Moreover many of these published references have been concerned with formulating rather empirical relationships of densification and/or pore closure with pressure¹⁶. None of the relationships has been universally applicable, most give an acceptable range over only a limited range of pressures. While it is generally accepted that densification of powders proceeds through a number of stages characteristic of specific modes of behaviour⁹, the precise factors relating to the powder's structural and geometrical properties and features that affect the powder behaviour during compaction have been very much in question.

A series of powders was therefore selected, based on availability from metal powder suppliers, representative of different crystal structures e.g. face-centred and body-centred cubic, and close-packed hexagonal, with varying degrees of morphology e.g. rounded, irregular dendritic. It was not possible to obtain any one powder e.g. atomised copper powder both in an annealed state and in a non-annealed or hardened condition. Thus a direct comparison of the effects of particle hardness for specific powders during compaction could

not be made. However, across the range of powders tested particle hardness varied considerably; thus some indications are given regarding general effects of particle hardness during compaction.

Due to restrictions for the purchase of capital equipment the means by which Isostatic compaction could be applied to the powders was limited to relatively small die-sets. These were of simple construction but capable of producing pressures up to and including 50 t.s.i. The sample compact size was restricted to a length not exceeding 2.25 in (57 mm) and a cross-sectional area of approximately 0.25 sq in (160 mm²). This in turn influenced the amount of powder required for the purpose of assessment. While some powder types were amply available following research programmes elsewhere, purchase of the remaining powders was limited to 10 kg lots.

All powders were sieved to specific size fractions, samples being isostatically pressed at 5 t.s.i pressure intervals over the pressure range 5 - 50 t.s.i.

Each powder fraction was assessed and characterised to include surface area, flow rate, apparent and tap densities, particle hardness and a Meyer microhardness analysis for particle work-hardening response during compaction.

On isostatically pressed compacts, density measurements were carried out followed by structural examination which included metallographic preparation of sectioned faces and S.E.M examination of fractured faces. The metallographic sections were utilised for an assessment of particle flattening and

work-hardening at particle-particle interfaces by micro-hardness surveys together with a quantitative assessment of pore-size distribution by image analysing computer. The effects of de-airing the compact before isostatic pressing were also included.

Two other areas of investigation are reported, both involving die compaction as opposed to isostatic compaction. The first concerns the testing of 'green' as-pressed compacts pressed to a 'dog-bone' tensile test-piece configuration in a floating die-set made available to the Department of Materials Technology from Industry. The second area concerns the assessment of a glass-ceramic material as a die-insert for die-compaction of powders used in conjunction with a mechanically cam-operated press which was also made available from Industry. In the former area the results of the tensile tests are inter-related with the density levels and pressing pressures associated both in the die and isostatically pressed compacts. In the second area the possibility of utilising ceramic and other insert materials for die-compaction is examined with particular reference to the desirability of reducing die-wall friction effects in order to eliminate the objectionable necessity for powder lubrication.

4. EXPERIMENTAL PROCEDURE

4.1 Metal Powders

In all six-materials in powder form were studied over the entire period of investigation, four of them representing the face-centred cubic structure, one body-centred cubic and

one close-packed hexagonal. Information regarding the powders is summarised as follows

(a) Nickel

A grade S powder produced by the Sherritt Gordon process, having an approximate spherical particle shape with occasional small 'protuberances' over the surface. The average chemical composition of the powder was Co 0.068%, Cu 0.007%, Fe 0.011%, S 0.020%, C 0.008%, balance Ni.

(b) Copper

Supplied by J and J Makins (Metals) Limited in two types

- (i) Electrolytic Copper (E.C.P.) prepared by electrolysis giving a dendritic structure modified slightly by a subsequent annealing process applied to the powder.
- (ii) Atomised Copper (A.C.P.) prepared by atomisation giving a substantially spherical particle which was not subsequently annealed.

No analysis of either powder was available.

(c) Aluminium

Supplied by The British Aluminium Company Limited as Baco atomised powder having particles roughly spheroidal in shape. The analysis given was Al 99.5% min., Cu 0.02% max., Si 0.25% max., Fe 0.35% max., Mn 0.02% max.

(d) Stainless Steel

Supplied by B.S.A. Metal Powders Limited in an atomised condition to AISI 316L specification having an irregular particle shape.

(e) Sponge Iron

Supplied by J and J Makins (Metals) Limited to 100P1 grade which is produced by chemical reduction giving a very porous irregular type of particle. The carbon analysis of the powder was <0.01% with a 0.45% hydrogen loss.

(f) Zinc

Supplied by J and J Makins (Metals) Limited following atomisation without any subsequent annealing giving an irregular particle shape. No analysis of the powder was available.

Crystallographically powders (a) - (d) belong to the face-centred cubic system, powders (e) and (f) to the body-centred cubic and close-packed hexagonal systems respectively. Comparisons of all the powders' shape characteristics are illustrated in Figures 1-7.

4.2 Isostatic Pressing Equipment

4.2.1. Monobloc Die-Set

Due to initial financial limitations only relatively inexpensive equipment could be manufactured for operation on University testing equipment in other Departments. Accordingly it was decided to utilise a technique previously employed by Bockstiegel¹⁶. This entailed the manufacture of a simple die set of 'monobloc' construction from a cold-work die tool steel with a movable top plunger and a positioned bottom end closure as illustrated in Figure 8. The die dimensions measured 6 in. outside diameter x 4 in. high with an internal bore of 1.5916 in. dia. giving a nominal cross-

sectional area of 2 sq.in. Isostatic pressing conditions were achieved by filling the bore with a heavily plasticized P.V.C. and subjecting the P.V.C. to pressure via the top plunger, with the opposite end sealed by the bottom end closure. Pressure was applied from a Denison 300 Ton Hydraulic Press (Figure 9) and to prevent extrusion of the P.V.C. from the confines of the die around the sides of the plunger and bottom plug, copper + 2% beryllium mitre rings were positioned as indicated in Figure 8.

4.2.2 Pressure Transmission Medium for Monobloc Die-Set

The uses of reversible gels as moulding materials and as pressure transmitting media have been investigated by Penrice^{17,18}. Based on Bockstiegel's technique¹⁶ Vinamould HMC/1028 grade was obtained from A.Tiranti Limited London, for use as the pressure transmission medium for the monobloc die. Vinamould HMC/1028 is a heavily plasticized P.V.C. which can be melted easily and cast from 120°C into a form which is flexible though sufficiently rigid to secure good permanency of detail.

A technique was devised which enabled a number of pre-cast moulds to be made up simultaneously, preparatory for subsequent compaction. Glass tubing of internal diameter slightly less than that of the die bore was cut up into 5 in. lengths, and the ends flame-rounded. The inner surfaces of the cylinders were lightly greased with a petroleum jelly then each was fixed upright onto a piece of plate glass by plasticene placed around the outside of the base. The Vinamould was

melted in a saucepan and a small quantity poured into each cylinder to a depth of approximately 0.25 in. When set, a bagged powder sample was fixed upright centrally on this PVC 'plug' by a small blob of Vinamould and the remaining space filled up by pouring more Vinamould into the glass cylinder until the powder sample was completely covered. Sufficient Vinamould was cast to ensure that at least a further 0.25 in. covered the bagged sample. When the Vinamould had cooled to room temperature the cylinders were removed from the plate glass and the moulds pushed out with the aid of a steel bar. Figure 10 illustrates the above technique.

This technique allowed a number of compacts to be pressed consecutively; each mould could be easily inserted into the die, the mitre rings, bottom end-closure and plunger assembled and pressing carried out. After pressing the moulds were easily ejected from the die, slit open with a knife to extract the now compacted sample and set aside for re-melting on subsequent occasions.

4.2.3 Wet-bag tooling

Initially a number of flexible rubber-type materials were assessed; these included neoprene and latex tubing both in the form of uncut lengths and pre-cut sheaths from a proprietary manufacturer of isostatic tooling. A simpler and more economical way of obtaining bags was eventually decided upon using a pre-vulcanised liquid latex solution. This is supplied under the trade name of "Revultex" in two grades M and H, the latter being chosen for the production of harder and more durable types of bags.

Lengths of aluminium rod 0.5 in. dia. were used as formers, the rods being hemi-spherically rounded at one end, each polished to a bright finish with metal polish. With the aid of a solution of alcohol saturated with calcium chloride as a coagulant the technique of producing bags was as follows:

- 1 Clean and degrease by dipping into coagulant solution and dry.
- 2 Dip into Revultex and dry for 2 minutes.
- 3 Dip into coagulant solution.
- 4 Dip into Revultex and dry for 2 minutes.

Operations 3 and 4 were repeated until a sufficient thickness had been built up, with a slight pause being allowed after each dip in Revultex to improve the smoothness of deposition. The dipped latex deposits on the aluminium formers were cured usually overnight until translucent and peeled off giving an open-ended cylindrical bag approximately 2.25 in. long.

This length of bag, after filling with powder and sealing with a rubber stopper, was the maximum that could be safely compacted in the Vinamould without danger of cracking of the compact. Allowance of "loss" of compaction volume within the die bore through the end bottom closure plug, and sufficient insertion of the top plunger with enough Vinamould to adequately encase the bagged compact reduced the effective bag length to approximately 2 in.

4.2.4 Compound Die-Set

Later, during the programme a second die-set was manufactured which allowed accurate calibration of the compacting

pressure to be made. Its operation was similar for the monobloc die, i.e. direct pressure application from a plunger to a fluid in a closed vessel. To safeguard against failure of the die under pressure a compound die assembly was produced with an outer cylinder shrunk onto the inner die thus rendering the latter in a state of residual compressive stress. The pressure vessel (Figure 11) consisted of a G110 maraging steel insert with an EN30 outer bolster. The assembly was effected by heating the bolster in a furnace to 500°C and inserting the inner die steel, after cooling in liquid nitrogen. The bore of the EN30 was protected during heating by filling with cast iron chippings. Calculations for the design of the die-set and safe operating pressures are given in Appendix 1. The bottom plug was made with a slight interference fit relative to the bolster and inner die and pushed into the assembly giving a permanent end closure. The top plunger and bottom plug were sealed by Viton O-rings backed by copper + 2% beryllium mitre rings. The chamber dimensions were 6 in. long with a bore diameter of 1.383 in. giving a cross-sectional area of 1.5 sq.in. Figure 12 illustrates the die-set positioned in the Denison 300 Ton press ready for use.

4.2.5 Pressure Transmission Medium for Compound Die-Set

The pressure transmitting medium should not "freeze" at high pressures and should not be of extremely low viscosity. Lowe and Goold¹⁹ studied the behaviour of several different liquids by subjecting them to increasing isostatic pressures and recording the output of a Manganin gauge. The applied

punch pressure and the Manganin gauge pressures were found to be the same for all liquids at pressures up to about 80 kg.mm^{-2} . Above this value considerable divergence occurred as indicated by their results shown in Figure 13. Accordingly the fluid chosen for the pressure transmitting medium, approximating most closely to the ideal line, was a mixture of 80 : 20 glycerol/ethylene glycol.

4.2.6 Calibration of Compound Die-Set

During the initial stages of the programme using the monobloc die set the values of pressure were derived simply from the recorded punch force, divided by the cross-sectional area of the die bore (2 sq.in.) While a similar technique could be applied to the compound die-set, it was decided to assess how the actual pressure generated within the fluid was related to that derived from punch force and cross-sectional area where possible errors could arise due to friction and loss of efficiency of fluid pressure transmission.

Accordingly it was decided to use a pressure transducer device based upon the construction of Manganin coils which have been used by Bridgman²⁰ up to 210 kg.mm^{-2} . Following discussions with N.E.L East Kilbride gauges were made by winding 42 swg Manganin wire non-inductively on perspex spools to give a total resistance of 100 ohm. Copper wires were attached to the Manganin wires through holes in the perspex spools to provide stronger fixing and the coils were subjected to pressure before use. Two pressure gauges were wired in adjacent arms of a Wheatstone bridge circuit, the imbalance being a measure of the

pressure differential. The gauge external to the pressure vessel prevented zero wandering by compensating for ambient pressure and temperature variations.

The terminal seals for the Manganin pressure gauge were made from steel cones 6.35 mm. long tapering from 3.17 mm to 1.59 mm. They were lapped into conical ceramic insulators which were lapped into appropriate holes in the end of the top plunger (ref. Figure 11). The internal and external leads were soldered to the steel terminals.

Calibration of the Manganin pressure gauge was carried out in the pressure vessel where the pressure applied to the plunger was measured using a 200 ton Elliot load cell with an output of 2 mV/V. During pressure application the output of the load cell and the Manganin gauge were recorded simultaneously on a 2 channel potentiometric recorder. The applied punch pressure and the Manganin gauge output were found to be linear up to 45 t.s.i. above which slight increasing divergence occurred. Table 1 presents a comparison between applied pressures and deduced internal pressures.

4.2.7 Calibration of Monobloc Die Set

The design of the monobloc die set did not allow for calibration using the same technique as that for the compound die-set. Penrice¹⁷ has published compressibility data and dimensional changes that occur on pressing and re-pressing 'Vinamould' reversible gel, which is shown to exhibit similar compressibility to liquids such as water and other fluids.

Thus on the basis of Lowe and Goold's work it was anticipated that the nominal applied pressure should equate over a fairly wide range to the true transmitted pressure over the compact.

As a verification, a sieved fraction of atomised copper powder was compacted in both the monobloc and compound die-sets, the latter having already been calibrated and the green densities compared over a range of pressure.

Table 2 presents this comparison, from which it can be seen that there is close agreement in densities over the entire range of compacting pressure. Details regarding these measurements are given in section 4.5.

4.3. Die Compaction Equipment

4.3.1. Dog-Bone Die-Set

This die-set only became available over the latter stages of the experimental work. Originally it was the intention to produce test-pieces isostatically, but difficulties had been experienced with isostatic tool manufacturers which eventually precluded making such specimens by that technique. Thus the more recent availability of the dog-bone die-set was incorporated into the pressing programme and a limited number of tensile test-pieces, die-pressed from zinc powder, was manufactured. Zinc was chosen partly on grounds of availability which allowed a sufficient number of test-pieces to be pressed from each of the sieved fractions. Also zinc is an easily compactible metal in powder form which was considered suitable enough for investigating work-hardening aspects at higher pressing pressures.

The die-set is based upon an M.P.I.F standard 10-63 die construction, incorporating a "floating" table on returnable springs, which when compressed, allows the table to move downwards relative to the bottom plug for ejection of the specimen. The position of this bottom plug was constant such that the ratio of fill-height to compressed height varied no more than between 2.2 - 2.7. The pressed surface area of the whole compact was exactly 1 sq.in. Figure 14 illustrates the main constructional features of the die-set and that of the specimen geometry, while Figure 15 illustrates the die-set positioned in the Denison 300 Ton press used also for pressure application. Since the die-set was supplied without any guide frame, location of the top plunger had to be carefully positioned on the powder fill cavity by hand before full pressure was applied. In addition the use of zinc powder for compaction created certain problems on ejection unless the die-cavity was lubricated by aerosol application of a silicone release agent. This overcame any need for powder lubrication which would have introduced variables and difficulties in assessing compaction behaviour.

4.3.2. Glass-Ceramic Die Inserts

The concepts and general philosophy relating to this area of die-compaction have already been described⁷. Basically, enormous advantages would be gained in the Powder Metallurgy industry if powders for die-compaction did not have to be lubricated. Ideally the perfect die would be one manufactured in a material that should be hard, dimensionally stable, capable of taking a high surface finish, fatigue resistant and with a

negligible coefficient of friction between it and the metal powders which are pressed. Although carbide and cermet dies have been assessed with some degree of success it was considered that glass-ceramic materials may possess many of the above listed die requirements.

It was thus decided to have a number of glass-ceramic inserts manufactured which would be shrunk-fit into EN26 steel bolsters of external profile and dimensions acceptable to a Betema mechanical cam-operated production press, a description of which has been made elsewhere²¹. Full details of the experimental procedure, die assembly, powder pressing programme are already documented⁷ while instrumentation procedure for monitoring the punch and ejection forces using standard die-steel tooling for top and bottom punches are described separately²².

4.4 Powder Measurements and Characterisation

4.4.1. Particle Sieving

Sieving was carried out on an Endecott Test Sieve Shaker using 8 in. (200 mm) dia sieves, to BS410 specification. The time of sieving was 1 hour, on sieves drawn from the following range:

<u>Mesh Number</u>	<u>Nominal Aperture Size μm</u>
72	212
100	150
120	125
150	106
170	90
200	75
240	63
300	53
350	45
400	38

After sieving the powder was collected, and the sieves lightly brushed to clear the perforations ready for the next run.

4.4.2. Flow Rate

This property, which expresses the rate at which a powder will flow under gravity through an orifice is of significance in die-filling prior to pressing. The determination of flow rate was carried out in accordance with the standard test²³, using the Hall Flowmeter (Figure 16). 50g of powder were weighed and placed in the Flowmeter, a finger being placed under the orifice during filling. The time to the nearest second was then recorded for the powder to empty itself from the Flowmeter. Three determinations for each powder were recorded and the average calculated. The Flowmeter was calibrated²³ using 150 mesh 'Standard Turkish Emery' until after careful machining and polishing, the average time for 50g. of emery to pass through was 40 sec.

4.4.3. Apparent density

The apparent densities of the powders were determined under standard conditions²⁴ under which powder was allowed to flow through the Hall Flowmeter into a previously calibrated 25 cm³ density cup (Figure 16). Excess powder overflowing into the cup was carefully scraped off level with the top of the cup and the amount remaining weighed to the nearest 0.01g.

The average of four determinations was recorded.

4.4.4. Tap density

This non-standardised property was determined by allowing powder to flow into a graduated cylinder and clamping it in the

Endecott Sieve Shaker and allowing it to vibrate for 10 min. After this period no further settling was observed, the volume was recorded to the nearest 0.25 cm^3 and the powder weighed as before. Good consistency was obtained with only three determinations.

4.4.5. Particle shape and surface area

Several methods have been proposed for the characterisation of particle shape. Heywood²⁵⁻²⁷ has defined various shape coefficients involving the length, breadth and thickness of a particle and derived surface and volume coefficients. Hausner²⁸ has proposed three shape coefficients including a surface factor

$$Z = \frac{C^2}{12\pi A} \quad \text{-----} \quad 1$$

where C = perimeter of projected particle
and A = area of projected particle

Values of Z for specific powders have been quoted elsewhere³.

In general it was considered that particle shape was a difficult property to accurately quantify and relate to other properties e.g. surface area, density and flow rate. The shapes of the different powders are qualitatively illustrated and compared in Figures 1-7 and it was decided that surface area would offer better interpretation and understanding of compact behaviour during densification. Accordingly, it was decided to measure surface area based upon a relatively simple technique, which intrinsically is not regarded as accurate as the B.E.T. method²⁹ but which under standard conditions enables good comparisons to be made of the relative surface areas of powders of low size distribution.

An air permeametry method was utilised, the principle of which is illustrated in Figure 17. The apparatus consisted of a 100 ml burette inverted over a beaker of de-ionised water, and attached at the top to a glass tube of known bore diameter. Discs of a coarse filter paper were made, approximately to this bore diameter, by an office punch. A small ring of P.V.C. tubing was pushed into one end of the tube and a disc of filter paper pushed through from the other end to rest on the P.V.C. ring. Powder was introduced to give a column length varying from 5 - 12 cm, and after lightly tamping the column was closed by introducing a second disc of filter paper from the other end of the tube. The length of the column was measured to the nearest 0.05 mm and the powder weighed subsequently to the nearest 0.01 g.

Water was sucked up the burette using a pipette filler to just below the tap, which was kept well greased to exclude air, and the tap closed. The cell was connected to the top of the burette by a rubber tube, so that air entering the burette must do so via the cell. The tap was opened and as the water level in the burette fell, air was sucked in through the cell. A stop-watch was used to record the time interval between the 100 ml. mark and the first 10 ml. and every successive 10 ml interval. A graph of $\log_{10}$ volume v. time was plotted and using the methods of calculation outlined in Appendix 2 the weight specific area in $\text{cm}^2 \text{g}^{-1}$ was derived.

4.4.6. Particle Microhardness

Samples of the 'as-received' powders were cold-mounted in a room-temperature curing Araldite CY219/HY219/DY219 resin

system * in 1 3/8 in. dia. pill boxes. After curing the samples were then polished on successively finer grades of emery papers before final polishing with 6 micron and 1 micron diamond paste.

The cold-mounting procedure was chosen so that the metallurgical structure would not be affected by heating effects from hot mounting which might cause some annealing of the powders. This would be very important in the case of Zinc which has a recrystallisation temperature below 100°C.

Hardness measurements were made using a Vickers DM 41 micro-hardness tester attached to a Vickers projection microscope. The procedure described in the standard manual³⁰ was carried out for initial setting-up and calibration of the eye-piece etc. A minimum of six indentations was made at each load which varied from 2 g to 100 g depending on the size of the particles and their hardness.

Meyer³¹ found that a relationship existed between indentation load and projected area of impression and proposed an empirical relation between the load and size of indentation. While Meyer's analysis was derived from spherical indenters the concepts may be applied to pyramidal indenters using the diagonal of indentation rather than the diameter of a spherical indentation as the measured parameter. Thus the relation:

$$P = a d^n \quad \text{-----} \quad 2.$$

where P = load (g)

d = indentation diagonal (μm)

n = Meyer exponent related to strain
hardening of metal

* See section 4.5.5.

was utilised where a log-log plot of load versus indentation diagonal produced satisfactory linear relationships for the powders tested. The slope of the Meyer line indicated the value of the exponent n . Although Meyer's analysis has been disputed elsewhere³², it has been found to apply over a limited range of loads; if the load is too small, the deformation around the indentation is not fully plastic and the above relationship is not obeyed.

4.5. Compact Measurements

4.5.1. Preparation

In view of the rather restricted length of compact that could be compacted, particularly with the 'monobloc' die-set, Latex rubber bags, as described in Section 4.2.3. were cut to an approximate length of 2 in. loosely filled with powder and gently tapped to ensure maximum filling. Small rubber bungs slightly oversize in diameter were lightly smeared with liquid Latex and fitted to the mouth of the bag to give a good tight fit. A final dip into liquid Latex of the stoppered end followed by curing ensured perfect sealing and air-tightness. After compaction the bag was removed from either the P.V.C. or glycerol pressure media, cleaned and slit to expose the powder compact (c.f. Figure 10). In some cases where the bag had adhered to the compact, this was carefully scraped off leaving a compact of less than 2 in. in length and approximately 0.40 in. in diameter.

4.5.2. De-airing Procedure

Removal of entrapped air within the powder body before compaction was carried out using a technique based upon that of van Buren and Hirsch³³. Before stoppering the bag a small wad of cotton wool was placed on top of the powder fill, otherwise the final sealing procedure was the same as that described above. A thin hypodermic needle connected to the syringe was carefully pressed into the stopper so as to just penetrate into the cotton wool. By connecting the upper end of the syringe with rubber tubing to a vacuum pump and pumping for ten minutes, removal of the entrapped air was effected to a sufficient degree which eliminated all 'loose' flexibility of the bag and compact. Withdrawal of the hypodermic needle automatically sealed the bag from ingress of air. However as a safety precaution prior to assembly of the bags either within the P.V.C. tooling or for immediate compaction in the compound die-set, the evacuated compacts were temporarily immersed in a beaker filled with the glycerol - ethylene glycol medium. This ensured that no air would re-enter the bag, the viscosity of the glycerol fluid being too high for any liquid internal contamination of the powder.

4.5.3. Green Density Measurements

Due to the irregularities in the shapes of the isostatically pressed compacts arising from stretching of the neck of the bag by the rubber stopper and small "flats" made on the bag prior to compaction in the process of handling, conventional methods of density measurements (i.e. direct weight and geometrical measurements of volume) could not be adopted.

Displacement techniques as verified by Nelson and Becker³⁴ can be extremely accurate for non-porous solids, but with powder compacts problems can arise due to absorption of the liquid during immersion. While an impregnation technique, based upon immersing the specimens in a saturated solution of polystyrene in acetone, was found to be technically satisfactory it was considered unsuitable for the measurement of several hundred densities. The mercury displacement method was therefore chosen as most viable. Stanworth³⁵ modified this technique using a displacement cell comprising one arm of a U - tube, where the equivalent change in the level of the mercury in the other limb was measured by a capacitance technique using a modified De Sauty bridge. This method contained certain advantages but its accuracy of approximately 1% was not considered sufficiently high.

The availability of accurate top-pan balances enabled the construction of a pycnometer illustrated in Figure 18 and based upon Archimedes' Principle. Since the specimens would tend to float on top of the mercury it was necessary to provide a stirrup sinker. The depth of immersion of the sinker was maintained constant by a fixed piece of platinum wire which made electrical contact with the mercury at the specified depth of immersion of the sample. A bulb connected to a battery through a simple circuit via the platinum wire, specimen and stirrup, and mercury indicated when contact was made.

The pycnometer was calibrated using polished and degreased steel balls of various diameters with the value for the density of mercury adjusted from the published value of 13.546 at 20°C, to that depending upon room temperature which varied slightly

above and below 20°C. Table 3 relates the calculated versus measured volumes for the steel balls tested. Slight negative errors were obtained in most cases which did not exceed 0.77%. The error tended to be smaller for the larger balls. Appendix 3 outlines the procedure for calculating the densities.

Although this technique is generally only applicable to metals which do not form amalgams with mercury it was found possible to measure compact densities of all the metal powders including copper. The immersion time of a few seconds was not sufficient to produce any reaction with the mercury.

Density measurements obtained from the die-pressed tensile test-piece compacts were derived from direct measurements of the thicknesses, which were uniform over the constant area of pressing (1 sq.in.) for each test-piece.

4.5.4. Fracture Surface Examination

Assessment of densification mechanisms and of compaction of powders generally has been greatly assisted by the use of the Scanning Electron Microscope. Since a green compact has relatively little strength it was considered that a clean fracture surface would offer the most meaningful indication of powder behaviour and deformation during densification. It would be expected that the fracture path would follow the line of least resistance which, in the case of green compacts, would be via the pores and inter-particle contact points and areas. Accordingly, compacts whose geometries were essentially cylindrical were carefully fractured to produce fracture surfaces, whose planes of fracture were approximately normal to the longitudinal direction of the cylinder. This was achieved

by lightly gripping the compact in a vice with soft metal jaws and applying a small impact blow to the end projecting from the vice. Samples were then sawn off to the size of a small tablet acceptable for examination in a Cambridge Stereoscan 2A Scanning Electron Microscope. Photographs were taken at various magnifications to illustrate specific features such as particle flattening and pore geometry over the range of compacting pressures utilised.

4.5.5. Procedure for metallographic examination

The purpose of this area of compact assessment was to prepare a polished planar surface from which (two-dimensionally), could be established:

- (a) a further indication of particle flattening,
- (b) the extent to which work hardening had taken place by micro-hardness tests over the particle - particle contact areas,
- (c) a quantitative measurement of the pore size distribution and extent of pore closure by an Image Analysing Computer.

Normal metallographic preparation techniques involving cutting and grinding would be invalid on green pressed powder metal compacts due to the ease by which powder particles would be dragged or plucked out of the compact during grinding. A simplified method of impregnation, based upon one used by Bockstiegel¹⁶, was used to seal and 'cement' the powder particles during metallographic preparation.

Corresponding halves of the compacts previously fractured, were lightly sectioned near to the fracture face, to provide

suitably sized samples for subsequent mounting and polishing, and then impregnated with an epoxy resin under vacuum. The resin chosen was a CIBA GEIGY Araldite system CY219: HY219 in a 2:1 weight ratio with up to 5% of accelerator DY219 added. This system has a room temperature viscosity of 7 - 12P and low exotherm, suitable for casting and encapsulation. A vacuum dessicator was modified to include a small two-level super-structure consisting of an upper ledge, on which were placed the specimens, and a lower platform containing a small beaker of the resin mix, which was pre-warmed slightly in order to reduce viscosity further but without causing premature setting. The arrangement is illustrated in Figure 19. The dessicator was connected to a vacuum pump and after 20 minutes pumping to de-aerate both specimens and resin at a pressure of approximately 1.3 Nm^{-2} , the dessicator was tilted to allow the specimens to slide off the ledge into the beaker of resin. Pumping was carried out for a further 20 minutes when the vacuum was released, the specimens extracted and curing allowed to proceed at room temperature for a period not less than 48 hours. After curing the specimens were mounted in the same epoxy resin system in which the original fracture face, considered to be that which would allow optimum penetration and impregnation of the resin, was presented for subsequent grinding and polishing. This was carried out as described in section 4.4.6.

4.5.6. Metallographic examination

Examination and photomicrography of the compact structures were carried out using both a Vickers Projection and a Reichart MeF2 microscope. Some assistance in establishing the boundary

conditions between particles, i.e. the interparticle junction was obtained by etching some of the powder compacts using conventional micro-etching reagents.

4.5.7. Work-Hardening Assessment

The extent to which particle work-hardening had occurred was evaluated by carrying out micro-hardness traverses across the sectioned faces where obvious, and wherever possible maximum interparticle contact had been made. Since a certain amount of particle flattening would occur at these contact areas it was important that in traversing the contact area in a two dimensional plane, sufficient particulate material would be present beneath the apparent junction of the particles to support the micro-hardness diamond indenter without leading to erroneous results. This possibility would be offset slightly by the added advantage of having infiltrated and cured resin in any pore space surrounding such limited material to act as a reinforcement medium during the actual deformation caused by the diamond indentations taken adjacent to the contact area. The procedure adopted was that basically described in section 4.4.6. except that indenting loads corresponding to an indentation diagonal of approximately 15 μ m produced on the loose powder particles were used.

4.5.8. Pore Size Distribution

Use of an automatic image analysing computer (Model : Quantimet B) was made initially on one selected powder (Nickel). The use and application of this instrument in the field of Materials Technology has already been described³⁶⁻³⁸. The

procedure adopted for examination of the Nickel compacts is that described separately⁶ and need not be repeated here. More recent work³⁹ however has thrown some doubt onto the viability and usage of this type of instrument for assessing pore-size distribution, and no further work was undertaken with the Quantimet B pending further analysis and interpretation of such findings.

4.5.9. Tensile Strength

Despite the expected overall low tensile strength and possible stress-concentration effects that could arise at the "shoulders" of the die-compacted tensile test-pieces, testing was carried out satisfactorily in standard Hounsfield Tensometer "Quick-Grip" chucks. A Hounsfield Tensometer machine manually operated with 250 lb. and 500 lb. beams was used to fracture the specimens.

5. RESULTS AND ANALYSES

5.1. Particle Characteristics

Full data for the characteristics of the seven powders investigated are given in Tables 4 - 10. While it is not the purpose here to analyse, in depth, 'loose' particulate behaviour per se, some general features and patterns of behaviour emerge from these data which have significance in the interpretation of the data associated with the green compacts produced from each powder type.

5.1.1. Flow Rate

Rapid flow rates are ideal characteristics of powders, since these lead to rapid transfer of powder from storage

containers to dies and within dies too.

In assessing flow rates with the Hall Flowmeter it is well known that very fine powders do not flow through the relatively small orifice due to the relatively rapid increase in specific surface area as particle size becomes very small.

The actual mass flow rate Q of a powder through an orifice is given by:

$$Q = \rho_a u \quad \text{-----} \quad 3$$

where ρ_a = apparent density

u = average particle velocity
passing through orifice.

Since by definition

$$Q = \frac{w}{At} \quad \text{-----} \quad 4$$

where w = total weight of powder flowing

A = cross-section of the orifice

t = total time for powder flow

Then

$$\rho_a = \frac{w}{Au.t} \quad \text{-----} \quad 5$$

This relationship for the seven powders tested is illustrated graphically in Figure 20. Although other factors including general effects of size, shape, surface roughness have contributory effects to flow rate the most important factor determining flow time is the apparent density of the powder. It can be seen from Tables 4 - 10 that flow rates generally increase with particle regularity and that flow is encouraged more in powders whose ratios of apparent density to true density : expressed as relative apparent density are higher.

5.1.2. Apparent Density

This property, like flow rate, is important because it determines the actual volume occupied by a mass of powder which ultimately determines the size of the compaction tooling and level of press motions necessary to densify the loose powder. Generally the apparent density depends on the density of the solid material, particle shape, size, surface area, and distribution, all of which affect how the particles are packed or arranged. Usually the relative apparent density falls within the range 0.2 - 0.5.

Naturally increasing the density of the solid material increases the apparent density of the powder. Similarly decreasing surface roughness and surface area to volume ratios reduce frictional forces between the settling particles and increase the apparent density by allowing the particles to fill more effectively the available free space.

Examination of Tables 4 - 10 confirms these features. Those powders having shape characteristics approaching rounded spherical particle form and of highest true density e.g. A.C.P. and S.G. nickel powders have the highest apparent densities. Powders of more irregular and/or lower true density have the lowest apparent densities. In addition all the powders, except stainless steel, exhibit decreasing apparent density with decreasing particle size. The only explanation offered for the stainless steel powder's reverse trend is one involving the formation of a relatively thin oxide film (as opposed to the expected thicker oxide film on the other powders) possessing a much lower coefficient of friction between particles.

In addition, although classed as "irregular", the surface features of the stainless steel powder are very smooth and rounded.

5.1.3. Tap Density

Should a loose powder be mechanically vibrated or tapped, the density of the powder mass increases from that of the apparent density. This is due to the induced movement of the particles causing them to separate and eventually settle to increase the packing fraction.

Hausner^{40,41} has reviewed factors which cause friction in a powder mass and has proposed an index, defined as the ratio of tap to apparent density, referred to as the "Hausner friction index." He reasoned that high friction between the particles results in low apparent density and that on tapping when the particles momentarily lose contact with each other no friction exists before the particles re-arrange themselves. Table 11 presents ratios of apparent to tap density for all the powders tested, when it can be seen that the ratio increases with increasing particle irregularity. In addition, as the specific surface area increases, i.e. as the particle size decreases the ratio tends to increase to quite high levels for some powders.

It is evident that the ratio of tap to apparent density is as much dependent on surface irregularity as the actual friction forces existing between powder particles. It has been shown elsewhere⁵ that the value of tap density, as a

means of interpreting particle behaviour during re-arrangement processes is not altogether satisfactory. However, the Hausner ratio for routine control tests could usefully serve as a guide to the degree of particle irregularity among powders.

5.1.4. Surface Area

The exact nature of a particle's surface topography will influence the frictional forces between particles. This aspect is important again, in bulk movement of powders and also during the establishment of inter-particle contact areas during compaction and eventual sintering. Although the powders were sieved to specific fractions the nature of the sieve aperture does not take into account whether a particle has an elongated axis relative to its overall shape, i.e. is cigar-shaped. Assuming that each powder type possessed perfect sphericity one could relate the resulting minimal specific surface area to particle size by the relationship

$$S.A. = \frac{6}{\rho_t} \cdot \frac{1}{d} \text{ ----- } 6$$

where SA = specific surface area

ρ_t = true density of powder

d = diameter of spherical particle.

Figures 21-23 present this "ideal" relationship and compare it with the actual trends for the seven powders tested. The S.E.M. illustrations (Figures 1-7) show that the A.C.P. is the most spherical and the actual values of S.A. do parallel very closely to the ideal line. Nickel with a very similar true density to

copper also parallels closely the ideal line, but this powder, although rounded in overall form, has small surface protuberances which cause greater divergence from ideality. While the axiality of the electrolytic copper powder particles is fairly uniform the dendritic nature of this powder creates a significant divergence from sphericity. This feature is also exhibited by the sponge iron powder, which has been produced by the chemical reduction method. Conversely the atomised aluminium, zinc and stainless steel powders all have smoother surfaces but dimensionally irregular features. This would suggest that packing fractions would be less dependent on frictional effects and more dependent on geometrical factors, and it is significant that these powders have the lowest flow rates, i.e. longer times of flow.

5.1.5. Meyer Hardness

The Meyer analysis was carried out on a $-125 + 106 \mu\text{m}$ size fraction on all the powders except the electrolytic copper powder which was assessed only for its intrinsic hardness. This was due to the extreme irregularity of this powder which prevented reliable hardness indentations to be recorded over the indentation load range used for the other powders.

The results are given in Table 12 and the log-log relationships between load and indentation diagonal are presented in Fig. 24. From Fig. 24 the Meyer exponent n was derived which is given in Table 12 along with each powder's microhardness assessed from an indentation diagonal of $15 \mu\text{m}$ corresponding to approximately 120 micro divisions on the microhardness ocular scale.

5.2. Pressure-Density Relationships

A knowledge of the relationship between compacting pressure and density is important because pressure, more than any other factor, largely controls the attainment of high density, high strength and low porosity in green compacts and markedly influences the same properties in the sintered product. Stemming from the need to predict a pressure for a given level of density, many empirical formulae have been proposed¹⁶ to describe the pressure-porosity or pressure-densification relationship, most of which are limited to specific ranges of pressure. Certain aspects regarding the desirability of relating density versus compacting pressure in linear form, in order to represent various stages or mechanisms of compaction have been discussed elsewhere^{4,9}. Despite the majority of equations that have been proposed they all reduce to about four basic types of which there are several sub-types or modified forms.

An analysis of the literature concerning these equations shows little fundamental consideration underlying their formulation. Two equations have been widely used in compaction studies on metallic and non-metallic systems : that of Kawakita⁴² and that proposed by Shapiro and Kolthoff⁴³ and independently by Konopicky⁴⁴.

The former equation applicable over a wide range of particulate matter relates the relative reduction in volume of the powder mass to pressure thus:

$$C = \frac{V_o - V_g}{V_o} = 1 - \frac{D_a}{D_g} = \frac{abP}{1+bP} \quad \text{-----} \quad 7$$

where C = relative reduction in volume

V_o = initial volume of the powder mass

V_g = volume of powder mass under
pressure P

D_a = relative apparent density of initial
loose powder mass

D_g = relative density of powder mass
under pressure P.

a, b = constants

Re-arranging the equation gives

$$\frac{P}{C} = \frac{1}{ab} + \frac{P}{a} \quad \text{-----} \quad 8$$

Equation 7 can be arrived at by considering that the change in porosity with pressure could be proportional to some power of the porosity:

$$\frac{-d(\text{porosity})}{dP} = k(\text{porosity})^x \quad \text{-----} \quad 9$$

where k and x are constants.

If work-hardening is considered to be the operative mechanism in affecting pore-closure and considering that under the effect of load the pore closure rate may take the parabolic form with porosity, similar to the work hardening rate expressed by the square of n in the Meyer formula.

Thus putting x = 2 allows equation 9 to be written:

$$\frac{-d(1-D)}{dP} = k(1-D)^2 \quad \text{-----} \quad 10$$

where D = relative green density.

Equation 10 can be re-arranged and integrated between density limits corresponding to those for loose powder fill conditions and under pressure conditions:

$$\int_{1-D_g}^{1-D_a} - \frac{d(1-D)}{(1-D)^2} = k \int_{P_0}^P dP \quad \text{-----} \quad 11$$

where $1-D_a$ = initial porosity i.e. at
zero pressure P_0

$1-D_g$ = porosity at pressure P

$$\text{This gives } \frac{1}{1-D_g} - \frac{1}{1-D_a} = kP \quad \text{-----} \quad 12$$

$$\text{or } \frac{D_g - D_a}{(1-D_g)(1-D_a)} = KP \quad \text{-----} \quad 13$$

$$\text{From equation 7 where } C = \frac{V_0 - V}{V_0} = \frac{D_g - D_a}{D_g}$$

allows equation 13 to be re-written :

$$\frac{CD_g}{(1-D_g)(1-D_a)} = KP \quad \text{-----} \quad 14$$

Let $1-D_a = a$

$$\text{Then } \frac{CD_g}{1-D_g} = aKP \quad \text{-----} \quad 15$$

From equation 12 and re-arranging

$$D_g = \frac{(1-a) + a.k.P}{1 + a.kP} \quad \text{-----} \quad 16$$

and substituting in equation 15

$$\text{gives } C = \frac{a^2 kP}{(1-a)(1+\frac{a}{1-a}) k.P} \text{ ----- } 17$$

$$\text{Let } b = \frac{ak}{1-a}$$

$$\text{Then } C = \frac{ab P}{1 + bP} \quad \text{which is the original form of}$$

Kawakita's equation

The application of the Kawakita equation of state to the various powders tested is given later.

The second equation of state which is similarly analysed. is essentially a first order differential equation. Heckel⁴⁵ considered the "kinetics" of densification and likened the process to a first order chemical reaction in which the change of density with pressure is proportional to the pore fraction,

$$\text{i.e. } \frac{dD}{dP} = K (1-D_g) \text{ ----- } 18$$

where $K = \text{constant}$

$D_g = \text{relative density under}$
pressure P .

Integrating each side between relative apparent density D_a and D_g gives :

$$\int_{D_a}^{D_g} \frac{dD}{1-D} = K \int_{P_0}^P dP \text{ ----- } 19$$

$$\text{i.e. } \ln \frac{1}{1-D_g} = KP + \ln \frac{1}{1-D_a} \text{ ----- } 20$$

$$\text{or } \ln \frac{1}{1-D_g} = KP + A \quad \text{-----} \quad 21$$

$$\text{where } A = \ln \frac{1}{1-D_a}$$

Equation 21 has been widely used where the constants K and A have been interpreted in terms of work-hardening and the point at which densification actually commences under pressure.

Initial analysis of the Kawakita and Konopicky-Shapiro equations of state has already been made^{8,9} and need not be repeated here. However a fuller analysis will be made later and related to the work-hardening undergone by the particles, as revealed by S.E.M. and micro-hardness measurements.

5.3. Isostatic Compaction Studies

5.3.1. Effect of Particle Size

Data for relative green densities of three powders, having different particle geometries, pressed over a pressure range from 5 - 50 tsi have already been published⁵. The density - pressure relationships plotted on linear axes are given in Figures 25-27 from which it can be seen that the effect of particle size is quite small on overall densification. While the nickel and electrolytic copper powders exhibit a slight diminution of densification level as the particle size decreases, the iron powder, conversely, exhibits a slight increase. This is more noticeable over the higher pressure regions and is considered to be due to the more restricted particle movement which occurs at higher pressures. Also since the iron powder possesses a high degree of internal porosity, classified as

intra-particle porosity, which would be expected to disappear through cold welding at higher pressures, this should contribute to a more effective overall porosity reduction with pressure. Thus finer particles with a higher specific surface area would compress to solid particles in addition to external work-hardening effects and the elimination of inter particle porosity as pressure increases.

In the presentation of the data given in a separate paper⁸, particle size was also found to have a minimal effect on densification level with pressure. (Table 13 and Figure 28).

5.3.2. Effect of Particle Geometry

A comparison between powders of different particle geometry was limited to that of copper which was available in the atomised and electrolytic forms. Table 13 presents values of relative green densities for the atomised and electrolytic forms over a pressure range 0 - 0.42 KN.mm⁻² (0 - 27 tsi) in various size fractions. Figure 28 presents four size fractions from these data graphically, as density versus pressure where the irregularity of the E.C.P. is reflected in the overall lower densification levels, compared to that of the regular and near-spherical form of the A.C.P. Although figure 28 presents only a qualitative comparison between copper powder in only two geometric forms of similar hardness, a more quantitative approach utilising the Kawakita and Konopicky equations of state is presented in section 5.3.6.

5.3.3. Effect of De-Airing

Considerable interest has been shown, particularly in the ceramics industry, towards evacuating the air from within the powder body, which would normally be sealed within the flexible bag or tool. This has had some success in reducing "back-pressures" set up within air-pockets created between the compact surface and inner surface of the tool, which has led to cracking in parts of complex geometry. Relatively little reported work⁹ on the de-airing of metal powder compacts has been made which justified the inclusion of this area of investigation in these studies.

Particle size fractions of $-125 + 106 \mu\text{m}$ from each powder, except the electrolytic copper were isostatically pressed in the non-de-aired and de-aired conditions. The latter condition was prepared as described in section 4.5.2. Table 14 presents the data for relative green densities in these two conditions obtained over a pressing pressure range 5 - 50 t.s.i. for the six powders tested, while Figures 29-34 illustrate these density-pressure comparisons graphically on linear axes.

Certain features become evident from these results. Firstly, the effect of de-airing the powders is generally minimal insofar as affecting the level of green density for a given compacting pressure. Some increases in relative green density have resulted in de-airing the nickel, stainless steel, iron and zinc powders particularly for lower compacting pressures, while a more negative effect, i.e. lower densification

has resulted for equivalent compacting pressures for the A.C.P. and aluminium powders. This latter effect has also been reported⁸ for E.C.P. Only in the A.C.P. does the density-pressure relationship for the de-aired condition parallel very closely that for the non-de-aired condition. In all the other powders the density levels were more variable though for the aluminium, stainless steel, and zinc powders the difference in density was much less for higher compacting pressures.

Possibly the principal effect of de-airing the powder body is to restrict interparticle movement, since by withdrawing the air the effect is to initially stiffen the bagged compact which may promote some mechanical interlocking. This view is partly supported by the fact that those powders whose surface features are more topographically irregular become more interlocked together under the early stages of compaction. However, no specific conclusions can be drawn from this area; the advantages of de-airing are probably more related to external compact geometry rather than particle characteristics and particle geometry.

The second feature evident from Figures 29-34 are the high relative green densities obtained in the aluminium and zinc powders. Furthermore there seems to be a pressure range over which no further increase in densification occurs. This range could well correspond to the stage of bulk compression where no further inter-particle movement is possible and the whole powder body becomes truly subjected to hydrostatic pressure

resulting in elastic compression only. The aluminium and zinc are the two softest powders, despite their work-hardening capabilities which are comparable in some cases to the other powders. The A.C.P. and sponge iron of intermediate hardnesses are also approaching the stage of bulk compression at 50 t.s.i. compacting pressure, while for the nickel and stainless steel, which have the relative highest particle micro-hardnesses, densification is still increasing even at 50 t.s.i. though at decreasing rates. Clearly the pressure levels over which bulk compression occurs are influenced by the particle micro-hardness; the work-hardening and particle flattening during densification is discussed later.

5.3.4. Applicability of Kawakita's equation of state

From the data given in Tables 4 - 10, 13 and 14, values for the compression ratio C were evaluated for the various powders under pressure P and the ratio of P/C plotted versus compacting pressure according to equation 8. Figure 35 illustrates the relationship for the nickel, atomised copper, aluminium, stainless steel, iron and zinc powders while Figures 36-38 present the relationships for various size fractions of the nickel, iron and electrolytic copper powders. Additional data for the atomized and electrolytic copper powders, which were directly compared to each other have been published separately⁸. All these graphs indicate very good linearity over a wide compacting pressure range. The slope of each graph is given by $1/a$ while the intercept at zero pressure on the P/C axis, although mathematically indeterminate

is given by $1/ab$.

5.3.5. Applicability of Konopicky-Shapiro equation of state

The applicability of this equation of state has already been described^{5,6,8}. However a similar appraisal to that described above (section 5.3.4) was made and Figure 39 illustrates plots of $\ln 1/(1-D_g)$ versus compacting pressure for nickel, atomised copper, aluminium, stainless steel, iron and zinc, while Figures 40 - 42 illustrate similar plots for various size fractions of the nickel, iron and electrolytic copper powders.

It is very evident that linearity is limited to very specific ranges of pressure with distinct changes of slope occurring for the different plots according to the type of powder. In nearly every case, the linearity does not extend to low compacting pressures rather the linearity is more evident over pressure ranges from approximately 10-25 t.s.i. and 25-45 t.s.i. with a change of slope occurring around 25 t.s.i. These slopes corresponding to the constant K in equation 21 were measured along with intercepts A, on the $\ln 1/(1-D_g)$ axis at zero pressure where possible.

5.3.6. Interpretation of the constants in the Kawakita and Konopicky - Shapiro equations of state.

Within the overall scope of the experimental work an initial attempt was made to interpret the constants in terms of interparticle friction⁵ in isostatically pressed compacts.

In this area of particle behaviour, considered to be most relevant in the earlier stages of compaction it was found that interparticle friction could be interpreted better from a ratio of D_A/D_a , where D_A is the extrapolated value of relative density at zero pressure using the Konopicky - Shapiro equation of state, and D_a , the relative apparent density. This was established from the data resulting from compacting various size fractions of the S.G. nickel, iron and electrolytic copper powders. In view of the expected sliding and re-arrangement that occurs between particles at low pressures it was proposed that, if a value of density can be derived from zero pressure, e.g. by extrapolating a suitable (linear) density - pressure relationship which may deviate from linearity at low pressures due to restacking, then that value of density will more fully account for friction conditions than that of tap density D_t , utilised in the Hausner^{40, 41} friction index: D_t/D_a .

Since the Kawakita relationship involves the pressure function in the ordinate and simultaneously is mathematically indeterminate at zero pressure it was contended that only the Konopicky-Shapiro constant A , allowing a relative density D_A ($D_A > D_t > D_a$) to be measured at zero porosity, would be more meaningful. Figure 43 illustrates, typically, this concept which allowed values of D_A to be obtained from such plots represented by Figures 40-42. Although equation 21 specifies the constant $A = \ln \frac{1}{1-D_a}$, the intercept at zero pressure gives a displaced value of $\ln \frac{1}{1-D_a}$ where the displacement B (Figure 43) is given by

$$B = A^1 - \ln \frac{1}{1-D_a} \text{-----} \quad 22$$

$$\text{where } A^1 = \ln \frac{1}{1-D_A}$$

and the density contribution D_B from individual particle movement and re-arrangement, is given by

$$D_B = D_A - D_a \text{-----} \quad 23$$

Hausner claimed that dividing the compressing ratio D_g/D_a by his friction index D_t/D_a giving a final ratio D_g/D_t , was a constant for a series of size fractions of electrolytic iron powder though this was later disputed by Sheinberg⁴⁵. No such constancy was obtained from the pressed size fractions of the three powders tested; however dividing the compression ratio by the "new" index D_A/D_a gave improved constancy irrespective of particle size and compacting pressure and in this respect the index D_A/D_a is therefore considered to be an improvement on the index D_t/D_a originally proposed by Hausner for characterising friction between powder particles.

Beyond the transitional restacking stage where the majority of particle movement is expected to occur, the particles will then undergo successive elastic and then plastic deformation with local work-hardening occurring at the particle-particle interfaces depending on the strain-hardening capacity of the material. The distinct changes of slope featured in the Konopicky-Shapiro plots suggest that these densification rates are associated with specific mechanisms of compaction. No such changes of slope were revealed in the Kawakita plots.

The generally accepted interpretation of the Kawakita constants regards a , i.e. the reciprocal of the slope factor, as a measure of the initial (loose) porosity of the powder, while the constant b is regarded as a "coefficient of compression" related to the plasticity of the powder. The slope factor K of the Konopicky-Shapiro relationship has often been regarded as a useful means of expressing densification, though this has been challenged by other workers⁴⁷.

The outcome following compaction of the nickel, iron and electrolytic copper powders showed that for the two slope factors K : designated K_1 and K_2 for the intermediate and higher pressure ranges, respectively, the values of K_1 showed little variation within each fraction of powder tested. K_1 had the highest values for the electrolytic copper and the lowest for the nickel powder. More variability in the values of K_2 were obtained within each of the three powders though again the values in themselves increased generally with increasing particle irregularity.

A later study⁸ compared the isostatic compaction behaviour of the two copper powders which have similar micro-hardnesses but different shape characteristics. In that study emphasis was directed upon the effect of particle geometry, with material features, deformability etc. regarded as essentially constant. The results of an independent study on the compaction behaviour of pharmaceutical powders by Hersey et al⁴⁸ who established values of $1/b$ derived from Kawakita's relationship, led them to conclude that this reciprocal value

indicated a measure of consolidation irrespective of the mechanism of compaction. Hersey et al found that $1/b$ values were lower for more brittle powders and higher for powders which deformed more by plastic deformation. In the comparison between the A.C.P. and E.C.P. $1/b$ values for the A.C.P. were almost twice those for the E.C.P. the values generally being independent of particle size. This indicated that $1/b$ values and hence the constant b are more dependent on particle geometry rather than on work-hardening capacity. The fact that both powders were expected to respond similarly to plastic deformation and/or flow was revealed in the similarity of the K_1 slope factors. [N.B. since this study involved only an intermediate pressure range of 0-27 t.s.i only the first stage Konopicky-Shapiro slope factor could be compared.]

Extending the Kawakita analysis to the S.G. nickel and iron powders represented graphically in Figures 36 and 37 and including the comparison between the two copper powders (the electrolytic powder being more extensively surveyed as in Figure 38), Table 15 summarises values of a , $1 - D_a$ (i.e. initial porosity), and $1/b$ for the various size fractions tested. Similar values for the mono size fractions of the remaining powders: stainless steel, aluminium and zinc are presented in Table 16.

From these data it can be seen that the a values are in good agreement with the $1 - D_a$ values, representing initial loose porosity, for all powders including the different size

fractions tested. However, no correlation could be made between $1/b$ values and any powder property, which included surface area, flow rate, micro-hardness or work-hardening capacity. Significantly $1/b$ values were the lowest in the two softest powders aluminium and zinc and highest for the nickel which had the least work-hardening capacity and representing one of the harder powders. These values are at some variance with Hersey et al's results and it would seem that particle geometry may probably influence the magnitude of the $1/b$ values as much as the ability of the particles to deform.

Heckel⁴⁵ had originally proposed that the Konopicky-Shapiro slope factor K was inversely proportional to the material's yield stress while Greenspan⁴⁹, who confirmed that a change of slope occurred at very high pressures, utilizing the Konopicky-Shapiro relationship, suggested that in this region particle strain-hardening was more dominant. Thus the measured slope factors K_1 and K_2 of a monosize fraction (125-106 μ m) of all the powders tested were related to published values^{50, 51} of yield stress and the measured Meyer index n . These values are all summarised in Table 17. [N.B. the yield stress value for S.G. nickel was derived from a consideration of the stress-strain behaviour of a number of high nickel alloys whose alloy content approximated to that in S.G. nickel].

Good linear correlations between K_1 and the reciprocal of yield stress ρ_0 , and between K_2 and the Meyer index of work hardening n were obtained as evident in Figures 44 and 45.

The least squares fitting of a line to the data in Figure 44 gave the relationship

$$K_1 = 0.015 + 0.177 \frac{1}{\rho_0} \text{ ----- } 24$$

indicating support to Heckel's proposal that K_1 is proportional to $1/\rho_0$. Thus for materials of relatively high yield stress the initial densification rate would not be expected to be very high whereas softer powders should densify easier before significant work-hardening occurs. The correlation between K_2 and the Meyer index n is interesting since powders with the higher n values, associated with higher work-hardening capacity, should enable densification to proceed at a higher rate, i.e. higher K_2 slope factors. This stage of compaction will pre-determine the stage of bulk compression when the capacity for further particle deformation becomes exhausted. Only if particle movement or disturbance at points of geometrical irregularity e.g. caused by fragmentation, occurred would there expected to be any further plastic deformation. The situation with the zinc and aluminium powders is that these two materials have relatively low flow stresses with the zinc having the greater capacity for work-hardening. Thus both powders have initially densified at the same rate, but the zinc has densified over the higher compacting pressure range (15 - 30 t.s.i.) at a higher rate than for aluminium. Both materials exhibit similar slope change values, over which densification by particle flattening and then that governed by work-hardening has occurred. Beyond 30 tsi both powders have ceased to densify indicating that the stage of bulk

compression has been reached.

5.3.7. Pore Closure

An attempt to define the stages of compaction in terms of pore closure⁶ was made on isostatically pressed nickel. In terms of availability at the time this powder was selected for having the most uniform particle geometry as well as high flow rate and specific surface area : factors which would least affect any assessment of pore closure through metallographic or S.E.M. studies.

Following compaction of a specific size fraction ($- 150 + 106 \mu\text{m}$) of S.G. nickel over the pressure range 5 - 50 t.s.i. compacts were both fractured for S.E.M. fracture surface examination and prepared for metallographic examination by impregnation, as described in section 4.5. The metallographically prepared samples were then examined both optically and by Quantimet and subject to micro-hardness measurements; full details concerning the experimental technique and data will not be repeated here since these are submitted separately⁶. The main conclusions that emerged from this study of pore closure were that particle work-hardening and flattening was more evident at pressures in excess of 25 t.s.i. While this level of compacting pressure is a little lower than the point at which the Konopicky-Shapiro relationship changes slope ($K_1 \rightarrow K_2$), the above features, together with the degree of closure of larger pores with respect to the finer pores within the compacts suggested that a pressure threshold exists over which deformation modes

undergone by the particles are significantly different. The data from the Quantimet scans of pore size and porosity levels indicate that little variability occurred within the distribution of total porosity fraction for the larger pores over the higher compaction pressure range 25 - 50 t.s.i. Up to 20 - 25 t.s.i. the larger pores underwent continual shrinkage, when variability was limited only to the smaller pores whose fractions of total porosity tended to increase, the other fractions of pore sizes remaining approximately constant.

These results however were not considered fully conclusive to identify fully the compaction stages proposed. Subsequent work carried out on the Quantimet has led to some doubt being cast on the validity of pore size assessment based partly on instrument control and standardisation and secondly on preparation of specimen technique. No further work was thus carried out with the existing Quantimet B.

5.3.8. Fracture Surface Examination

In order to quantify, as accurately as possible, the degree of particle flattening, the following technique was carried out. Each compact's fracture surface, upon examination in the S.E.M., was photographed at five random positions where particle flattening was most evident. The developed negatives were then enlarged on a photographic enlarger to twice the photographed magnification factor and a transparent plastic ruler was then used to measure the diameters of flattened areas. Where the flattened area was circular, albeit presented at an angle which appeared elliptical,

the major axis was recorded as the diameter. Other areas which were not circular but appeared "face-on" or normal to the photographic plane were measured by the use of graph paper and the measured area converted to that of a circle from which an equivalent diameter was derived. From the magnification factor of the negative and that of the enlarged image mean values of diameter, representing the flattened contact areas, were evaluated from the various regions and these are presented in Table 18. Representations of the particle flattening are illustrated in Figures 46-52 which include a comparison of the effects of de-airing on the A.C.P. only. Any effects arising from de-airing would be expected to be more readily apparent on a spherical powder than an irregular one. [N.B. fracture surfaces representing the de-aired A.C.P. compacts pressed at 15 and 45 t.s.i. and stainless steel pressed at 5 t.s.i. were not available for examination due to inadequate specimen availability. This was similarly the case for micro-examination and work-hardening assessments.] Figures 46 - 52 indicate that the more regular powders e.g. the S.G. nickel and A.C.P. and to a certain extent the stainless steel allowed fairly accurate assessments of particle flattening to be made due to the near sphericity of the contact areas. However, assessment of the contact areas for the remaining powders was not so simple and the results, with their slightly higher level of scatter, must be viewed with a lesser degree of accuracy. In the case of the more regular powders the progress of particle flattening can be interpreted from a geometrical

view-point. This concept is taken from the idealised model of hard-spheres in closest contact, often used to describe atomic packing. Initially if all spheres were in closest contact, each sphere would be in contact with 12 nearest neighbours. Assuming that during isostatic pressing the transmission of forces is ideally distributed through each sphere each sphere would become flattened over its surface to a 12 sided polyhedron, becoming a perfect dodecahedron in the ultimate case when all porosity is eliminated. This concept is illustrated in Figure 53 where a sphere or particle of diameter D subjected equally to twelve uniformly distributed force points becomes plastically deformed to a dodecahedron consisting of twelve surfaces, each being a regular pentagon of side a .

The volume of a regular dodecahedron is given ⁵² as $7.6631 a^3$ and the ratio of the inscribing circle diameter d to a as 1.376. Since the volume of the original sphere and the resulting dodecahedron must be the same a simple calculation gives the ratio of $\frac{d}{D}$ as 0.562. Taking a mean particle diameter of the sieved fraction $-125 + 106\mu\text{m}$, as $115.5\mu\text{m}$ gives a maximised value of d as $64.9\mu\text{m}$ (d_m). Thus from a geometrical point of view the progress of densification in terms of particular flattening may be followed by relating the value of d/d_m (which should maximise to unity) with compacting pressure. Extending this concept to the other powders, values of d/d_m are also presented in Table 18 and graphically in Figures 54-59 which include the comparison between A.C.P. in both non de-aired and de-aired conditions.

In every case particle flattening developed extremely rapidly (up to 20 t.s.i. pressure for most powders), then proceeded at a very much reduced rate when beyond 35 t.s.i. for most powders, little or no further flattening occurred. Over this pressure level, the ratio d/d_m approached unity (at very high pressures, occasionally exceeding unity) for the softer powders. For the harder powders, particularly the nickel and stainless steel, the ratio d/d_m tended to maximise to a constant value which was lowest for the stainless steel. It is repeated that due to the irregularity of most of the powders an accurate quantitative analysis of particle flattening proved difficult. This largely arose from the fact that irregular powders could be subjected to quite severe local deformation at "high spots" and irregularities on their surfaces. Only the A.C.P. particles tended to approximate to the dodecahedron model proposed above. However, it is interesting to note that where points of inflexion at slope changes occur in the d/d_m plots, these approximate very closely to the points of inflexion expressed in the Konopicky-Shapiro plots given in Figure 39. This would suggest that particle flattening responsible for the major increase in densification is largely related to densification generally. At relatively high pressures, particle flattening and work-hardening is expected to maximise when the inability to deform further by plastic work-hardening produces the condition of bulk compression. The S.E. Micrographs suggested that where deformation at higher pressures had occurred, particularly on the more

irregular powders only particle flattening had been produced. No evidence was seen of any fragmentation or particle fracturing occurring transgranularly. The appearances of the fractured faces suggested that these were in fact part of the original particle surface and that in fracturing the specimens for examination, fracturing had occurred interparticulary rather than transparticulary. The comparison between the ACP in the non de-aired and de-aired conditions (Figure 55) was interesting since the de-aired state produced an overall reduced degree of flattening which was in accord with the overall reduced density level compared to the non-de-aired state (ref Figure 30).

5.3.9. Metallographic Examination

After impregnation and metallographic preparation, each compact sample was examined optically and then photographed to illustrate, two-dimensionally, the level of porosity and degree of interparticle involvement following different compacting pressures. These are illustrated in Figures 60-66 which parallel the S.E. Micrographs illustrated in Figures 46-52. In order to highlight the interparticle contact regions or "boundaries" some of the compact structures were lightly etched : the A.C.P. in 5% alcoholic ferric chloride and the zinc in 2% nital.

Paralleling the interpretation of the results of the S.E.M. fracture examination, only the more regular i.e. spherical powders have contributed to interparticle flattening (Figures 46-48) whereas the nature of the interparticle

boundary regions in the other pressed powder compacts are more irregular. For the highly compressible aluminium and zinc powders it is difficult to identify the original particulate geometries of the powders. However, for all powders it can be seen that particle flattening has commenced immediately at the lowest compacting pressure (5 t.s.i.) and rapidly developed there-after at higher pressures. The A.C.P. structures have developed rapidly where the inter-particle "triple-points" i.e. the junctions between three neighbouring sectioned particles are all equi-angular to the classic 120° often found in recrystallised polygonised structures. This indicates that only in such spherical powders is the transmission and distribution of forces of an equal nature through original points of contact. In the more irregular powders, e.g. sponge iron, densification and the elimination of porosity must proceed more erratically with particle deformation proceeding in a less uniform manner.

The sequence of photomicrographs typifying the structures produced, following increasing compacting pressures serve to illustrate adequately : firstly the uniformity or otherwise of pore elimination, and secondly the deformation of particles and extent of densification according to the regularity of the particle geometry.

5.3.10 Particle Work-Hardening

Following metallographic examination, micro-hardness impressions were recorded at regions immediately adjacent to inter-particle boundaries in all the impregnated and sectioned

compacts. The indenting loads used were the same as those defining the original particle micro-hardnesses expressed in Table 12. A minimum of 10 readings were obtained from each sample and a mean value calculated. For purposes of comparison with the extent of particle flattening these values of micro-hardness are recorded in Table 18 and graphically in Figures 54 - 59.

It can be seen that in virtually every case particle work-hardening has occurred very rapidly over the lower pressure regions but less so over higher pressure regions. There are similar points of inflexion at pressure levels to that for the plots of d/d_m , which were previously compared to the Konopicky-Shapiro plots of density versus compacting pressure. It is significant to note that over the higher pressure regions, where particle flattening is still progressing then so does work-hardening to a similar degree. Where particle flattening maximises out to a constant level, no further work-hardening occurs indicating that further plastic deformation is very minimal if any, and that deformation or bulk compression is more likely to be elastic, rather than plastic, in nature. The only real exception to this trend of behaviour appears to lie with the S.G. nickel where work-hardening has continued beyond the stage where particle flattening has maximised. No explanation can be offered for this pattern except the possibility of the protuberances being subjected to locally higher pressures initially restricting deformation elsewhere over the particle surface. Such a situation would lead to

a rapid development of flattening and groups of particles allowing deformation to then develop elsewhere at a later stage.

The reduced degree of particle flattening in the de-aired A.C.P. compacts produced a corresponding reduction in particle micro-hardness levels compared to the non de-aired condition, which further establishes the dependence of particle flattening on densification and work-hardening.

5.4. Die Compaction Studies

5.4.1. Green Tensile Strength

Published work concerning the tensile strength of as-pressed metal compacts is minimal; the most recent account has been that of Strömberg et al⁵³ who investigated the effect of interparticle contact area on the strength of a spherical fraction of isostatically cold-pressed aluminium. They concluded that compaction occurred in two stages, where at some high intermediate pressure, interparticle sliding occurred in a way that did not increase densification but made it easier for further deformation to occur. They considered it likely that the pressure at which this sliding took place was dependent on the work-hardening rate of the powder as well as the powder size and morphology. They also concluded that for a given particle size, the strength of the compact σ_t is proportional to the radius cubed of the interparticle boundary, i.e. dependent upon the inter-particle

metallic contacts made during compaction:

$$\sigma_t = K \left\{ \frac{a}{R} \right\}^3 \text{-----} \quad (25)$$

where a = radius of the grain boundary between two spherical particles of radius R

K = a constant $\approx 0.16 E$ in the case of metals,
 E being the modulus of elasticity.

Although this equation was derived on the basis of non-plastically deformed powder, the authors considered the expression should equally well describe the strength of plastically deformed powders. While the elastic properties would not be much affected by deformation, changes in a due to cold pressing will bring about changes in R and vice versa. However, as the compact approaches full density, it is not known whether this inter-dependence between a and R can be strictly maintained. On the model system proposed in section 5.38 the radius a should maximise to a specific value while R should minimise similarly. Thus the equation predicts that under full density the strength should also maximise.

Some doubt must be expressed concerning the viability of interparticle sliding occurring at high pressures, expressed by Strömberg et. al, particularly with a regular spherical-type of powder. No evidence was revealed in S.E.M. photographs as to this effect, though an interesting feature of their work was the inversion of tensile strength with compacting pressure. This is graphically illustrated in Figure 67. This inversion was also confirmed from the

strength value derived from the cube of the radius of contact surface between deformed particles assessed from S.E.M. photomicrographs, using a technique similar to that described in section 5.3.8.

Since zinc has been shown to compact isostatically to 95% of its theoretical density it was decided to investigate if the tensile strength at the relatively high compacting pressures producing this high density was related more to the particle size (than area of contact) according to the well known Petch⁵⁴ relationship:

$$\sigma_c = \sigma_o + Kd^{-\frac{1}{2}} \quad \text{-----} \quad (26)$$

where σ_c = cleavage stress for fracture

d = grain diameter

σ_o and K are constants.

Below a critical grain diameter the law changes to

$$\sigma_c = K^1 d^{-\frac{1}{2}} \quad \text{-----} \quad (27)$$

and crack propagation becomes the critical factor.

Above a critical grain diameter crack nucleation becomes the critical factor.

To test this applicability of Petch's equation sieved fractions of zinc were compacted over the pressure range 5 - 50 t.s.i. but at a reduced number of pressure levels. This was governed mainly by the limited amount of zinc powder obtained from sieving to specific fractions and to allow for at least five compacts to be pressed at each fraction. [The recent availability of the dog-bone die set precluded a full-scale investigation into tensile strength of all the powders

utilised in the isostatic compaction studies, though this is a useful area for further studies.] Accordingly the zinc fractions were compacted at pressure levels of 5, 13, 20, 24, 30, 36, 40 and 50 t.s.i. The density levels were evaluated by direct weighing and measurement, and then pulled to destruction in the Hounsfield tensometer. Table 19 summarizes the mean values of tensile strength σ obtained, along with values of relative green density D_g for each sieved fraction at each of the pressing pressures. Table 19 also includes values of $d^{-\frac{1}{2}}$ derived from a mean value of particle size within the limits of the sieved fractions.

The relationships between tensile strength and density with compacting pressure for the various fractions are presented graphically in Figures 68-74 similar to that in Figure 67 and includes the degree of scatter arising in measured tensile strength as each compacting pressure. Additionally Figure 68 includes the density variation of the -125+106 μ m size fraction compacted isostatically. It can be seen from these results that there is, in fact, an inversion in tensile strength with compacting pressure mainly for the coarse and intermediate size fractions. The two fractions -45+38 μ m and -38+00 μ m do not exhibit any inversion though a slight break exists in the tensile strength - compacting pressure relationship in the former fraction. A fair degree of scatter occurred in the tensile values particularly at the higher compacting pressures. However, density values were remarkably consistent, the trend of values showing a slight reduction in level with increasing fineness of particle size over the entire compacting pressure range. Thus it is

evident that zinc at least, follows a similar pattern of behaviour to that of aluminium reported by Strömberg et al. However, in view of the irregular nature of the zinc powder it was decided not to subject the fracture surfaces of the tensile test pieces for S.E.M. examination since it was expected that an even greater degree of scatter in measurements for particle flattening would occur in the die-pressed samples compared to the $-125+106\mu\text{m}$ fraction pressed isostatically and previously examined by S.E.M. An attempt to relate the tensile strength with the cube of the radii of contact areas, expressed in Table 18, i.e. $(d/2)^3$ did not produce any linear relationship as found by Strömberg et. al. for their studies on aluminium.

Converting the dimensions of mean particle diameter from μm to in. and plotting the tensile strength of compacts pressed to the maximum compacting pressure of 50 t.s.i. versus (particle diameter) $^{-1/2}$ according to the Petch equation, produced a relationship illustrated in Figure 75. This would indicate that the linearity, over specific ranges of particle size, supports the proposal that tensile strength is a function of particle size. However, this is only approximating towards the ideal relationship, since at lower compacting pressures further divergence from linearity occurred with erratic relationship existing over the complete range of compacting pressure. The nature of the interparticle bonding forces and levels of strength developed there are clearly a function of compacting pressure since the tensile strength continues to increase beyond the point where density maximises (ref Figures 68-74). This indicates that tensile strength is

not a function of interparticle contact area, at least beyond the point where interparticle contact area itself maximises as previously established. Rather strength must be more influenced by the nature of the bonding forces between particles and this in turn can be affected by a number of factors e.g. oxide films, adsorbed layers etc.

The inversion in strength is more difficult to explain though is unlikely to be influenced by particle re-arrangement. Such a mechanism might be envisaged to occur in highly irregular powders where bridging and vaulting developed at intermediate compacting pressure levels, may suddenly collapse under higher pressures causing particle movement . . . In the case of aluminium and zinc both of which are relatively soft powders, a more likely explanation for the inversion is the fact that beyond a certain critical pressure the particles become highly work-hardened and that any increases in contact area and/or pressure application are counterbalanced by a relaxation mechanism due to large residual stresses. This could take place over a very shallow region of the particle surface leading to a reduction in strength without affecting the density. It is interesting to note that for the isostatic and die-pressed -125+106 μ m fraction the level of compacting pressure where work-hardening maximises, coincides with the pressure level where the inversion occurs in tensile strength namely 25 t.s.i. Clearly this is a region open for further useful study, preferably on isostatically pressed tensile specimens rather than on specimens die-pressed; the latter technique being accountable for the degree of scatter in results arising from die-wall friction effects and non-uniformity of distribution of compacting pressure.

5.4.2. Glass Ceramic Die Inserts

The philosophy behind this area of investigation relates to the recognised production problems and difficulties encountered in the P.M. industry, relating to the widespread use of admixed lubricants pre-mixed with metal powder prior to die compaction. This is necessary to reduce die-wall friction and particularly ejection pressures which, otherwise, can cause even worse problems in the P.M. industry with uni-axially pressed and ejected parts. While the addition of lubricants may usefully reduce die-wall friction, problems arise during powder handling, compaction and subsequent sintering. The lubricant addition reduces the flow rate of a powder, and since the density of the lubricant is very low, tends to occupy a relatively large volume, hence reducing the green density of the compact. The green strength is thus affected by the quantity of added lubricant which following compaction, must be carefully removed during a de-waxing operation before full sintering. This last feature necessitates some restrictions with certain types of sintering furnace on accurate temperature control during the entire sintering process.

The concept of glass ceramics as die inserts has been described previously⁷ and need not be repeated here. The attractive properties and simplicity of manufacture associated with glass ceramics were considered sufficient to justify a programme to assess their potential as inserts for die compaction. The view was held that glass-ceramics could

offer distinct advantages in reducing die wall friction and in the long term could be developed, possibly along with other ceramics, where die-wall friction could be reduced sufficiently to obviate the necessity for lubricating the powder altogether.

The experimental details concerning the assemblage of glass-ceramic inserts into steel bolsters and the assessment of punch and ejection forces on sponge iron powder in both lubricated and unlubricated conditions has been fully described⁷. The results demonstrated a difference in compatibility of the ceramic surface with zinc stearate coated and plain iron powder particles. This compatibility showed a slight but positive advantage over a standard tool-steel die surface for lubricated iron powder, but with unlubricated powder some chemical interaction or bonding mechanism developed between the two surfaces which became aggravated by abrasive wear. This led to a point of such deterioration that circumferential cracking occurred at the upper region of the inserts producing failure of the ceramic in the form of a thin disc that flaked off during ejection. No further measurements became possible.

6 DISCUSSION OF RESULTS

6.1. Introduction

Production process techniques used currently in the P.M. industry are generally well established and in many cases offer little scope for variations or modifications. These may concern powder supply, powder handling and monitoring, pressing procedures with all the attendant tooling consideration and sintering operations. Nevertheless the patterns of powder behaviour that have been established during compaction, within this study, should provide considerable scope for improvement in quality with respect to properties of metal sinterings.

In many areas of the P.M. industry today, quality control on raw material powders is minimal and very often only density of the compacted part provides a measure of control related to a production run of parts manufactured from a particular batch of powder. Admittedly this is perhaps the easiest property to determine, particularly on certain die-pressed parts of uniform geometry. While many parts do not require the achievement of a very high density, demands for quality and improvements in properties, e.g. strength and fracture toughness of many other sintered parts have to be achieved if P.M. parts are to compete with those produced by other means. The following discussion reviews the results of this study with this attitude in mind.

6.2. Particle Characteristics

The production of metal powders can be carried out in a number of ways. A particular product, resulting from a process such as atomisation, is often secondary to the economics of the process. That is to say the process controls the metallurgical structure and geometry of the product which may not always be acceptable to the P.M. industry on the latter grounds. This is despite what may be an attractive price-structure associated with the product. Many powders are in fact, produced by atomisation giving a rounded more regular form than those produced by (say) chemical means. Unfortunately since the atomisation process is essentially a casting process the hardness of the powder particles may be unduly high with an irregular cast-type of structure. While a rounded particular structure contributes to better powder handling, i.e. flow rates, high apparent and tap densities the "as-atomised" structure may restrict extensive particle deformation essential to achieve high densification at the intermediate pressure levels often utilised in industry. A simple solution would be to vacuum or inert-anneal such atomised powders so that not only would the particle hardness be reduced, but reduced to a more consistent level instead of the variable hardnesses often found in atomised powders.

The irregularity of powders, typically produced by chemical or mechanical means, is often associated with high surface area and high interparticle friction. This

irregularity can create problems in classification or characterisation by simpler techniques such as sieving, which does not take into account axiality and shape characteristics of the particles. This irregularity, however, is considered more important in powder handling and loose packing behaviour during die-fillings procedures than the frictional effects existing between powder particles. While ratios of tap density to apparent density, expressed as the Hausner friction index, have been shown to increase with increasing particle irregularity, this increase is considered to be mainly due to the inability of a particular system to come to an equilibrium packing fraction on account of particle irregularity rather than on intrinsic interparticle friction forces.

Since a more regular system of loose particles should tend towards the ideal close packing system of spheres where every particle is in contact with 12 nearest neighbours it follows that this should promote a better transmission or distribution of forces within the compact and hence more uniform densification. This would be of more significance in die compaction where density distributions are traditionally variable in the pressed compact. Extending this concept further the results of the literature survey have indicated that considerable improvements in loose or apparent densities could arise through multi-size distributions of particles. Not only does this increase the apparent density but also increases the overall contact regions between particles.

This should assist sintering of the part to a higher state of strength, since one of the principal functions of the sintering process is to encourage neck growth, and the more contact regions there are the more interparticle bonding should develop.

6.3. Isostatic Compaction Studies

6.3.1. Effect of Particle Characteristics

While the effect of particle size on overall densification levels has been shown to be quite small the significance of this fact could be utilised in allowing for more consistency in pressed parts. Invariably, powder grades provided by powder manufacturers are supplied to a very nominal size distribution e.g. "100 mesh". This, in reality means the powder batch consists of a particle size distribution from a '100 mesh' size down to "fines" with variations in size distribution existing from batch to batch. Although this seems to be acceptable to the P.M. industry improvements towards ensuring consistency of products could be made by sieving powder batches into specific size fractions, either by the manufacturer or user. Thus compaction of monosize fractions of known size should ensure a higher degree of reproducibility of densification than that arising from powder of variable size distribution which has often led to inconsistencies in pressed parts. Furthermore if finer mono-size fractions of powder were compacted, the increased specific surface area while possibly contributing to a slight decrease in green densification in certain cases should promote a higher rate of sintering. Activated sintering of very fine

powders is an area that is being increasingly studied just lately.

The effect of particle geometry has been shown to have more of an effect on densification than particle size. The comparisons drawn between regular and irregular powders in loose handling are equally reflected during compaction. Regular powders densify to higher degrees than irregular powders as typified by the comparison between the two copper powders, (atomised and electrolytic).

6.3.2. Effect of De-Airing

This process of evacuating the air from a sealed bag of powder preparatory to isostatic compaction has been found not to have any significant effect on densification levels achieved over the pressure range employed in the programme. However, this observation is only pertinent to the relatively small size of compact which of necessity could be compacted with existing apparatus. Current application of isostatic techniques in the manufacture of large billets or pre-forms for eventual sinter forging may offer a useful investigation into de-airing procedures with respect to both densification and homogeneity of product and freedom from defects at geometrical changes of section.

6.3.3. Applicability of Equations of State

The internal geometry of real powder systems is often too complex to be amenable to exact mathematical treatments. Empirical curve-fitting can partly substitute strict mathematical deduction but somewhat weakens the link between

theory and experimental evidence. None of the many equations of state that have been proposed has been derived from real fundamental considerations of particle behaviour over the various ranges of compacting pressure. Practically all equations have expressed compacting pressure as a function of density or vice-versa. The results have indicated that density is influenced collectively by particle characteristics and features such as geometry, work-hardening capability and hardness. Despite these influencing factors the applicability of the Konopicky-Shapiro equation of state has more successfully indicated the mechanisms of compaction that the powders have undergone during compaction. The Kawakita equation, while giving good linear relationships for all the powders studied over the pressure range employed in this study, does not account satisfactorily for any behavioural pattern in terms of the constants utilised within the equation. Conversely the constants of the Konopicky - Shapiro equation can be related more directly to particle behavioural modes during compaction such as particle deformation and flattening and the onset of bulk compression. In such work on powder compaction behaviour it becomes important to depict the experimental results in such a way that one can clearly distinguish between the mechanisms involved. In this respect the Konopicky-Shapiro equation is considered to be more effective than the Kawakita equation.

6.3.4. Densification Mechanisms

The analysis of the data and information arising from the S.E.M. and metallographic studies coupled with the applicability

of the Konopicky-Shapiro equation of state has led to clear indications regarding the patterns of behaviour and densification mechanisms undergone by each powder.

(a) The stage of transitional restacking

This is the stage where inter-particle friction forces along with particle irregularity are most likely to affect initial densification. The stage has been quantitatively described in terms of punch movement in die compaction studies; the literature review^{2-4,9} refers to a number of cases where inter-particle movement, sliding and restacking takes place at relatively low applied pressures. In isostatic pressing such movements can not be so readily monitored and interpretation of the extent of this densification stage can only be derived either from structural examination of the compact after pressing or from density-pressure relationships.

The applicability of the Konopicky-Shapiro equation of state has been successful in this respect since the extrapolation of the $\ln 1/(1-D)$ relationship with pressure to zero pressure produces a value of density considered to be effective in describing the limit of inter-particle movement within this stage. Furthermore the ratio of this extrapolated density value to apparent density is considered to be an improvement on the Hausner friction index for characterising friction between powder particles. The Hausner index, incorporating tap density, tends to assume that tap density is a limiting factor in loose powder movement, beyond which "cohesive" densification commences. Many powders have shown this to be not so. However tap density could be regarded as a limiting loose-fill density level, but as previously mentioned this is

influenced more by particle irregularity than anything else. An interesting area of study would be to compare apparent, tap, and extrapolated density values at zero pressure for a spherical powder e.g. A.C.P. in relatively clean and lightly oxidised conditions. The two surface conditions should afford a more meaningful assessment of friction in terms of surface condition for a uniform state of particle geometry.

Structurally all the compacts pressed at the minimum compacting pressure show evidence of particle deformation which is relatively light. The metallographically sectioned samples show little interparticle involvement and strength is relatively low, a feature evidenced by the tendency for many of the more harder, regular powders to crumble at these lower compacting pressures. This is a feature worth emphasising that although the more irregular powders achieve lower densification levels their strength may be relatively higher than that of more regular powders due to mechanical interlocking, and very locally higher pressure points being developed at points of irregularity.

(b) The stage of local flow

Once particle movement ceases, further deformation in plastically deformable metal powders is by particle work-hardening and the development of interparticle contact areas. The S.E.M. and metallographic studies have clearly indicated how both these features develop. A point is worth making with regard to the measurement of micro-hardness at particle - particle junctions. Clearly the degree of work-hardening

experienced at the surface can not be precisely assessed by indentation tests. The area immediately next to the surface can be so tested, but unless extensive deformation has built up within the particle the measurements taken as close as practically possible to the junction area may not fully indicate the true degree of work-hardening. However, some meaningful trends have been demonstrated by the graphs of particle hardness with compacting pressure. The measurements of particle flattening, indicative of work-hardening, thus reflect more positively the progress of work-hardening.

While particle flattening and work-hardening can serve to describe quantitatively on a microscopic level the degree of work-hardening, macroscopically the rate of densification is more precisely assessed from the Konopicky-Shapiro relationship. Here two specific rates have been revealed for most powders. The higher densification rate has been shown to be related to the flow stress of the material where the softer more ductile materials densify very rapidly. Beyond a critical pressure the densification changes, which is governed by the material's work-hardening capability. Those powders which have the highest work-hardening capacity [high values of Meyer Index n] densify at the higher rate in this "second" stage of local flow while those powders having very limited work-hardening capacity (low values of n) densify at much reduced rates. In this respect the slope factors K_1 and K_2 of the Konopicky-Shapiro equation of state are

very significant with respect to metallurgical structure and condition of metal powders.

Over this stage of local flow it is worth comparing again the effects of particle size and geometry. For a given particle geometry, size effect has had only a minimal effect on densification level. Slope factors K_1 and K_2 , in the majority of cases, showed little variation within each fraction of powder tested. This suggests that despite the difference in surface area between the coarser and finer fractions of the powder the mechanisms of compaction undergone by each fraction are essentially the same. From this it may be inferred that surface area and interparticle friction are not factors that directly influence densification over this intermediate pressure range for a given particle geometry. The variability of the Kawakita $1/b$ constant within each fraction of powder tested proved to be a less reliable means of classifying a powder's behaviour to plasticity. Neither does the Kawakita equation indicate any of the stages undergone during compaction.

(c) The stage of bulk compression

Only two of the powders: aluminium and zinc reached this stage of compaction. Any further increase in compacting pressure beyond the "second" stage of local flow produced no further increase in densification. Examination of the photomicrographs of these two materials compacted over this higher pressure range confirms that porosity is negligible throughout the structure.

It is clear that this stage is reached only in the softest powders for equivalent compacting pressures. It would be expected that extending the pressure range a little higher for the copper powder would similarly achieve bulk compression and likewise for the harder materials at even higher pressures. Again, this serves to indicate the advantages of densifying powders in their softest metallurgical condition to achieve a maximum attainable density level for a given compacting pressure.

6.4. Die Compaction Studies

6.4.1. Tensile Strength

Generally this property is much less specified in green pressed P.M. compacts than density. The principal requirements insofar as strength is concerned are that as-pressed compacts possess sufficient green strength to allow them to be handled and transported prior to sintering. Additionally, in the case of isostatically pressed compacts, strength should be sufficiently high to counteract any constraining influences set up in the flexible bag on the decompression cycle and during ejection.

The study of tensile strength and its variation in the die-pressed zinc compacts shows that not only does strength increase with compacting pressure but is significantly dependent on particle size. This is quite a positive effect as opposed to density variation with particle size where the latter has little effect on density level. Furthermore, the peculiar inversion exhibited at intermediate pressure levels

on the coarser powders is not paralleled by any density inversion or work-hardening inversion. Examination of the particle contact flattening values for zinc indicates a slight inversion, as compacting pressure increases.

However, in view of the high degree of irregularity of this powder and the range of scatter experienced on assessing particle flattening from S.E.M. fracture examination no real significance can be inferred from the inversion at this stage.

This is an area of study which could prove very profitable in furthering the understanding of the rôle of surface behaviour in powder compaction. Little is known regarding the nature of surfaces and their effect during compaction. Yet powders are prone to many influences during handling and prior to compaction: e.g. adsorption of gas from the atmosphere, oxidation and the effects of additives and stearate-type lubricants.

6.4.2. Glass Ceramic Die Inserts

Subsequent considerations of the bolstering technique utilised in the programme have shown that marked improvements could be made in this area to avoid high peripheral "wedge" stresses set up in the upper regions of the ceramic during ejection. In the technique described⁷ it was revealed later that a relatively large volume of the upper part of the ceramic is mainly unsupported, thus minimising the degree of locked-in compressive stress after shrink-fitting. Under such circumstances the insert would be prone to brittle fracture through the combination of tensile stress and stress-raising

effects set up both under compression and ejection.

The results, however, gained from the programme were encouraging enough to warrant further investigation into glass-ceramics and possibly other ceramic materials e.g. silicon nitride and zirconium oxide as die-insert materials.

7 CONCLUSIONS

The conclusions arising out of the published work and that submitted herein are enumerated as follows :

7.1. Powder Characteristics

- 1 The flow rate of a powder is directly related to its apparent density. Flow rate increases with particle regularity and in powders of high relative apparent density.
- 2 Powders of higher particle irregularity have lower values of relative apparent density and vice-versa.
- 3 Increasing particle irregularity produces higher ratios of tap density to apparent density (Hausner friction index). For a given particle geometry the ratio increases further as particle size decreases.
- 4 Powders produced by the atomisation process have varying degrees of regularity, from near-spherical geometries to rounded but more irregular geometries. Surface areas are generally lower in these powders compared to those powders produced chemically. Atomised powders also exhibit less divergence from an "ideal" relationship of surface area with particle size based upon perfect sphericity.
- 5 The assessment of the Meyer work-hardening index affords a useful means of establishing the extent and

capability of a metal powder to plastic deformation and work-hardening during compaction. Zinc was found to have the greatest capacity for work-hardening and S.G. nickel the least capacity.

7.2. Isostatic Compaction

- 1 Isostatic compaction as a production process has been extensively reviewed and compared with other compacting processes e.g. die compaction. The process has developed to a stage where improved automation particularly in the dry-bag process can lead to attractive production output rates for relatively simple shapes. In other respects isostatic compaction can not as yet rival production rates with die compaction. Dimensional accuracy is limited for surfaces in contact with the flexible bag though improved accuracy and tighter control of tolerances are possible using profiled formers. Density levels with isostatic compaction are higher than those achieved with die compaction; this was confirmed for the compaction of a monosize ($-125+106\mu\text{m}$) fraction of zinc powder.
- 2 The effect of particle size on the densification of S.G. nickel, sponge iron, A.C.P. and E.C.P. was found to be minimal. Density levels in various size fractions of these compacted powders showed only minor variations over a compacting pressure range of 5-50 t.s.i.

- 3 The effect of particle geometry on densification of powders of comparable metallurgical characteristics was limited to a comparison between A.C.P. and E.C.P. The comparison showed that the more regular A.C.P. densified to a much higher degree than the more irregular E.C.P. at equivalent compacting pressures.
- 4 The effect of de-airing monosize ($-125+106\mu\text{m}$) fractions of loose powder fills of all powders produced only slight variations in density level for equivalent compacting pressures. Slight increases in density following de-airing occurred in the S.G. nickel, stainless steel, iron and zinc powders particularly for lower compacting pressures, while slight decreases occurred in the A.C.P. E.C.P. and aluminium powders. For the small size of the compacts tested it is thus concluded that de-airing has no significant effect on densification.
- 5 The applicability of the Kawakita equation of state produced good linearity over the compacting pressure range 5-50 t.s.i. for all powders. The interpretation of the constants led to a satisfactory comparison being drawn between the reciprocal of the slope factor a and the initial loose porosity of the powder. No correlation could be made between $1/b$ values and any powder property, particularly that of work-hardening capability and plasticity.

- 6 The applicability of the Konopicky-Shapiro equation of state was found to be more successful and appropriate in describing the different modes of compaction in each of the powders tested.
- 7 Extrapolation of the first linear region to zero pressure, using the Konopicky-Shapiro equation of state, gave a value of relative density D_A . The ratio of this value to relative apparent density, i.e. D_A/D_a accounted more satisfactorily for interparticle friction than the Hausner friction index. Dividing the compression ratio of relative green density D_G to relative apparent density D_a i.e. D_G/D_a , by D_A/D_a rather than D_t/D_a , gave values showing improved constancy irrespective of particle size and compacting pressure for S.G. nickel, E.C.P. and iron. The value D_A is considered to be an effective limiting value of density beyond which interparticle movement essentially ceases, and quantitatively describes the limit of transitional restacking.
- 8 The stage of local flow can be described from the Konopicky-Shapiro equation of state as occurring in two sub-stages. Beyond the stage of transitional re-stacking the rate of densification expressed by the first slope factor K_1 is inversely proportional to the flow stress of the powder. Above a critical compacting pressure the rate of densification expressed by the second slope factor K_2 is then directly proportional to the capacity of work hardening of the powder expressed by the Meyer index n .

- 9 The stage of local flow can also be described from
- (a) fracture surface examination by S.E.M.
 - (a) metallographic examination and micro-hardness indentation tests carried out at interparticle boundary regions.
 - (c) quantitative assessment of pore closure.
- 10 Fracture surface examination by S.E.M. was quantified by measuring the diameter or "equivalent diameter" d of flattened particles and relating mean values of d to a maximum diameter d_m based upon a dodecahedron model of particle deformation. The ratios of d/d_m plotted against compacting pressure indicated regions where particle deformation had occurred at different rates. These regions are similar to those corresponding to the slope factors K_1 and K_2 of the Konopicky-Shapiro equation of state.
- 11 Plots of interparticle boundary micro-hardness values against compacting pressure showed that particle work-hardening had occurred very rapidly over the lower compacting pressure regions and less so over the higher pressure regions. The graphs indicated regions where particle work-hardening occurred at different rates over pressure regions similar to those derived from the S.E.M. fracture examination and Konopicky-Shapiro equation of state.
- 12 The quantitative assessment of pore closure in S.G. nickel compacted over the compacting pressure range 5-50 t.s.i. indicated a pressure threshold at 20 t.s.i. where particle work-hardening and deformation rates changed.

- 13 The stage of bulk compression over the compacting pressure range of 5-50 t.s.i. was attained only by the two softest powders : aluminium and zinc. Beyond 25-30 t.s.i. no further increase in density occurred.

7.3. Die Compaction

- 1 The tensile strength of zinc compacts increased with compacting pressure over the pressure range 5-50 t.s.i. An inversion in strength occurred for coarse and intermediate size fractions over the pressure range 20-35 t.s.i.
- 2 For equivalent high compacting pressures tensile strength increased as particle size decreased. A tentative relationship between strength and particle size, at high compacting pressures, is proposed based upon the Petch equation.
- 3 The variation in tensile strength with pressure could not be accounted for in terms of density variation, nor on interparticle contact area of flattening.
- 4 The inversion in strength with compacting pressure is attributed to a complex process involving particle work-hardening, relaxation of residual stresses and involvement of surface bonding energies, rather than particle slippage or re-arrangement.

- 5 Possibilities lie in the further development of ceramics as inexpensive die materials for powder compaction in which die-wall friction could be significantly reduced.

8 SUGGESTIONS AND FURTHER WORK

- 1 A more fundamental investigation into surface phenomenological behaviour of powders would be expected to account more satisfactorily for :
 - (a) friction effects between particles ;
 - (b) interparticle attraction during loose handling and die-filling operations ;
 - (c) the cohesive strength between particles during densification .
- 2 An investigation into the improvement of loose or apparent densities of powder bodies by multi-size distributions of powder fractions would be of importance in improving the efficiency of die-filling procedures. Additionally compaction would commence at an initially higher density and be expected to lead to higher density levels on compaction. Alternatively equivalent densities could be achieved for reduced compacting pressures. This is an area of interest in the Civil Engineering industry with respect to load carrying vehicular surfaces of packed aggregates.
- 3 It is recommended that further investigation be carried out into the effect of metallurgical structure on densification behaviour for a given particle geometry. This could be achieved most conveniently in monosize fractions of a spherical powder in different states of hardness.

- 4 The present work and above recommendations could be usefully extended to include many pre-alloyed powders where alloying additions and solid-solution effects would be expected to have quite significant effects on densification behaviour and rates. This area would be of particular relevance in the nickel-base superalloy and high-quality steel e.g. stainless and tool-steel, industries.
- 5 There is ample scope for more detailed studies of the structural features of compacts, e.g. the examination of fracture surfaces, the investigation of work-hardening response both by hardness tests and preferably by X-Ray diffraction studies and the study of pore closure. These are all features considered to be important and effective during later sintering of the compact to higher density and strength.
- 6 On a production processing basis the isostatic process would benefit by studies into the effects of variables on quality and consistency of product, such as:
 - (a) rate of compaction ;
 - (b) dwell time ;
 - (c) decompression rate ;
 - (d) tooling design ;

These variables are considered important with regard to densification and would profitably lead to improvements in the isostatic process in competition with other metal fabrication processes.

- 7 Further work is proposed in the formulation of suitable insert materials and tooling design with a view to reducing die-wall friction effects during die-compaction. This could lead to the elimination of the current practice of adding lubricants to metal powders.

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LIST OF TABLES

TABLE 1	..	Calibration of Manganin Gauge
TABLE 2	..	Comparison of Green Density of a $-125+106\mu\text{m}$ fraction of ACP isostatically pressed in the Monobloc and Compound Die-Sets
TABLE 3	..	Calibration of Mercury Pycnometer
TABLE 4	..	Characteristics of S.G. Nickel Powder
TABLE 5	..	Characteristics of Atomised Copper Powder (A.C.P.)
TABLE 6	..	Characteristics of Electrolytic Copper Powder (E.C.P.)
TABLE 7	..	Characteristics of Aluminium Powder
TABLE 8	..	Characteristics of Stainless Steel Powder
TABLE 9	..	Characteristics of Sponge Iron Powder
TABLE 10	..	Characteristics of Zinc Powder
TABLE 11	..	Ratios of Tap Density to Apparent Density
TABLE 12	..	Particle Microhardness and Meyer Analyses
TABLE 13	..	Relative Densities of Isostatically Pressed Atomised and Electrolytic Copper Powders
TABLE 14	..	Relative Densities of Isostatically Pressed Powders in non de-aired (N.D.A) and de-aired (D.A.) conditions
TABLE 15	..	Comparison of Kawakita constants for different size fractions of S.G. Nickel, Iron, Atomised and Electrolytic Copper Powders
TABLE 16	..	Comparison of Kawakita constants for a monosize fraction ($-125+106\mu\text{m}$) of stainless steel, aluminium and zinc powders
TABLE 17	..	Comparison of Konopicky-Shapiro constants with yield stress and work-hardening data for a monosize fraction ($-125+106\mu\text{m}$) of all powders
TABLE 18	..	Comparison of particle flattening data with compacting pressure for a monosize fraction ($-125 + 106\mu\text{m}$) of six powders
TABLE 19	..	Values of tensile strength and relative density of die-pressed zinc powder fractions for different compacting pressures

TABLE I

Calibration of Manganin Gauge

Applied Pressure t.s.i.	Manganin Gauge	
	Potential mV	Deduced Pressure tsi
10.0	9.2	10.0
15.0	13.9	15.0
20.0	18.2	20.0
25.0	23.0	25.0
30.0	27.5	30.0
35.0	32.2	35.0
40.0	36.9	40.0
45.0	41.1	45.0
50.0	45.8	49.9
55.0	50.4	54.9
60.0	55.2	59.8
65.0	59.5	64.7

TABLE 2

Comparison of Green Densities of a -125 + 106 μ m
fraction of A.C.P. isostatically pressed in the
Monobloc and Compound Die-Sets

Compacting Pressure t.s.i.	Relative Green Densities	
	Monobloc Die	Compound Die
5	0.775	0.772
10	0.836	0.837
15	0.879	0.875
20	0.907	0.906
25	0.922	0.917
30	0.935	0.936
35	0.946	0.944
40	0.945	0.950
45	0.955	0.953
50	0.954	0.957

TABLE 3

Calibration of Mercury Pycnometer

Diameter of Ball cm.	Calculated Volume cm ³	Measured Volume cm ³	% Error
0.793	0.261	0.259	-0.77
0.952	0.452	0.452	0.00
1.111	0.718	0.716	-0.28
1.270	1.073	1.072	-0.09
1.905	3.620	3.619	-0.03

TABLE 4

Characteristics of S.G. Nickel Powder

Particle Size μm	Relative Apparent Density	Relative Tap Density	Flow Rate sec (50g)	Specific Surface Area m^2g^{-1}	
				Actual	"Ideal"
-150+125	0.494	0.575	21.7	0.0067	0.0049
-125+106	0.488	0.574	21.6	0.0072	0.0058
-106+90	0.477	0.575	22.6	0.0089	0.0069
-90 +75	0.473	0.573	21.8	0.0101	0.0082
-75 +63	0.471	0.572	21.0	0.0118	0.0098
-63 +53	0.469	0.573	21.1	0.0137	0.0116
-53 +45	0.458	0.563	21.5	0.0222	0.0138
-45 +38	0.430	0.542	21.7	0.0250	0.0162
-38 +00	0.409	0.529	22.3	0.0290	0.0306

TABLE 5

Characteristics of Atomised Copper Powder
(A.C.P.)

Particle Size μm	Relative Apparent Density	Relative Tap Density	Flow Rate sec (50g)	Specific Surface Area m^2g^{-1}	
				Actual	"Ideal"
-150+125	0.576	0.638	17.5	0.0058	0.0049
-125+106	0.576	0.637	16.9	0.0065	0.0058
-106+ 90	0.576	0.636	16.2	0.0075	0.0068
-90+75	0.575	0.635	16.0	0.0083	0.0081
-75+63	0.574	0.633	15.8	0.0100	0.0097
-63+53	0.569	0.632	15.5	0.0126	0.0115
-53+45	0.565	0.626	15.4	0.0135	0.0137
-45+38	0.566	0.627	16.3	0.0152	0.0161
-38+00	0.560	0.654	22.2	0.0296	0.0304

TABLE 6

Characteristics of Electrolytic Copper Powder (E.C.P)

Particle Size μm	Relative Apparent Density	Relative Tap Density	Flow Rate sec (50g)	Specific Surface Area m.g ⁻¹	
				Actual	"Ideal"
-150+125	0.300	0.376	39.6	0.0183	0.0049
-125+106	0.304	0.378	39.0	0.0220	0.0058
-106+90	0.297	0.381	38.6	0.0290	0.0068
-90+75	0.297	0.382	38.4	0.0311	0.0081
-75+63	0.297	0.386	40.4	0.0348	0.0097
-63+53	0.295	0.384	40.6	0.0365	0.0115
-53+45	0.293	0.380	40.8	0.0380	0.0137
-45+38	0.291	0.366	40.9	0.0416	0.0161
-38+00	0.292	0.361	n.m.	0.0829	0.0304

n.m. = not measurable

TABLE 7

Characteristics of Aluminium Powder

Particle Size μm	Relative Apparent Density	Relative Tap Density	Flow Rate sec. (50g)	Specific Surface Area m^2g^{-1}	
				Actual	"Ideal"
-150+125	0.374	0.485	132	0.0284	0.0160
-125+106	0.370	0.488	119	0.0326	0.0191
-106+90	0.352	0.468	127	0.0485	0.0225
-90+75	0.356	0.479	122	0.0576	0.0267
-75+63	0.344	0.484	n.m	0.0678	0.0319
-63+53	0.347	0.498	n.m	0.0792	0.0380
-53+45	0.337	0.484	n.m	0.0870	0.0450
-45+38	0.340	0.498	n.m	0.0879	0.0531
-38+00	0.315	0.502	n.m	0.1273	0.1002

n.m = not measurable

TABLE 8

Characteristics of Stainless Steel Powder

Particle Size μm	Relative Apparent Density	Relative Tap Density	Flow Rate sec (50g)	Specific Surface Area m^2g^{-1}	
				Actual	"Ideal"
-150+125	0.277	0.371	54.0	0.0170	0.0055
-125+106	0.288	0.381	53.0	0.0191	0.0066
-106+90	0.293	0.397	48.0	0.0228	0.0077
-90+75	0.301	0.407	47.1	0.0241	0.0092
-75+63	0.317	0.421	44.4	0.0275	0.0110
-63+53	0.323	0.428	43.8	0.0311	0.0130
-53+45	0.325	0.433	42.7	0.0319	0.0154
-45+38	0.327	0.439	45.9	0.0335	0.0182
-38+00	0.326	0.452	n.m	0.0423	0.0344

n.m = not measurable

TABLE 9

Characteristics of Sponge Iron Powder

Particle Size μm	Relative Apparent Density	Relative Tap Density	Flow Rate sec (50g)	Specific Surface Area m^2g^{-1}	
				Actual	"Ideal"
-150+125	0.392	0.453	30.5	0.0151	0.0055
-125+106	0.384	0.449	31.5	0.0170	0.0066
-106+90	0.380	0.443	32.7	0.0179	0.0078
-90+75	0.374	0.441	33.0	0.0199	0.0092
-75+63	0.372	0.441	32.9	0.0235	0.0110
-63+53	0.367	0.443	35.2	0.0339	0.0131
-53+45	0.355	0.460	n.m	0.0324	0.0156
-45+38	0.345	0.480	n.m	0.0391	0.0184
-38+00	0.337	0.501	n.m	0.0482	0.0347

n.m. = not measurable

TABLE 10

Characteristics of Zinc Powder

Particle Size μm	Relative Apparent Density	Relative Tap Density	Flow Rate sec (50g)	Specific Surface Area m^2g^{-1}	
				Actual	"Ideal"
-150+125	0.394	0.474	58	0.0129	0.0061
-125+106	0.390	0.464	55	0.0142	0.0073
-106+90	0.388	0.465	52	0.0153	0.0086
-90+75	0.383	0.471	52	0.0170	0.0102
-75+63	0.384	0.475	47	0.0202	0.0122
-63+53	0.388	0.482	46	0.0244	0.0145
-53+45	0.388	0.484	45	0.0259	0.0171
-45+38	0.397	0.492	47	0.0294	0.0202
-38+00	0.399	0.544	n.m	0.0564	0.0382

n.m = not measurable

TABLE 11

Ratios of Tap Density to Apparent
Density

Particle Size μm	S.G Nickel	A.C.P.	E.C.P.	Alum inium	Stainless Steel	Iron	Zinc
-150+125	1.16	1.11	1.25	1.30	1.34	1.16	1.20
-125+106	1.18	1.11	1.24	1.32	1.33	1.17	1.19
-106+90	1.21	1.10	1.28	1.33	1.35	1.17	1.20
-90+75	1.21	1.10	1.29	1.35	1.35	1.18	1.23
-75+63	1.21	1.10	1.30	1.41	1.33	1.19	1.24
-63+53	1.22	1.11	1.30	1.44	1.33	1.21	1.24
-53+45	1.23	1.11	1.30	1.44	1.33	1.30	1.25
-45+38	1.26	1.11	1.26	1.46	1.34	1.39	1.24
-38+00	1.29	1.17	1.24	1.59	1.39	1.49	1.36

TABLE 12

Particle Microhardnesses and Meyer Analyses

Indent* Load P g	Log ₁₀ P	S.G. Nickel		A.C.P.		Aluminium		Stainless Steel		Iron		Zinc	
		d _{m.d.} *	log ₁₀ d	d _{m.d.} *	log ₁₀ d	d _{m.d.} *	Log ₁₀ d	d _{m.d.} *	log ₁₀ d	d _{m.d.} *	log ₁₀ d	d _{m.d.} *	log ₁₀ d
1	0	-	-	-	-	88	1.94	-	-	-	-	82	1.91
2	0.30	-	-	-	-	115	2.06	-	-	-	-	106	2.03
3	0.48	-	-	88	1.94	133	2.12	-	-	65	1.81	126	2.10
5	0.70	57	1.76	118	2.07	177	2.25	47	1.67	78	1.89	151	2.18
10	1.00	73	1.86	150	2.18	252	2.40	63	1.80	101	2.00	189	2.28
20	1.30	101	2.00	202	2.31	344	2.54	87	1.94	145	2.16	278	2.44
30	1.48	126	2.10	234	2.37	-	-	108	2.03	180	2.26	343	2.54
50	1.70	161	2.21	302	2.48	-	-	140	2.15	232	2.37	-	-
100	2.00	219	2.34	-	-	-	-	190	2.28	-	-	-	-
Microhardness M.H. (15μm)		224 (30g)		42.6 (5g) 38.0 ECP.		18.0 (2g)		260 (30g)		130 (20g)		28 (3g)	
Meyer Exponent 12		2.08		2.36		2.22		2.10		2.21		2.42	

* m.d. = micro-divisions

8 micro-divisions = 1 μm

TABLE 13

Relative Densities of Isostatically
Pressed Atomised and Electrolytic Copper
Powders

Particle Size μm	Compacting Pressure kN/mm^2									
	0.08		0.16		0.24		0.32		0.40	
	ACP	ECP	ACP	ECP	ACP	ECP	ACP	ECP	ACP	ECP
-212+150	0.766	0.590	0.818	0.706	0.861	0.777	0.902	0.799	0.929	0.836
+150+106	0.771	0.600	0.832	0.716	0.876	0.780	0.905	0.822	0.914	0.843
-106+75	0.765	0.596	0.833	0.702	0.876	0.759	n.d	0.804	0.930	0.839
-75+63	0.757	0.597	0.824	0.698	0.876	0.782	0.911	0.807	0.927	0.846
-63+53	0.773	0.596	0.833	0.696	0.880	0.754	0.909	0.803	0.922	0.846
-53+45	0.767	0.598	n.d	0.673	0.876	0.775	n.d	0.804	0.923	0.850
-45+38	0.770	0.591	0.829	0.673	0.878	0.773	0.912	0.802	0.929	0.843
-38+00	0.772	0.582	0.839	0.679	0.878	0.744	0.908	0.779	0.925	0.828

n.d = not determined

TABLE 14

Relative Densities of Isostatically Pressed Powders
in non-de-aired (N.D.A.) and de-aired (D.A.) conditions

Compacting Pressure tsi	S.G. Nickel		A.C.P.		Aluminium		Stainless Steel		Iron		Zinc	
	N.D.A.	D.A.	N.D.A.	D.A.	N.D.A.	D.A.	N.D.A.	D.A.	N.D.A.	D.A.	N.D.A.	D.A.
5	0.628	0.648	0.775	0.754	0.845	0.830	n.d.	n.d.	0.626	0.640	0.782	0.795
10	0.678	0.679	0.836	0.830	0.905	0.900	0.610	0.631	0.713	0.722	0.879	0.875
15	0.713	0.725	0.879	0.870	0.925	0.925	0.665	0.670	0.775	0.780	0.920	0.917
20	0.738	0.751	0.907	0.904	0.935	0.940	0.714	0.728	0.809	0.810	0.937	0.941
25	0.771	0.757	0.922	0.917	0.945	0.940	0.757	0.753	0.841	0.839	0.947	0.949
30	0.799	0.803	0.935	0.925	0.945	0.945	0.770	0.781	0.858	0.856	0.951	0.951
35	0.817	0.826	0.946	0.943	0.950	0.945	0.790	0.797	0.874	0.867	0.949	0.945
40	0.826	0.844	0.945	0.945	0.950	0.945	0.811	0.811	0.887	0.877	0.953	0.953
45	0.847	0.855	0.955	n.d.	0.950	0.945	0.825	0.823	0.893	0.887	0.953	0.956
50	0.865	0.870	0.954	0.947	0.945	0.945	0.830	0.834	0.892	0.896	0.953	0.953

n.d. = not determined

TABLE 15

Comparison of Kawakita constants for different size fractions of
S.G. Nickel, Iron, Atomised and Electrolytic Copper Powders.

Particle Size μm	S.G.Nickel			Iron			A.C.P.			E.C.P.		
	a	$1-D_a$	$1/b$	a	$1-D_a$	$1/b$	a	$1-D_a$	$1/b$	a	$1-D_a$	$1/b$
-150 + 106	0.509	0.509	9.21	0.592	0.612	2.66	0.429	0.424	3.95	0.695	0.694	2.14
-106 + 75	0.521	0.526	8.39	0.615	0.623	2.71	0.442	0.424	4.15	0.697	0.703	1.88
-75 + 63	0.535	0.529	8.29	0.622	0.628	2.61	0.462	0.426	5.05	0.697	0.703	1.88
-63 + 53	0.538	0.531	8.06	0.632	0.633	2.78	0.435	0.431	3.50	0.694	0.705	2.01
-53 + 45	0.538	0.542	8.06	0.636	0.645	2.23	0.441	0.435	3.63	0.708	0.707	2.27
-45 + 00	0.575	0.582	6.70	0.651	0.659	2.15	0.455*	0.434	4.27	0.719*	0.709	2.59

* evaluated on a size fraction -45 + 38 μm

$1/b$ values are all comparative, assessed from P/C values at zero pressure

TABLE 16

Comparison of Kawakita constants for
a monosize fraction (- 125 + 106 μ m)
of stainless steel, aluminium and zinc powders

Constant	Stainless Steel	Aluminium	Zinc
a	0.688	0.614	0.599
$1-D_a$	0.712	0.630	0.610
$1/b$	2.75	0.31	0.90

TABLE 17

Comparison of Konopicky - Shapiro constants with yield stress and work hardening data for a monosize fraction (-125 + 106 μ m) of all powders

Constant or Material Property	S.G. Nickel	A.C.P.	E.C.P.	Aluminium	Stainless Steel	Iron	Zinc
Slope Factor $K_1 \text{ t.s.i.}^{-1}$	0.024	0.058	0.040	0.093	0.032	0.031	0.096
Slope Factor $K_2 \text{ t.s.i.}^{-1}$	0.015	0.037	0.025	0.028	0.017	0.025	0.043
Yield Stress t.s.i.	20.0	4.0	-	2.25	15.0	9.0	n.d
Meyer Index n	2.08	2.36	n.d.	2.22	2.10	2.21	2.42
Slope Change Point $K_1 \rightarrow K_2, \text{t.s.i.}$	30	20	28	12	25	25	15
Slope Change Point $K_2 \rightarrow 0, \text{t.s.i.}$	-	-	-	30	-	-	30

n.d = not determinable

n.a = not available

TABLE 18

Comparison of particle flattening data with compacting pressure for a
a mohosize fraction (-125+106 μm) of six powders

Compacting Pressure t.s.i.	S.G Nickel			A.C.P.						Aluminium			Stainless Steel			Iron			Zinc		
				non-de-aired			de-aired														
	d μm	d/ dm	MH (30g)	d μm	d/ dm	MH (5g)	d μm	d/ dm	MH (5g)	d μm	d/ dm	MH (2g)	d μm	d/ dm	MH (30g)	d μm	d/ dm	MH (20g)	d μm	d/ dm	MH (3g)
5	38	0.59	228	37	0.57	47	38	0.59	45	43	0.66	26	-	-	-	51	0.79	136	29	0.45	29
10	49	0.76	232	44	0.68	56	43	0.66	47	55	0.85	29	37	0.57	300	55	0.85	165	45	0.69	35
15	56	0.86	247	50	0.77	63	-	-	-	58	0.89	32	43	0.66	310	56	0.86	170	53	0.82	40
20	59	0.91	256	55	0.85	65	54	0.83	60	59	0.91	35	44	0.68	320	60	0.92	180	61	0.94	41
25	60	0.92	280	57	0.88	65	56	0.86	60	62	0.96	35	46	0.71	321	61	0.94	189	58	0.89	42
30	59.5	0.92	285	58	0.89	65	58	0.89	61	64	0.99	37	48	0.74	328	63	0.97	200	59	0.91	42
35	59	0.91	290	61	0.94	65	60	0.92	62	65	1	37	51	0.79	330	65	1	200	64	0.99	42
40	60	0.92	295	64	0.99	66	63	0.97	65	65	1	37	51	0.79	332	65	1	200	70	1.08	42
45	60	0.92	300	65	1	67	-	-	-	65	1	37	50	0.77	334	65	1	202	65	1	44
50	62	0.96	300	65	1	69	65	1	66	65	1	37	51	0.79	335	65	1	202	72	1.11	44

TABLE 19

Values of tensile strength and relative density of die-pressed zinc powder fractions for different compacting pressures

Particle Size μm	Mean particle Dia. μm	$d^{-1/2}$ in	Compacting Pressure t.s.i.															
			5		13		20		24		30		36		40		50	
			σ tsi	Dg	σ tsi	Dg	σ tsi	Dg	σ tsi	Dg	σ tsi	Dg	σ tsi	Dg	σ tsi	Dg	σ tsi	Dg
-125+106	115.5	14.84	0.12	0.612	0.40	0.867	0.89	0.917	0.69	0.927	1.07	0.935	1.49	0.940	2.03	0.940	3.25	0.940
-106+90	98	16.10	0.14	0.610	0.64	0.859	0.94	0.915	1.38	0.925	1.89	0.934	1.27	0.938	2.20	0.938	3.30	0.938
-90+75	82.5	17.55	0.08	0.606	0.63	0.850	1.31	0.910	1.38	0.920	1.59	0.932	1.42	0.936	2.50	0.936	3.36	0.936
-75+63	69	19.19	0.09	0.606	0.60	0.855	1.17	0.910	1.38	0.924	2.53	0.938	1.83	0.941	2.64	0.941	3.40	0.940
-63+53	59	20.75	0.11	0.610	0.42	0.850	1.28	0.910	1.63	0.925	2.87	0.940	2.30	0.941	3.10	0.941	4.35	0.941
-45+38	41.5	24.75	0.11	0.616	0.73	0.862	1.10	0.911	1.39	0.926	1.80	0.936	1.95	0.936	2.55	0.936	4.58	0.936
-38+00	22	34.00	0.08	0.607	0.11	0.860	0.42	0.909	0.65	0.930	1.03	0.936	1.55	0.943	2.80	0.943	5.10	0.942

N.B. insufficient amount of -53 + 45 μm fraction available

LIST OF FIGURES

- FIGURES 1-7 (a) S.E.M. and (b) metallographic features of metal powders
- FIGURE 8 Monobloc Die Set
- FIGURE 9 Monobloc Die Set positioned in Denison Press
- FIGURE 10 Manufacture of P.V.C. "Tools"
- FIGURE 11 Compound Die Set
- FIGURE 12 Compound Die Set positioned in Denison Press
- FIGURE 13 Behaviour of various pressure transmitting fluids (Lowe and Goold ref. 19)
- FIGURE 14 Dog-Bone Die Set and Specimen Geometry
- FIGURE 15 Dog-Bone Die Set positioned in Denison Press
- FIGURE 16 Hall Flowmeter and Density Cup
- FIGURE 17 Air Permeameter
- FIGURE 18 Mercury Pycnometer
- FIGURE 19 Vacuum Dessicator for resin impregnation
- FIGURE 20 Relationship between flow rates and apparent densities
- FIGURES 21-23 Specific surface area versus Particle Size
- FIGURE 24 Meyer hardness relationships
- FIGURES 25-28 Density - Pressure relationships of multi-size fractions of powders
- FIGURES 29-34 Comparisons of Density versus Pressure for a monosize fraction ($-125 + 106\mu\text{m}$) of metal powders in non-aired (N.D.A) and de-aired (D.A.) conditions.
- FIGURES 35-38 Kawakita relationships
- FIGURES 39-42 Konopicky - Shapiro relationships

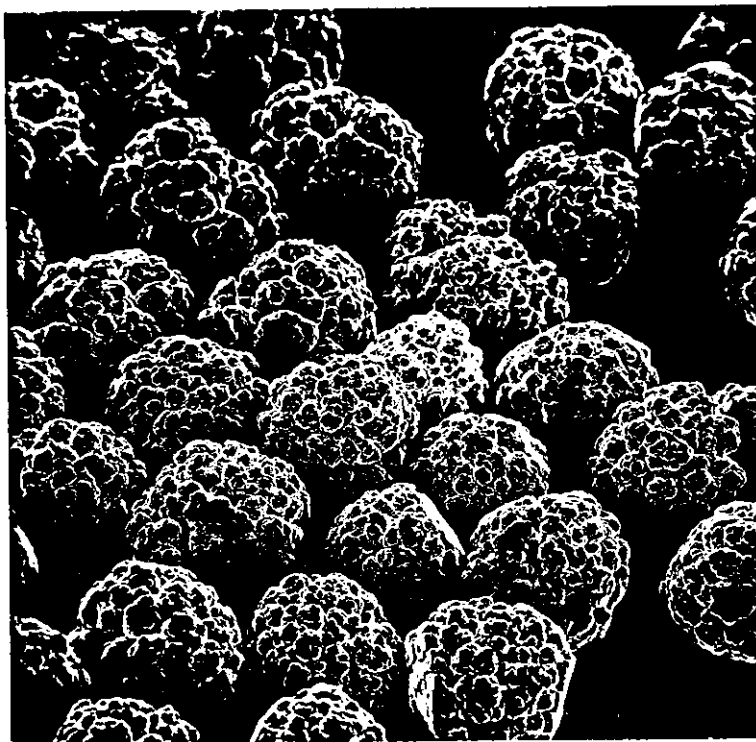
- FIGURE 43 Displacement of constant A at zero pressure in Konopicky - Ahapiro relationship
- FIGURE 44 Konopicky - Shapiro slope factor K_1 versus reciprocal of Yield Stress $1/\rho_0$.
- FIGURE 45 Konopicky - Shapiro slope factor K_2 versus Meyer work-hardening index n .
- FIGURE 46 Scanning Electron Micrographs of fracture surfaces of a monosize fraction ($-125 + 106\mu\text{m}$) of S.G. Nickel.
- FIGURE 47 Scanning Electron Micrographs of fracture surfaces of a monosize fraction ($-125 + 106\mu\text{m}$) of A.C.P.
- FIGURE 48 Scanning Electron Micrographs of fracture surfaces of a de-aired monosize fraction ($-125 + 106\mu\text{m}$) of A.C.P.
- FIGURE 49 Scanning Electron Micrographs of fracture surfaces of a monosize fraction ($-125 + 106\mu\text{m}$) of Aluminium
- FIGURE 50 Scanning Electron Micrographs of fracture surfaces of a monosize fraction ($-125 + 106\mu\text{m}$) of Stainless Steel
- FIGURE 51 Scanning Electron Micrographs of fracture surfaces of a monosize fraction ($-125 + 106\mu\text{m}$) of Iron
- FIGURE 52 Scanning Electron Micrographs of fracture surfaces of a monosize fraction ($-125 + 106\mu\text{m}$) of Zinc
- FIGURE 53 Model conversion of a sphere to a dodecahedron
- FIGURES 54-59 Changes in contact area d/d_m and particle microhardness M.H. with compacting pressure for a monosize fraction ($-125 + 106\mu\text{m}$) of powders
- FIGURE 60 Photomicrographs of impregnated compacts of a monosize fraction ($-125 + 106\mu\text{m}$) of S.G. Nickel
- FIGURE 61 Photomicrographs of impregnated compacts of a monosize fraction ($-125 + 106\mu\text{m}$) of A.C.P.
- FIGURE 62 Photomicrographs of impregnated compacts of a de-aired monosize fraction ($-125 + 106\mu\text{m}$) of A.C.P.
- FIGURE 63 Photomicrographs of impregnated compacts of a monosize fraction ($-125 + 106\mu\text{m}$) of Aluminium

- FIGURE 64 Photomicrographs of impregnated compacts
 of a monosize fraction ($-125 + 106\mu\text{m}$) of
 Stainless Steel
- FIGURE 65 Photomicrographs of impregnated compacts of
 a monosize fraction ($-125 + 106\mu\text{m}$) of Iron
- FIGURE 66 Photomicrographs of impregnated compacts of
 a monosize fraction ($-125 + 106\mu\text{m}$) of
 Zinc
- FIGURE 67 Changes in tensile strength and density of a
 monosize fraction ($75\mu\text{m}$) of spherical
 aluminium powder (Stromgren et al. ref.53)
- FIGURES 68-74 Changes in tensile strength and density of
 multisize fractions of zinc powder with
 compacting pressure
- FIGURE 75 Relationship between tensile strength and
 particle diameter of zinc powder compacted
 at 50 t.s.i.

FIGURES 1 - 7

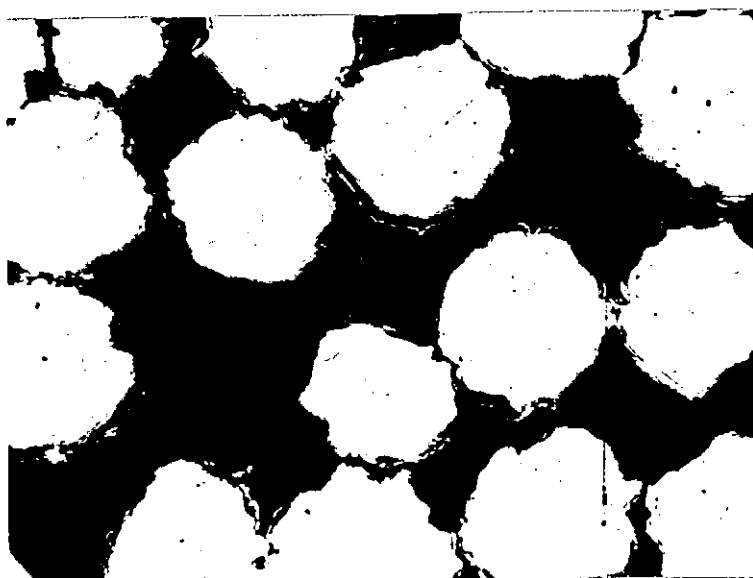
(a) S.E.M. and (b) metallographic features
of metal powders.

- | | |
|----------|------------------------------|
| FIGURE 1 | S.G. Nickel |
| FIGURE 2 | Atomised Copper (A.C.P.) |
| FIGURE 3 | Electrolytic Copper (E.C.P.) |
| FIGURE 4 | Aluminium |
| FIGURE 5 | Stainless Steel |
| FIGURE 6 | Sponge Iron |
| FIGURE 7 | Zinc |



X 200

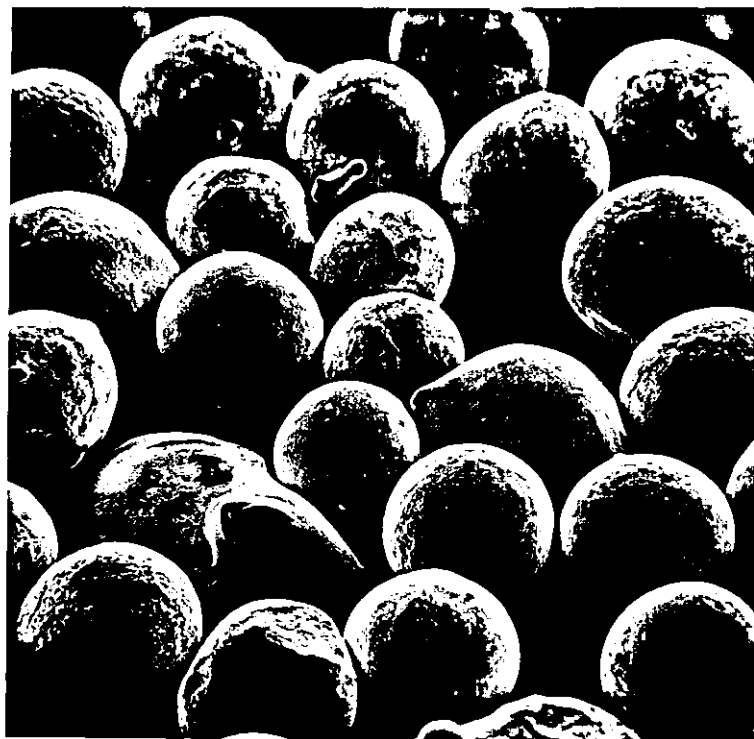
(a)



X 200

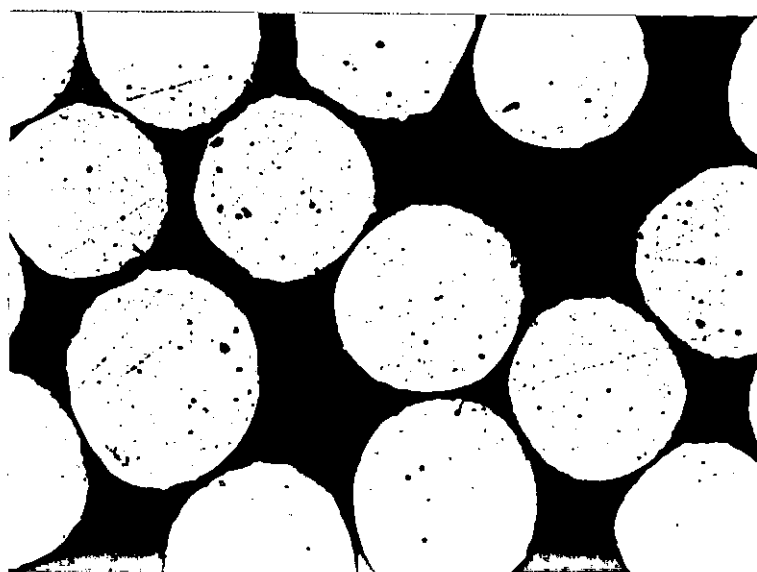
(b)

FIGURE 1



X 200

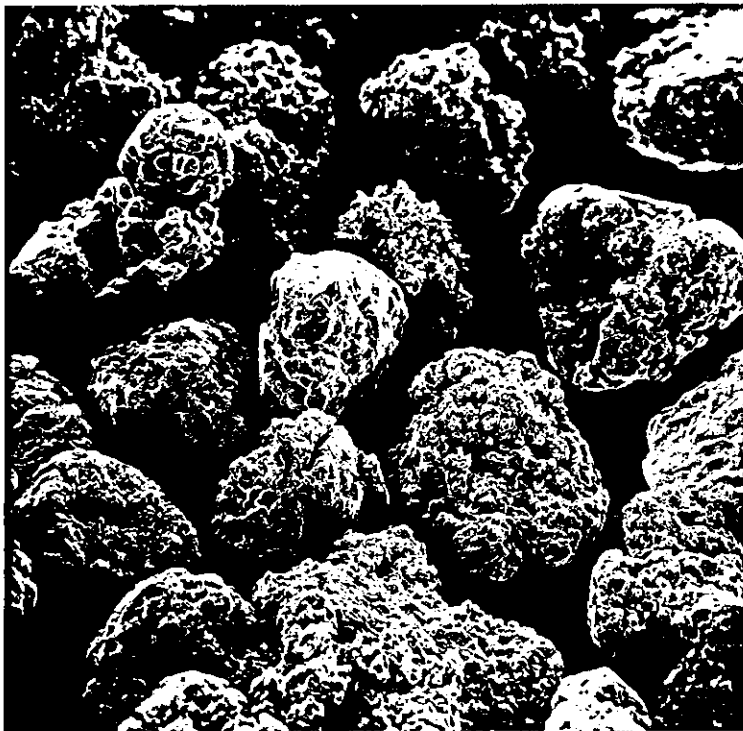
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X 200

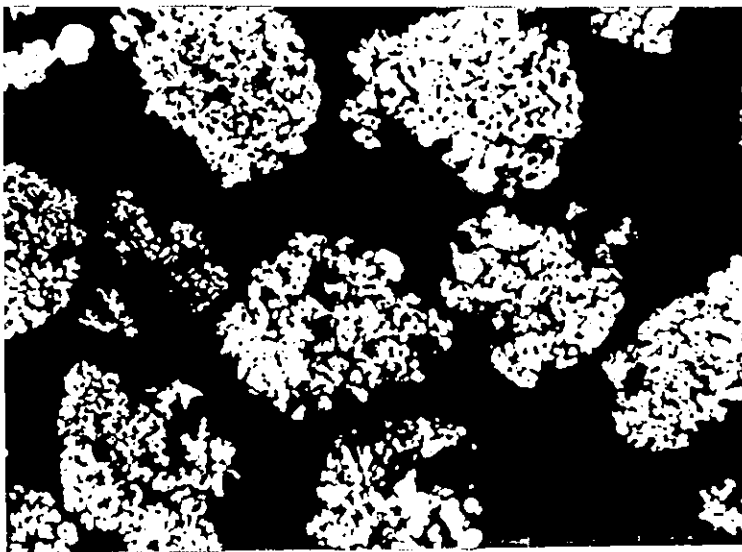
(b)

FIGURE 2



X 200

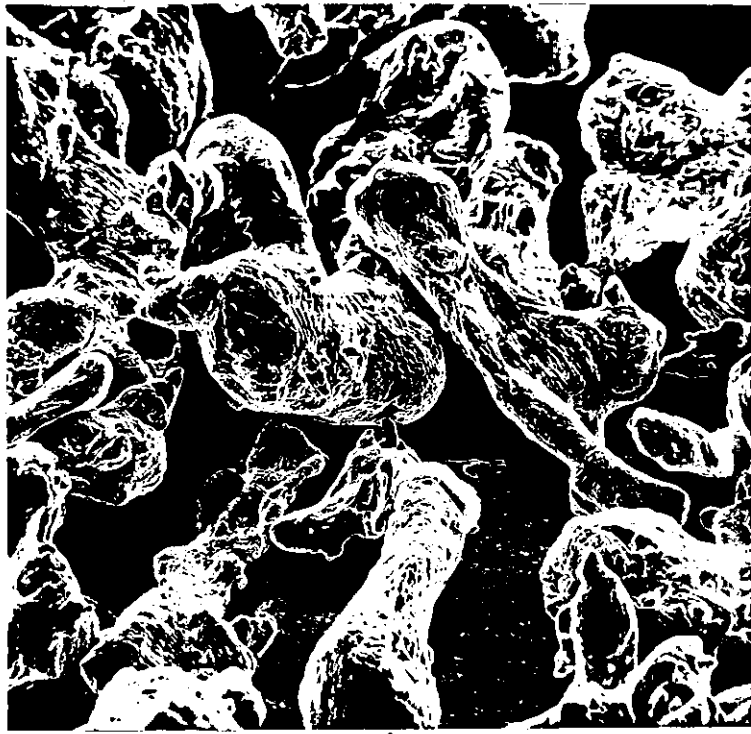
(a)



X 200

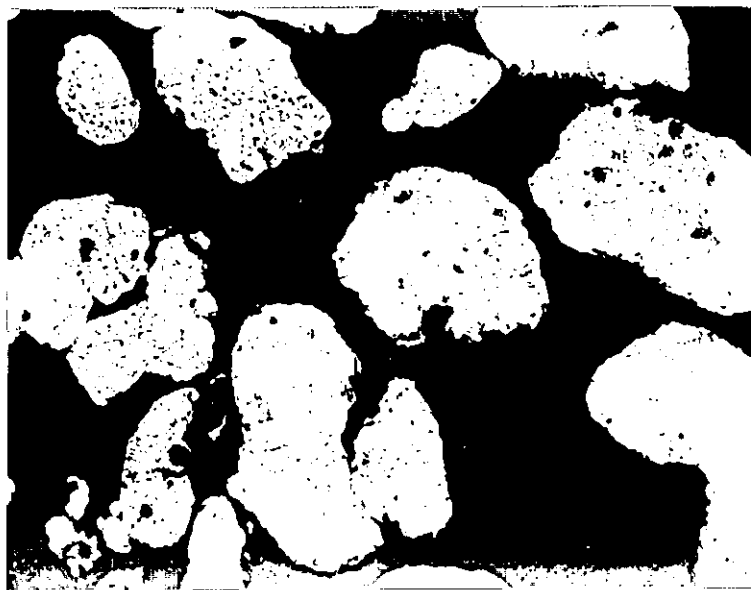
(b)

FIGURE 3



X 200

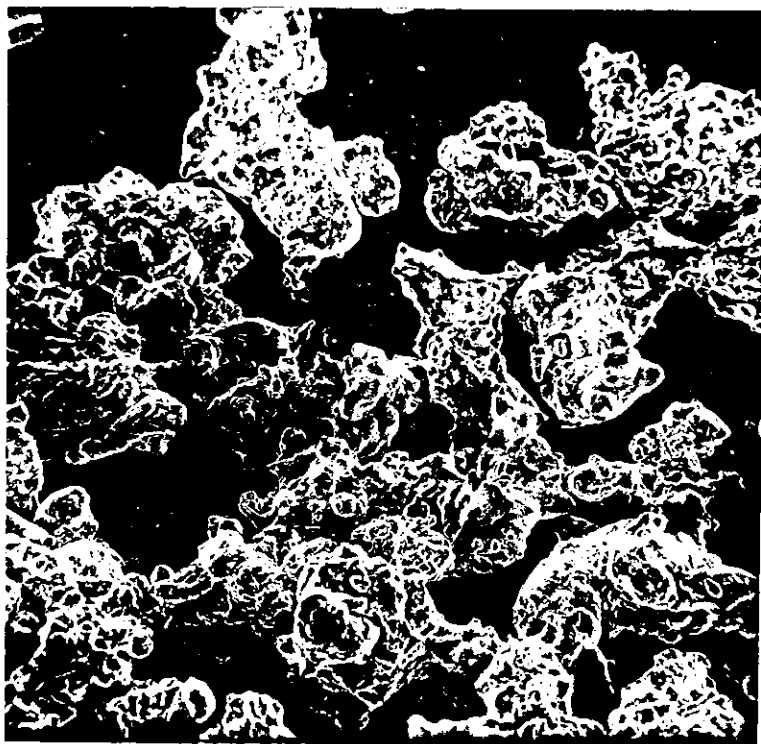
(a)



X 200

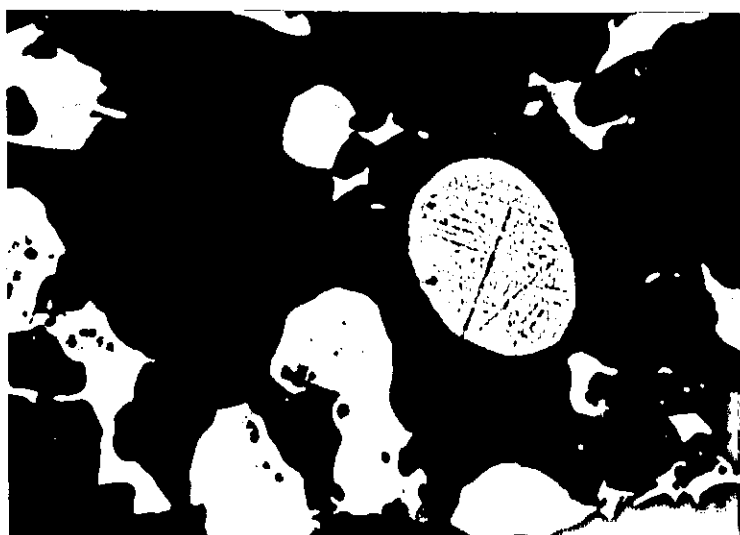
(b)

FIGURE 4



X 200

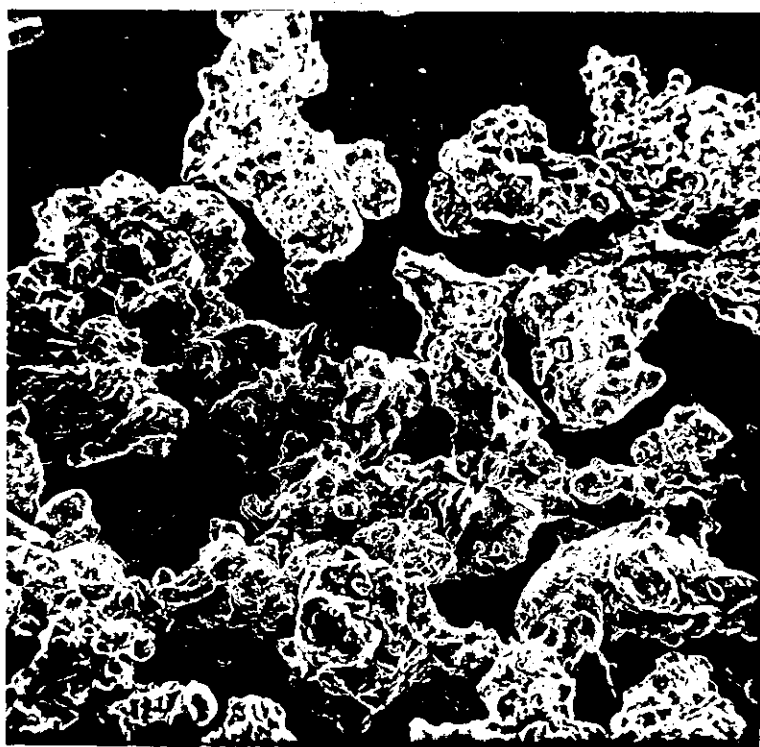
(a)



X 200

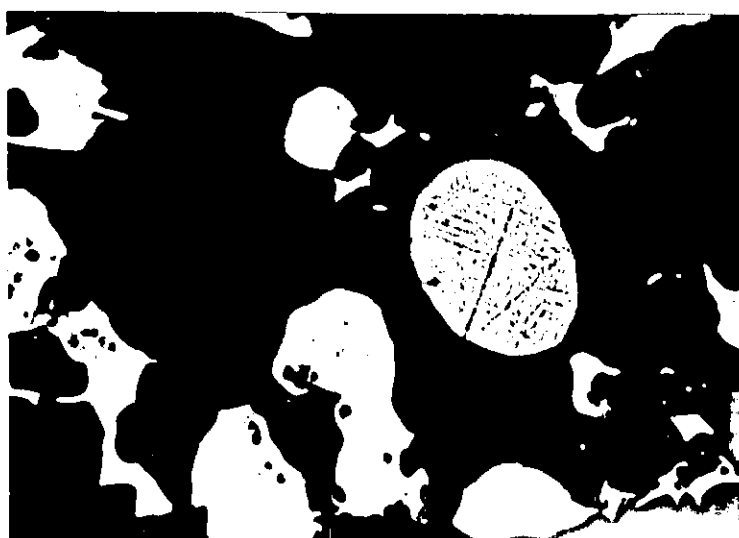
(b)

FIGURE 5



X 200

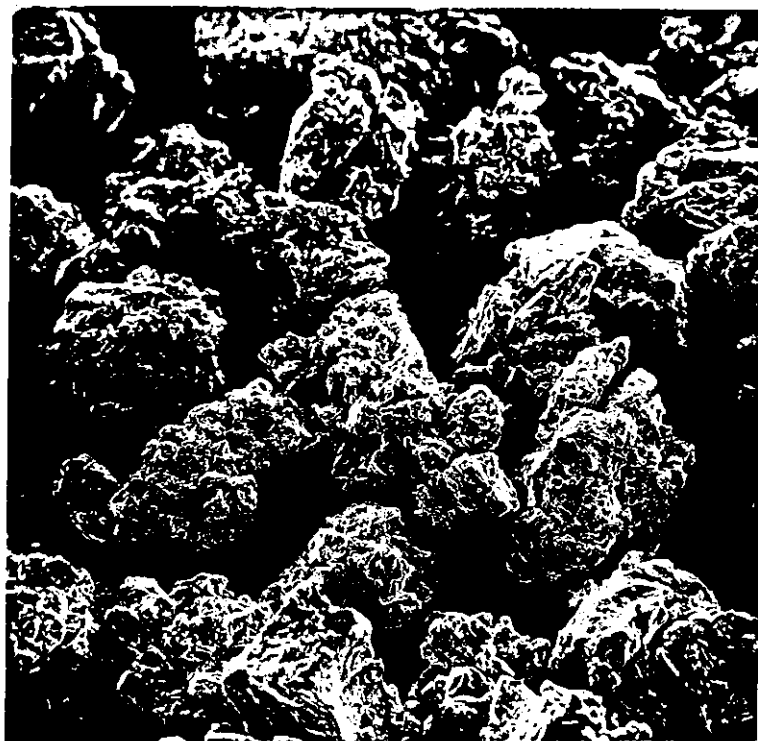
(a)



X 200

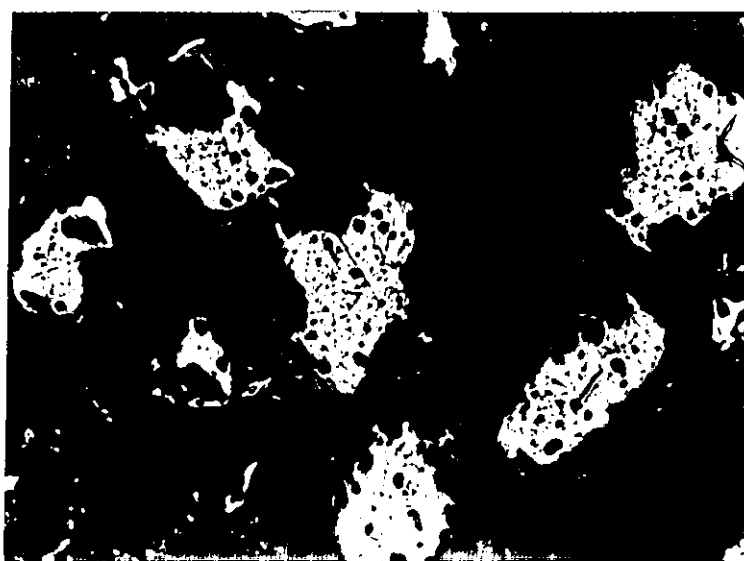
(b)

FIGURE 5



X 200

(a)



X 200

(b)

FIGURE 6



X 200

(a)



X 200

(b)

FIGURE 7

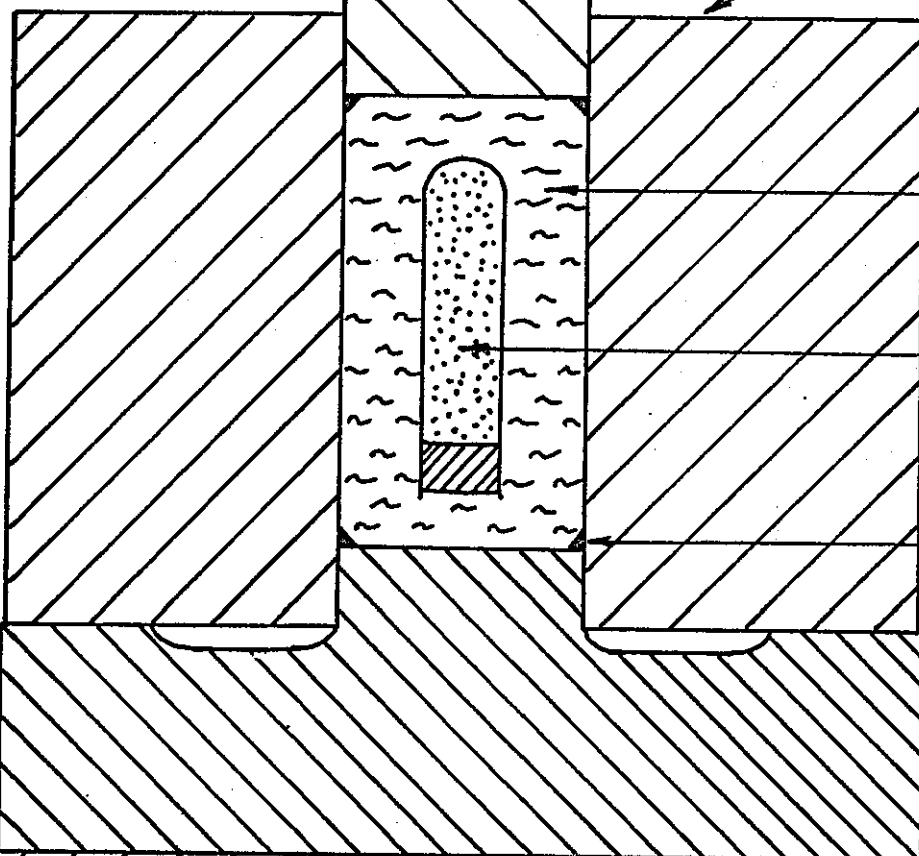
300 TON HYDRAULIC PRESS

TOP PLATTEN



TOP PLUNGER

MONOBLOC DIE



PLASTICIZED
PVC

POWDER
SAMPLE

Cu-Be MITRE
RING

BOTTOM END
CLOSURE

BOTTOM PLATTEN

FIGURE 8 Monobloc Die Set

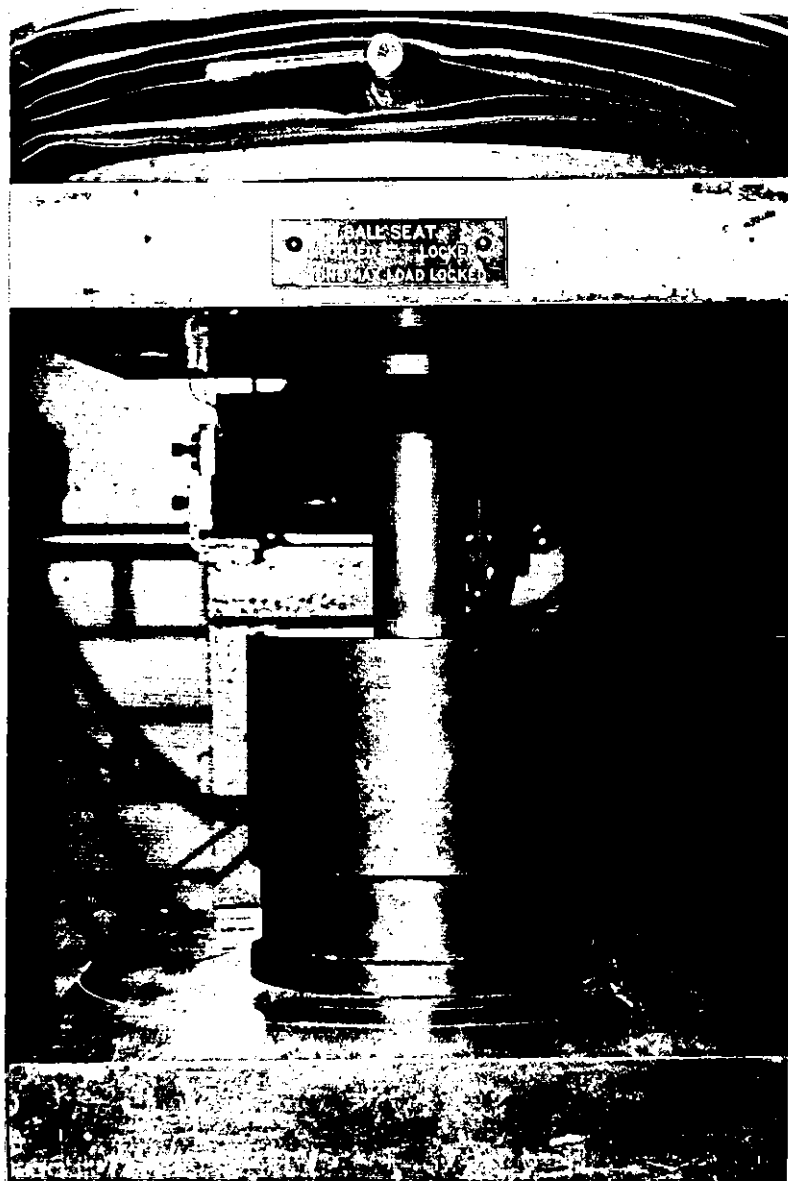


FIGURE 9 Monobloc Die Set positioned in
Denison Press

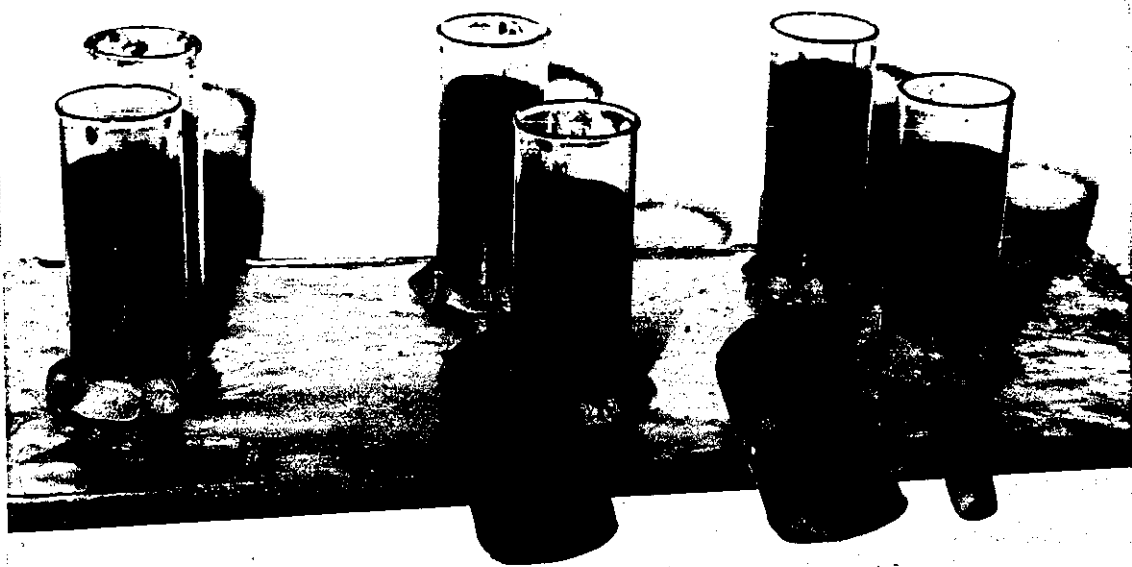


FIGURE 10 Manufacture of P.V.C. "Tools"

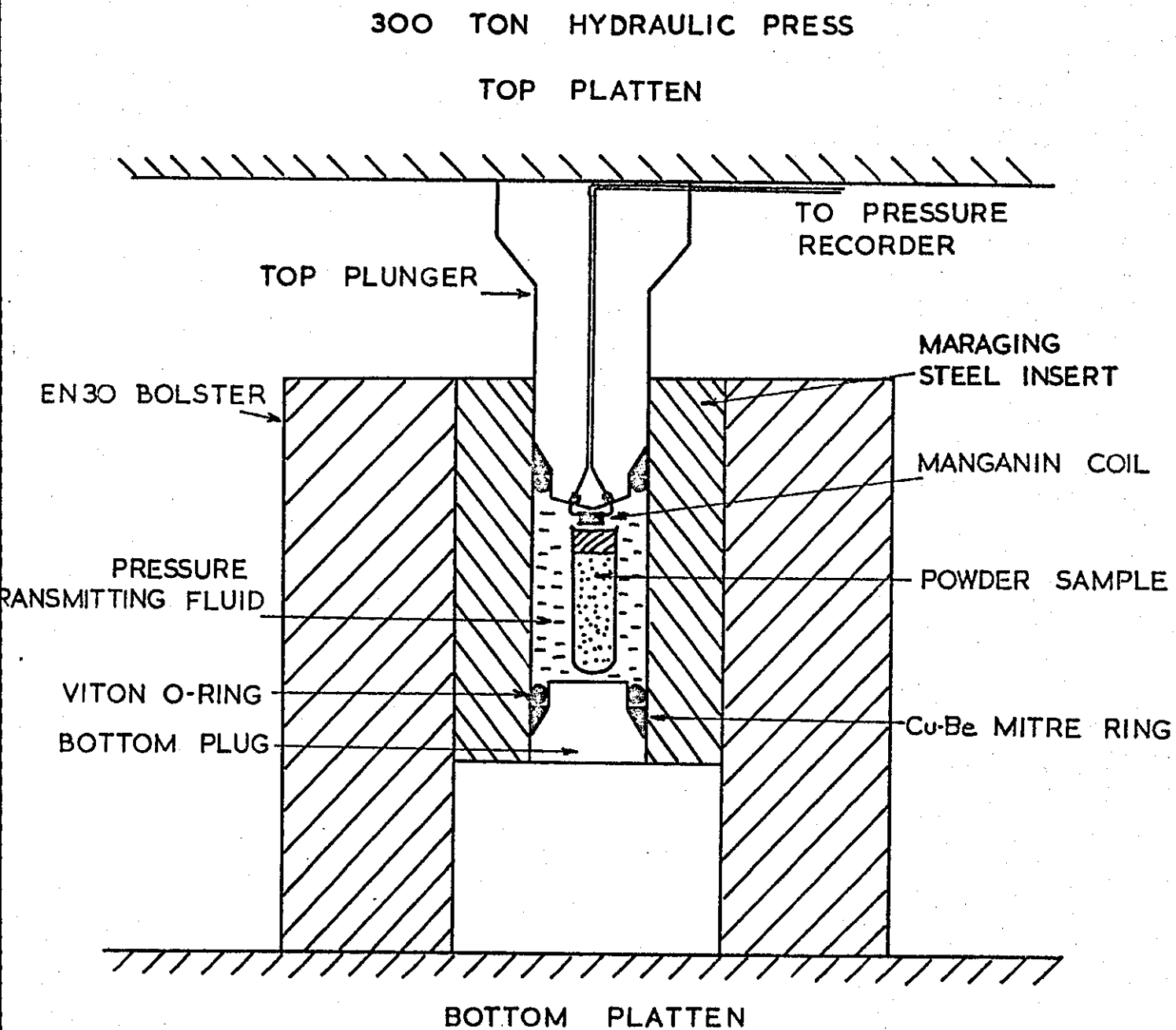


FIGURE 11 Compound Die Set

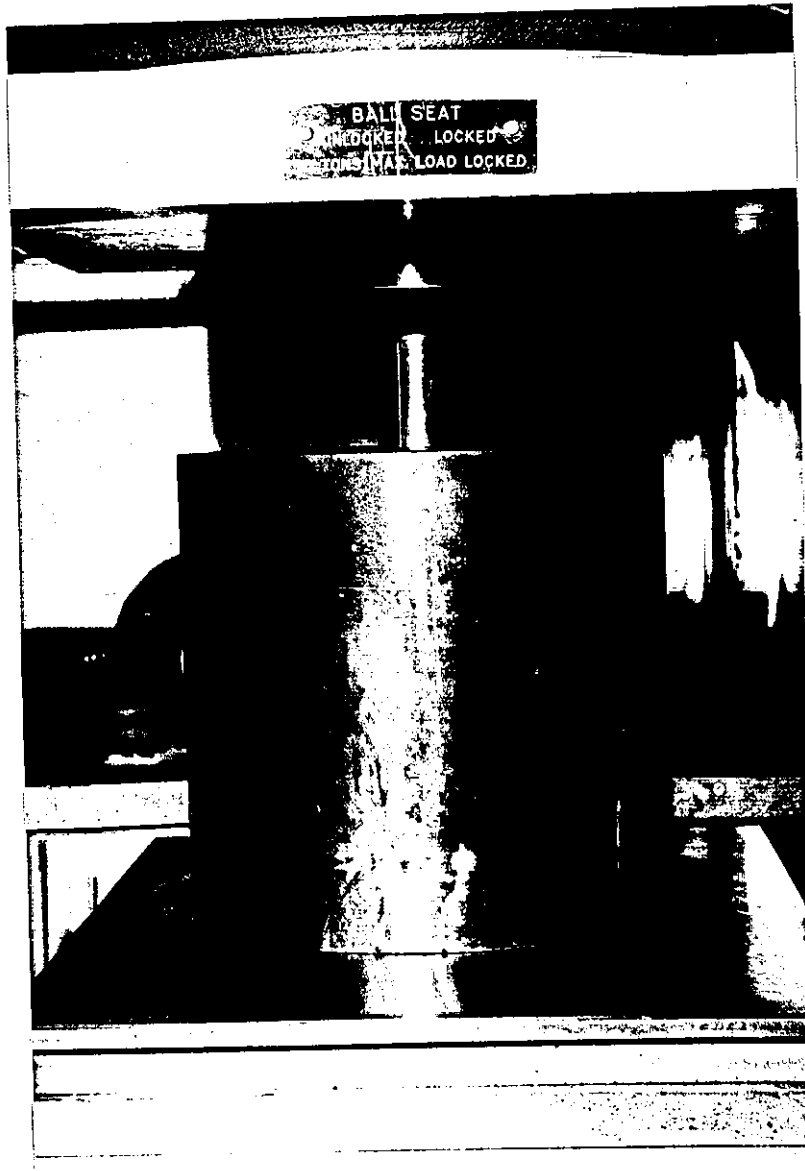


FIGURE 12 Compound Die Set positioned in
Denison Press

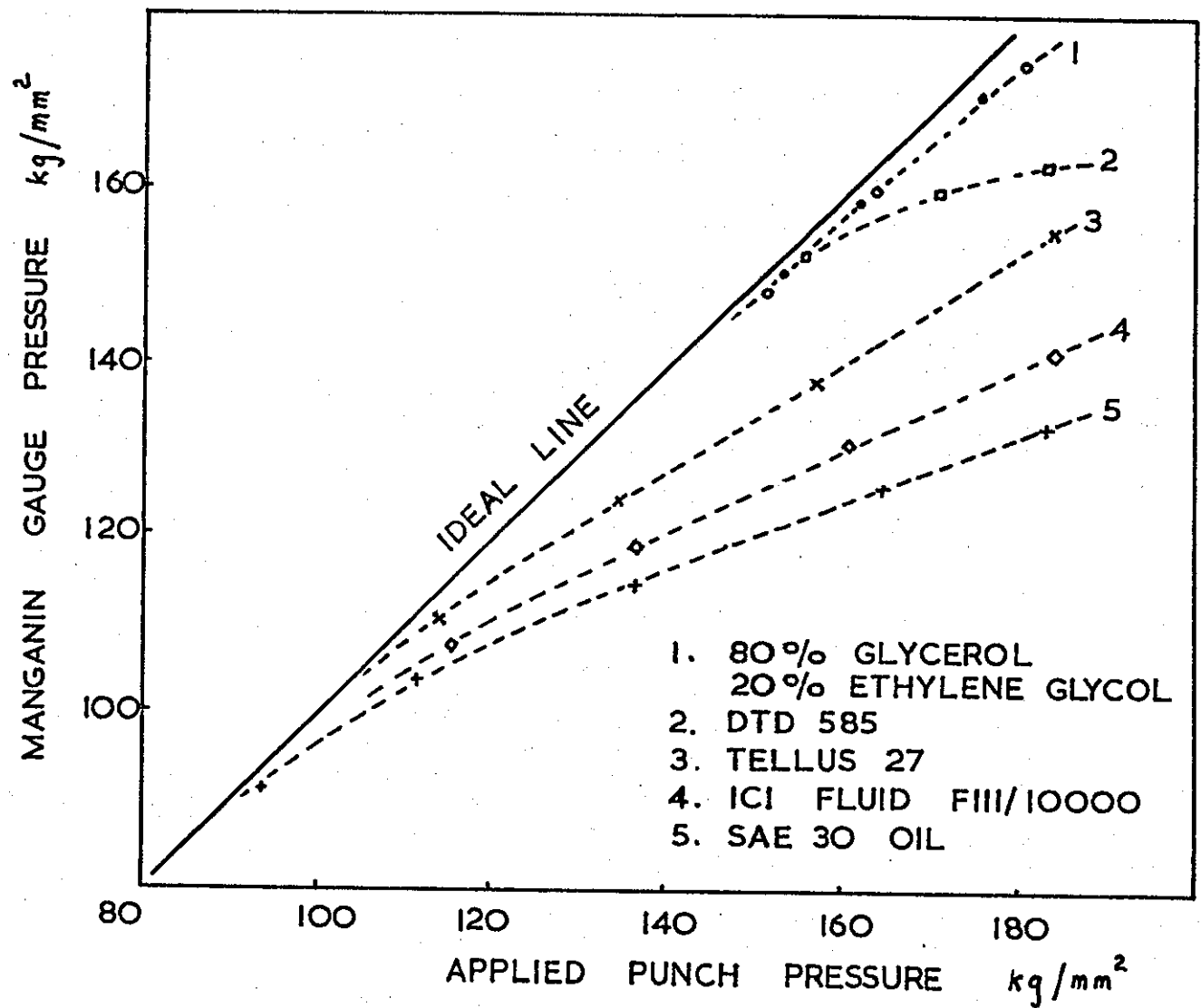


FIGURE 13 Behaviour of various pressure transmitting fluids
(Lowe and Goold ref. 19)

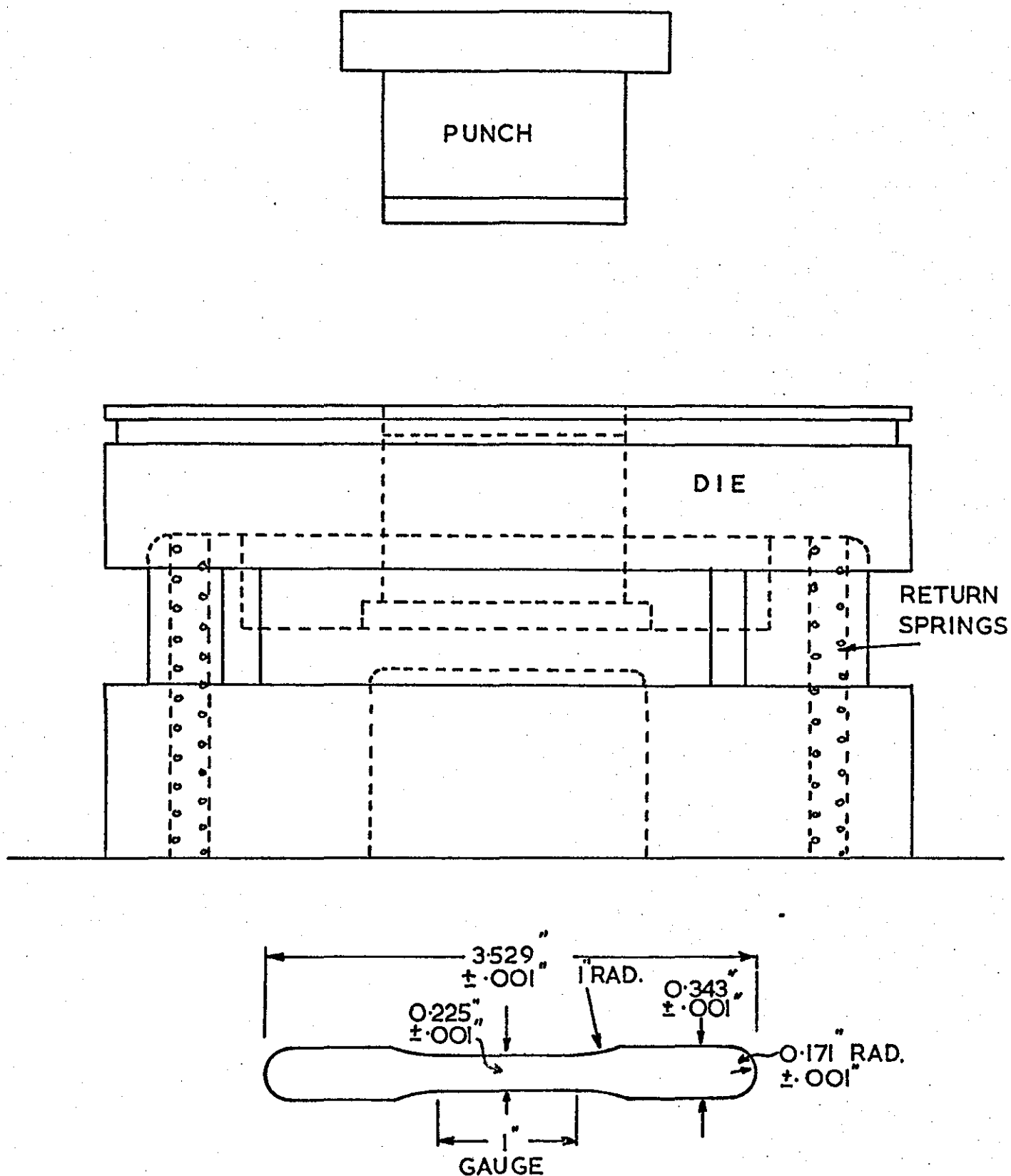


FIGURE 14 Dog-Bone Die Set and Specimen Geometry

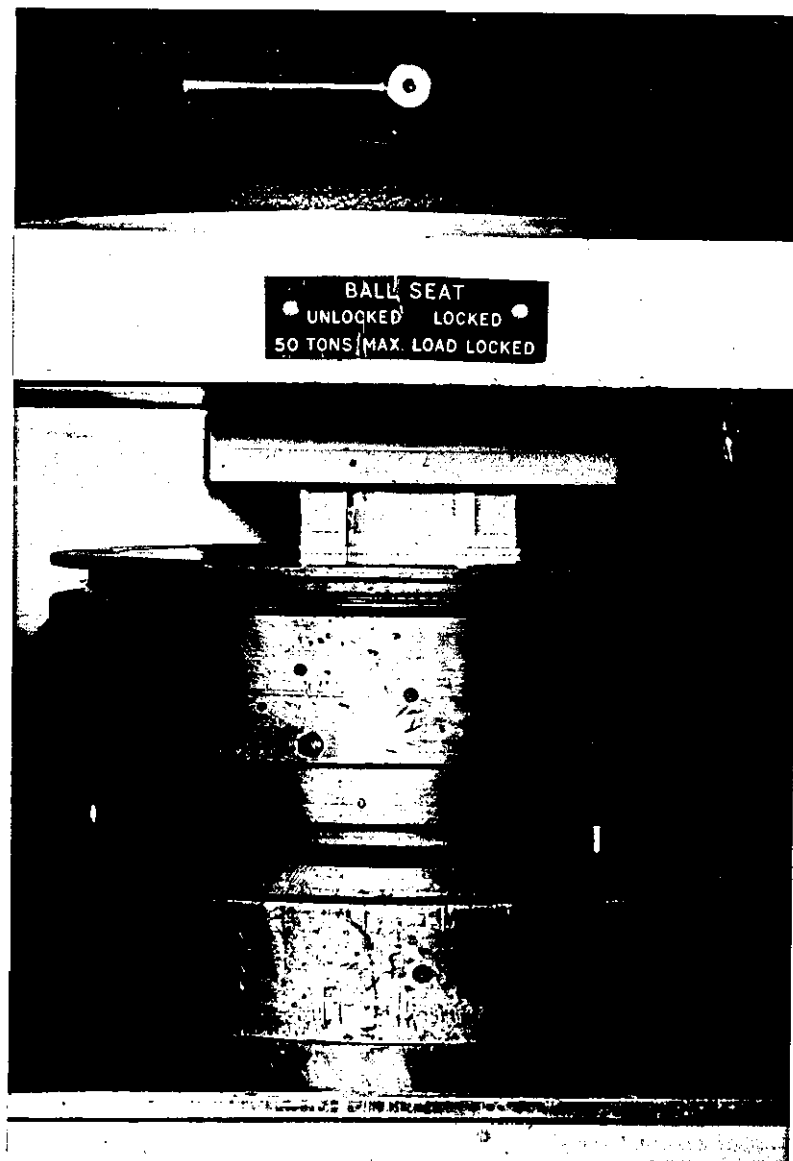


FIGURE 15 Dog-Bone Die Set positioned in
Denison Press

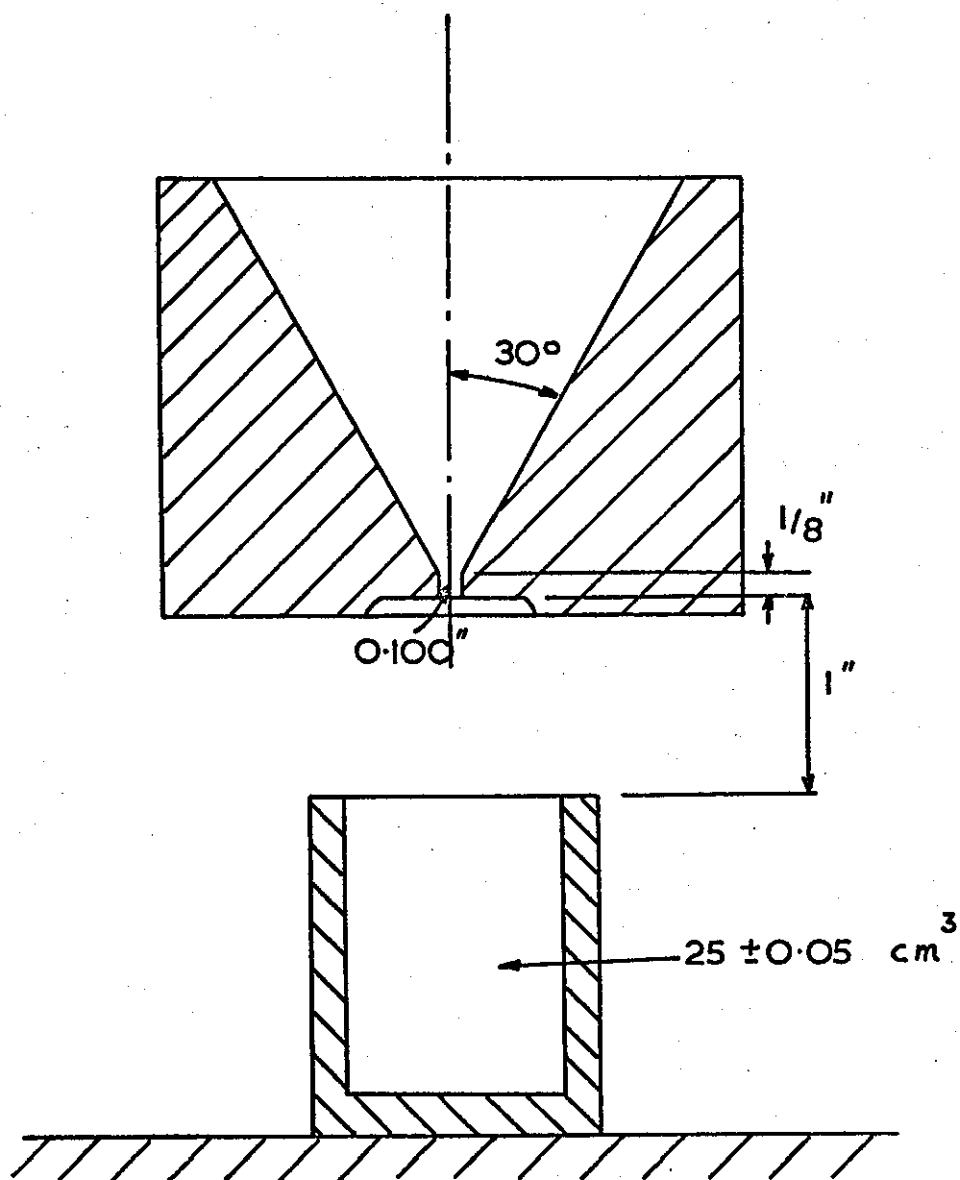


FIGURE 16 Hall Flowmeter and Density Cup

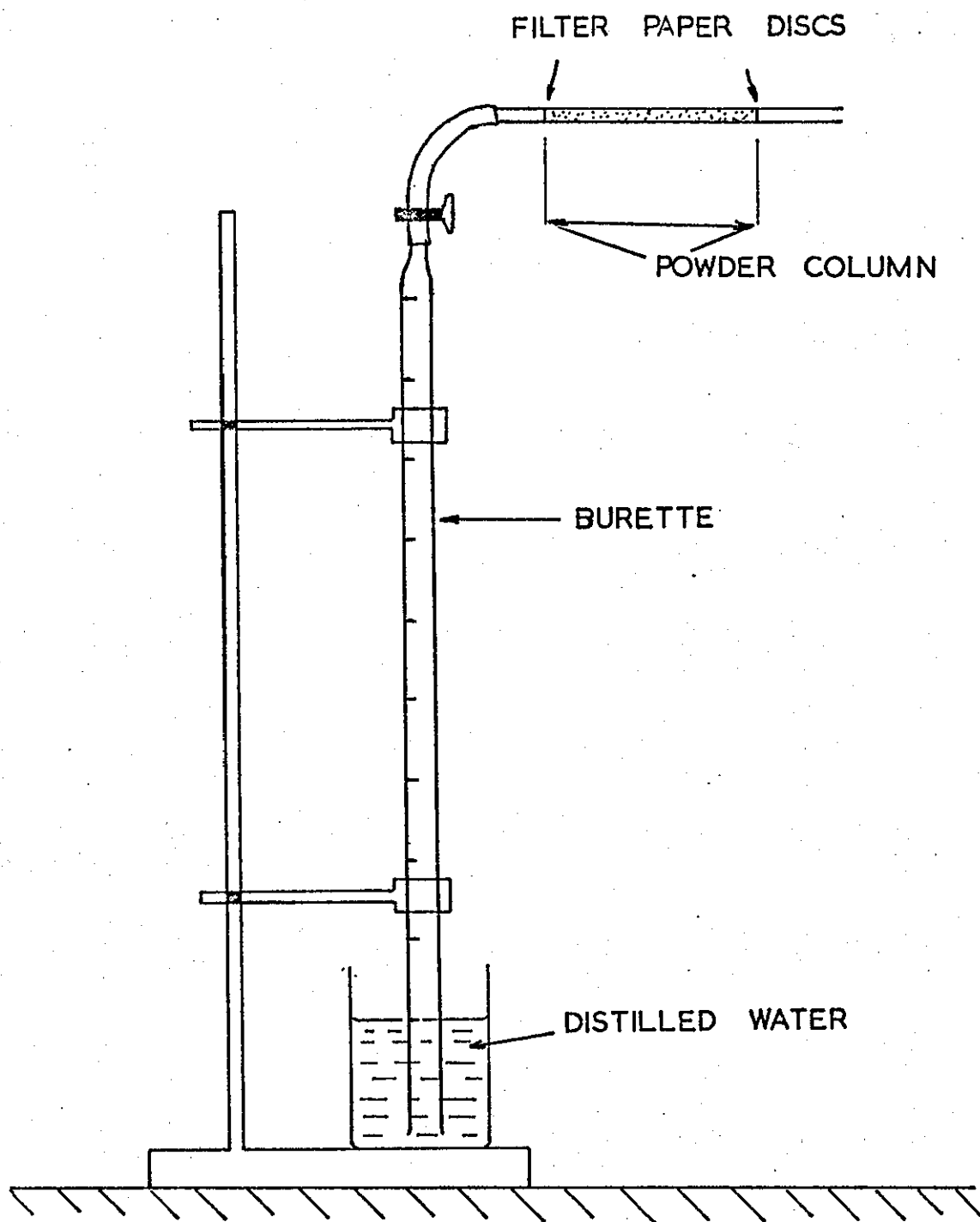


FIGURE 17 Air Permeameter

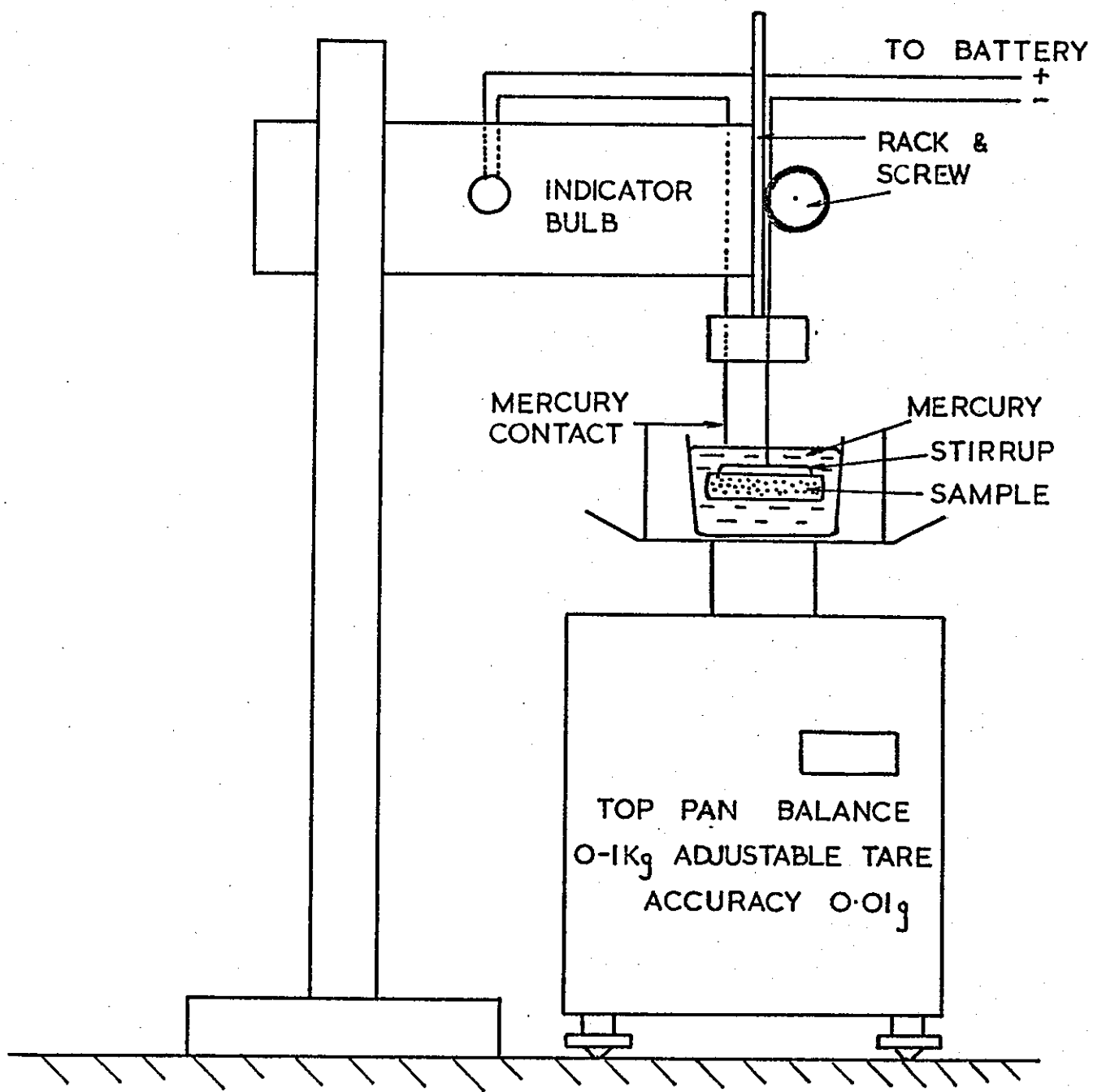


FIGURE 18 Mercury Pycnometer

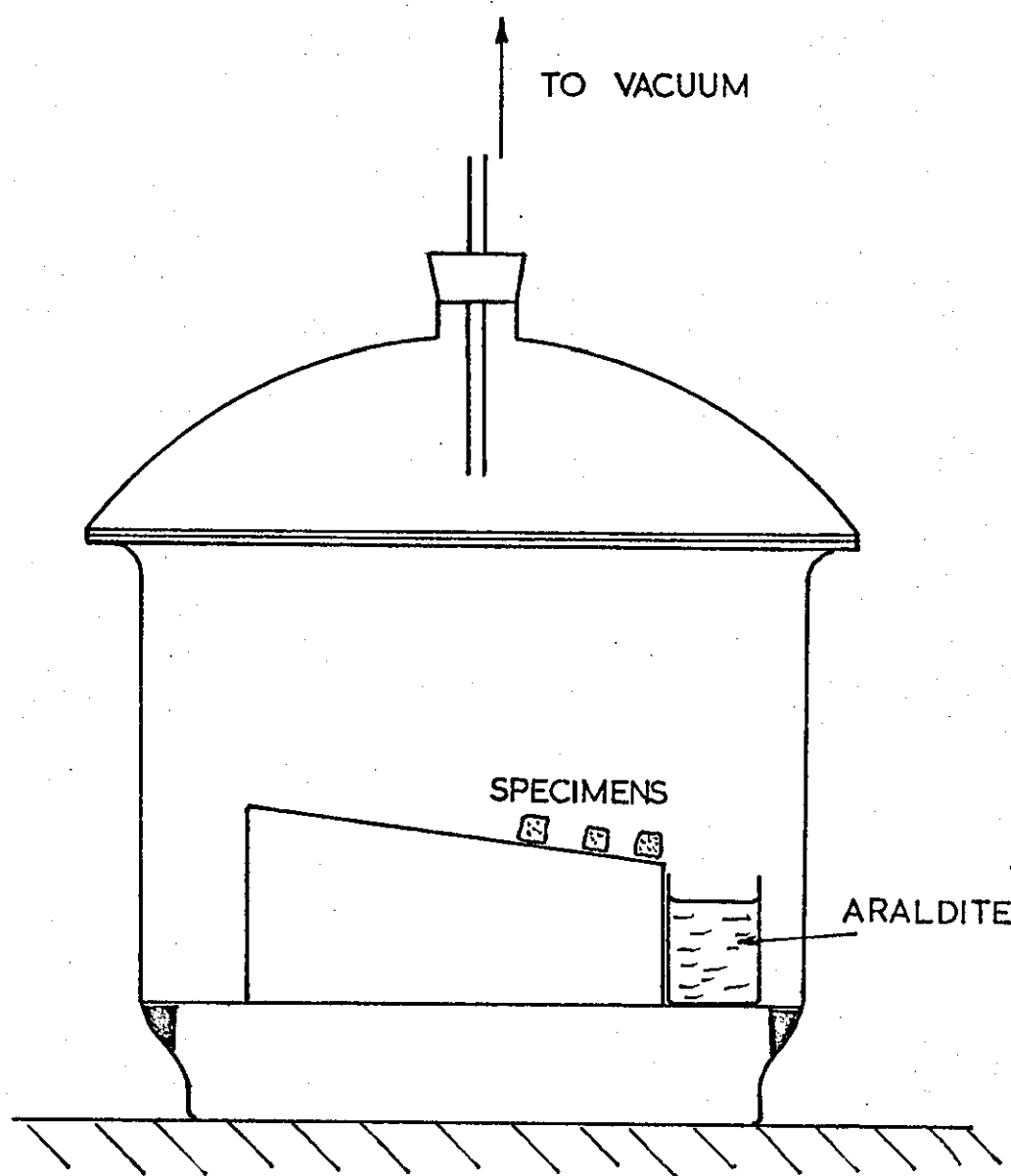


FIGURE 19 Vacuum Dessicator for resin impregnation

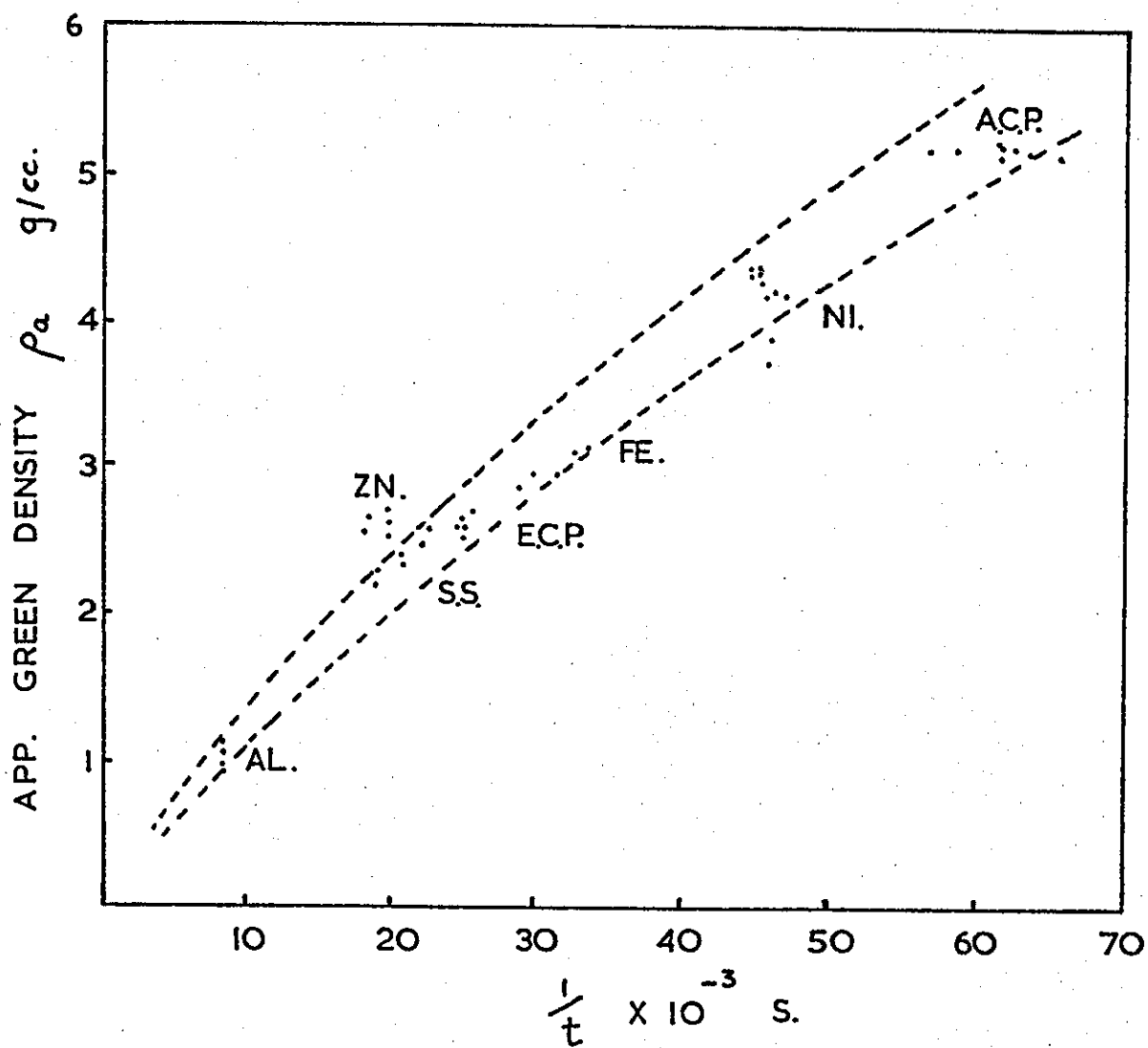


FIGURE 20 Relationship between flow rates and apparent densities

FIGURES 21 - 23

Specific surface area versus Particle Size

- FIGURE 21 S.G. Nickel A.C.P. & E.C.P.
FIGURE 22 Stainless Steel, Iron & Zinc
FIGURE 23 Aluminium

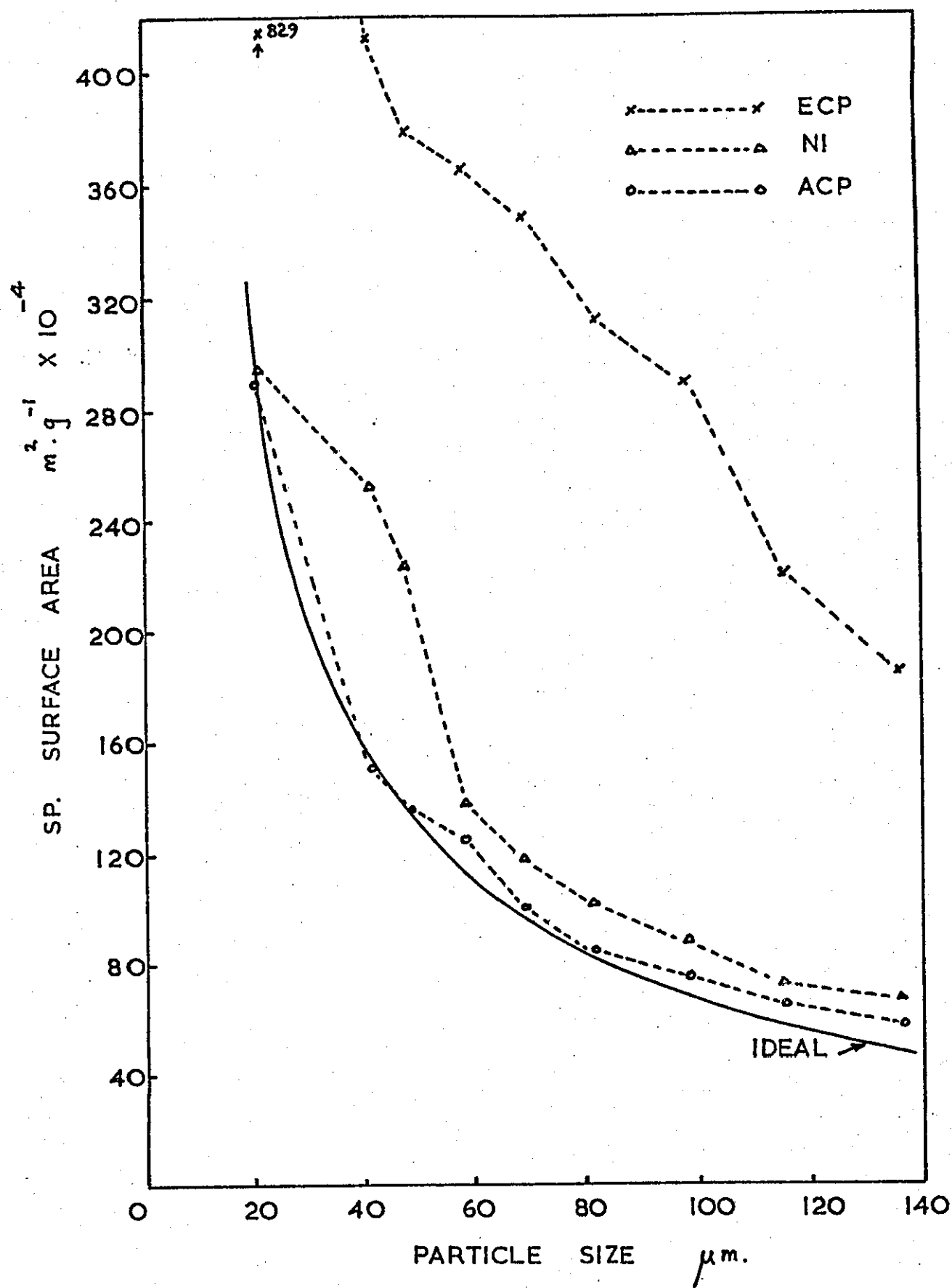


FIGURE 21

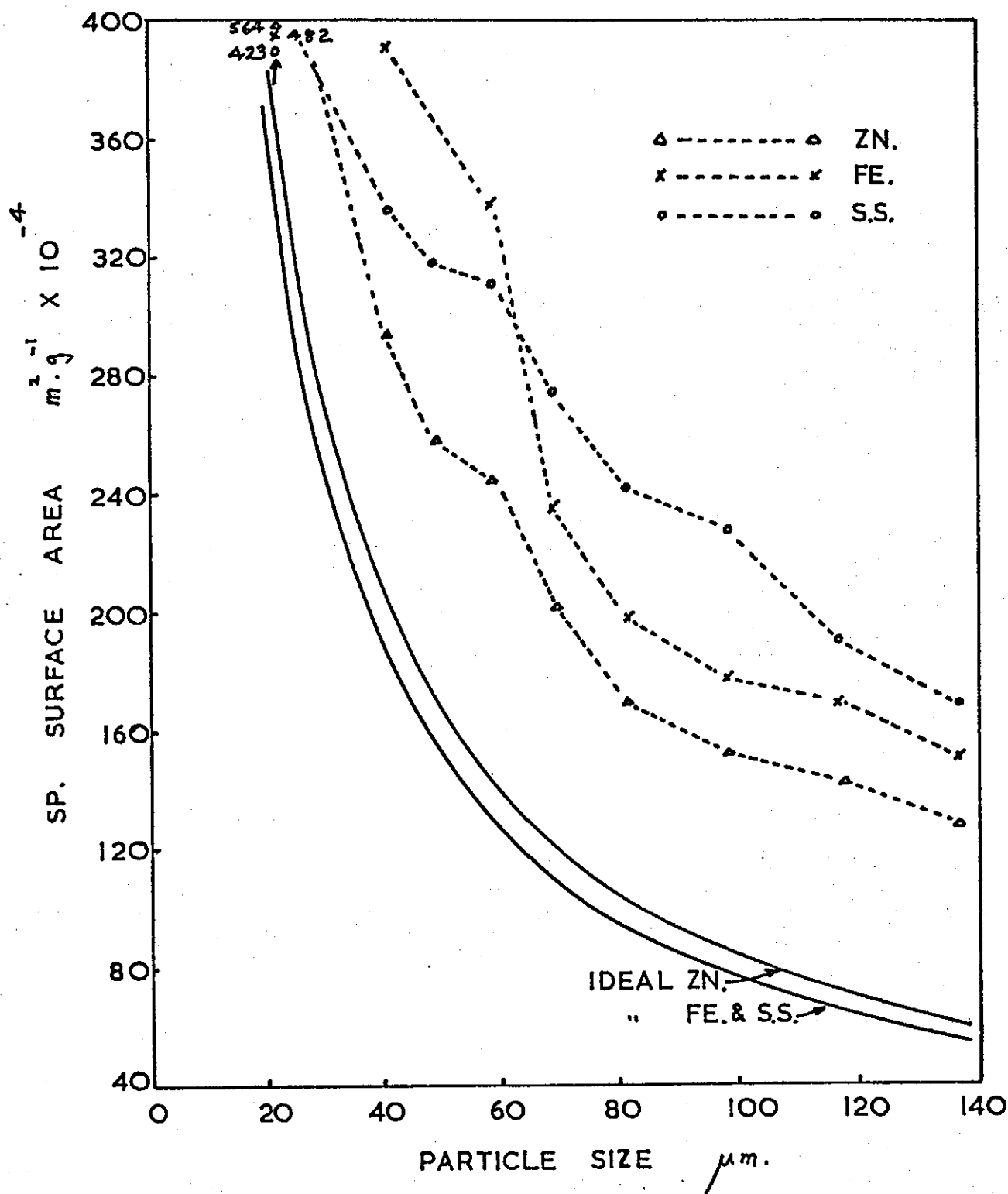


FIGURE 22

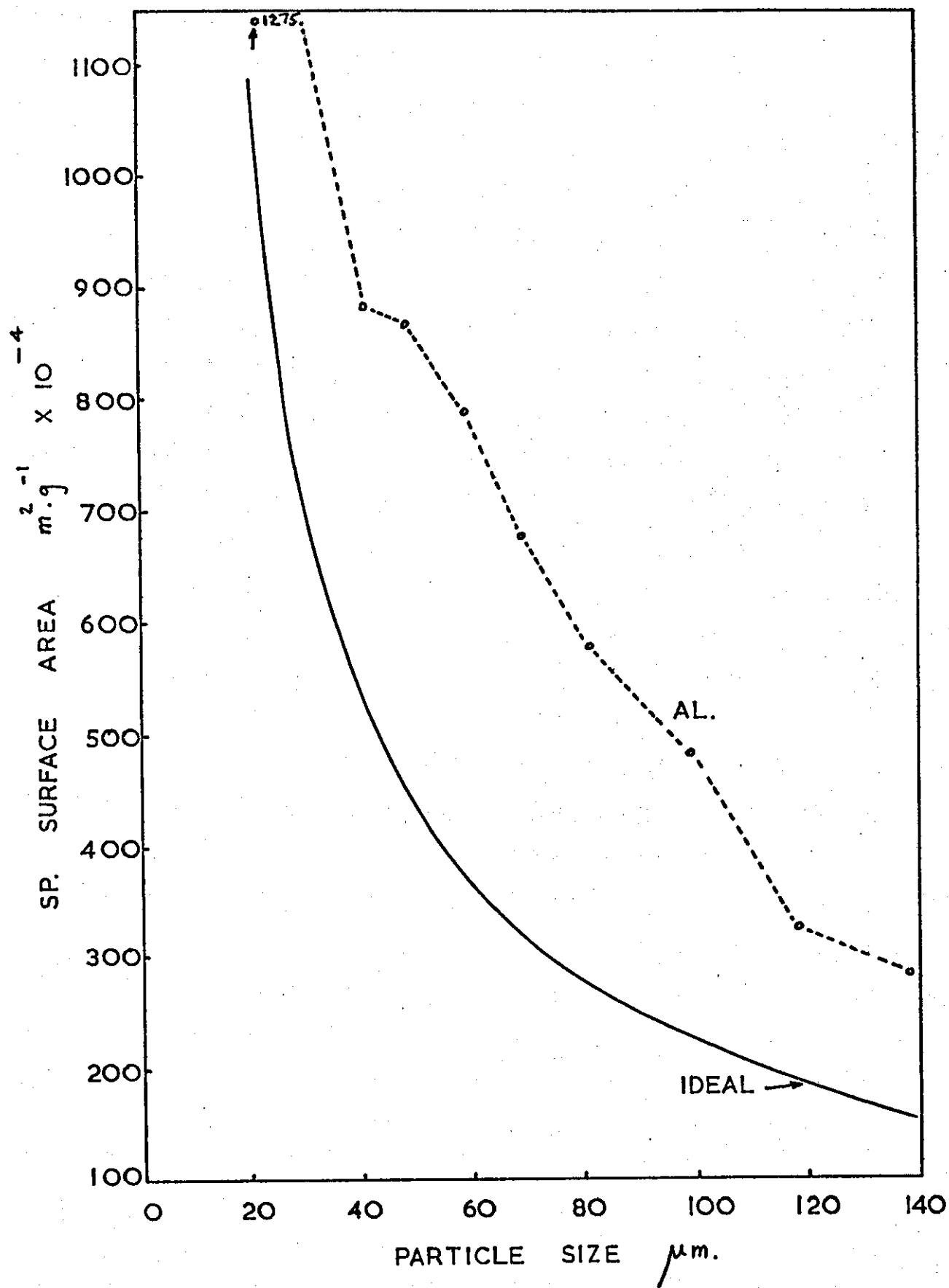


FIGURE 23

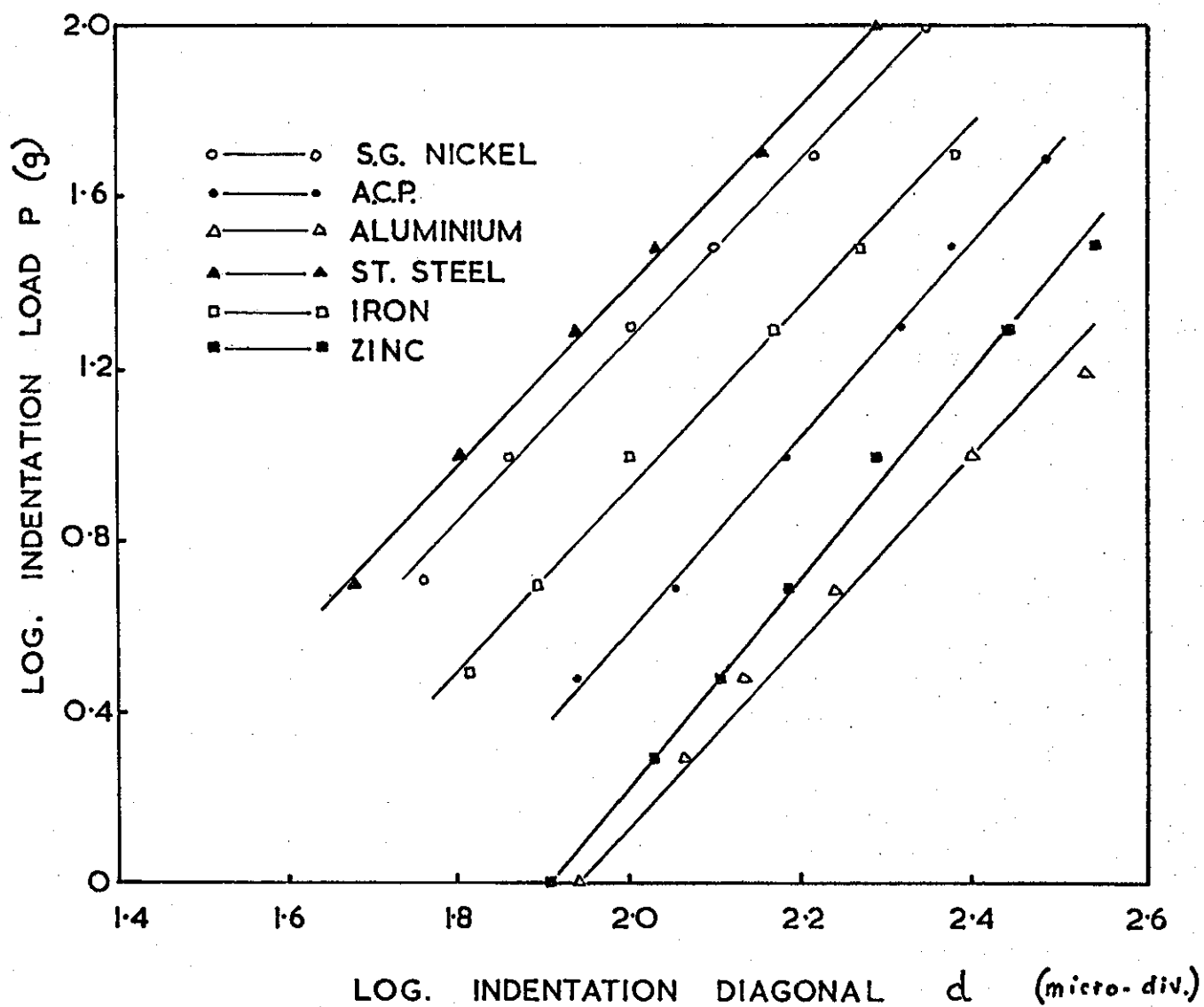


FIGURE 24

FIGURES 25 - 28

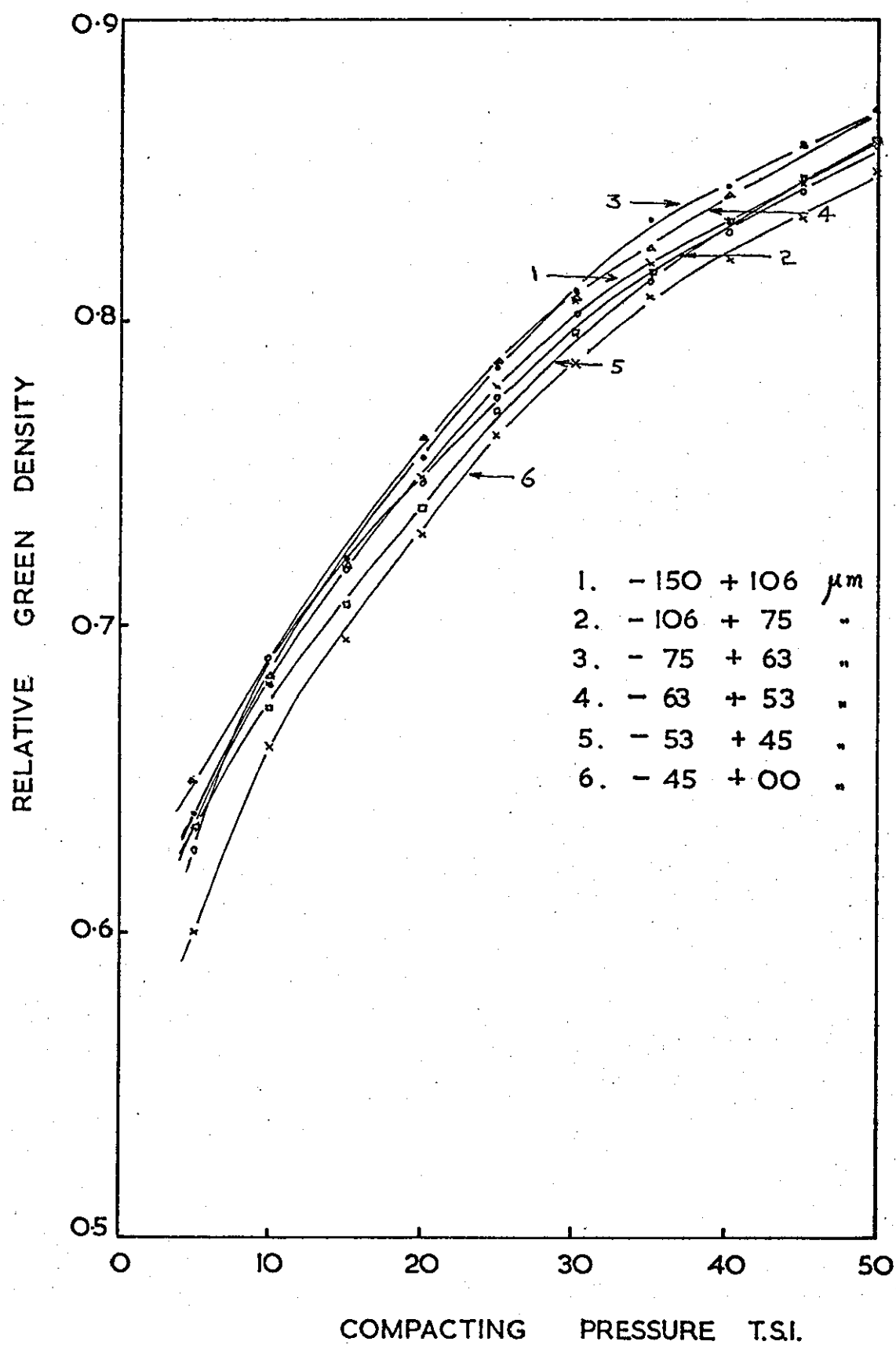
Density - Pressure relationships of multi-size
fractions of powders

FIGURE 25 S.G. Nickel

FIGURE 26 Iron

FIGURE 27 E.C.P.

FIGURE 28 Comparison between A.C.P. and E.C.P.



. FIGURE 25

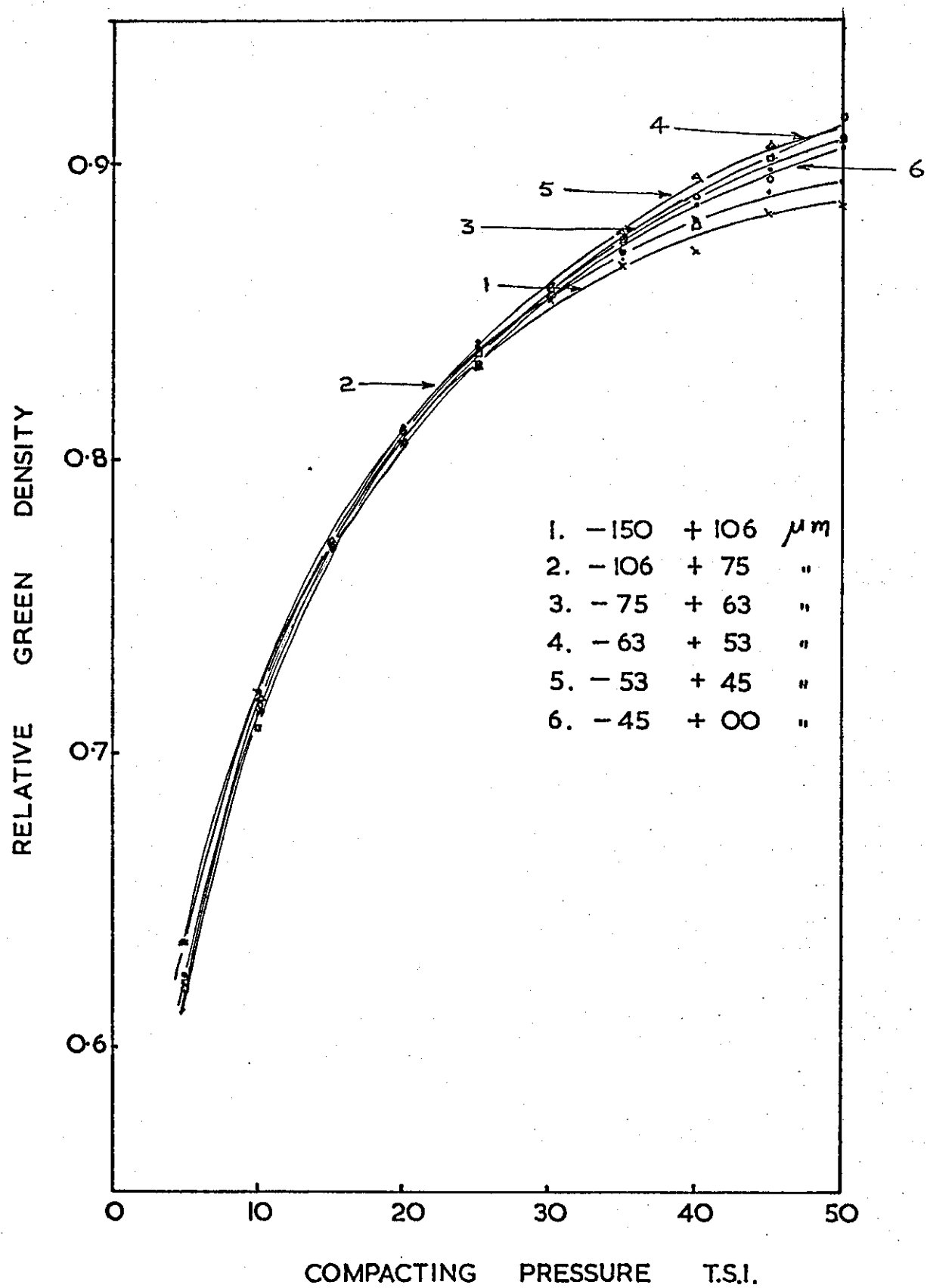


FIGURE 26

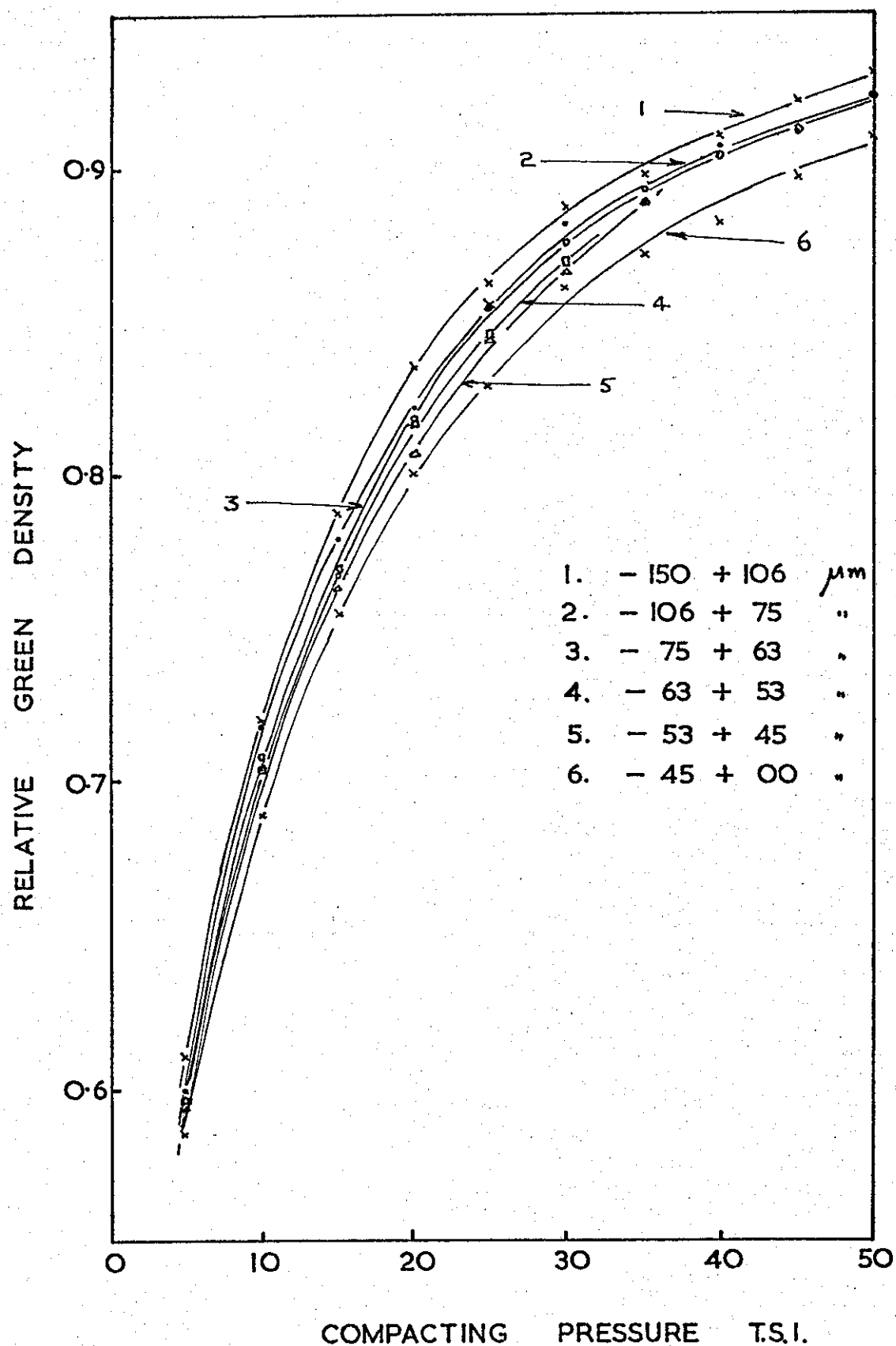


FIGURE 27

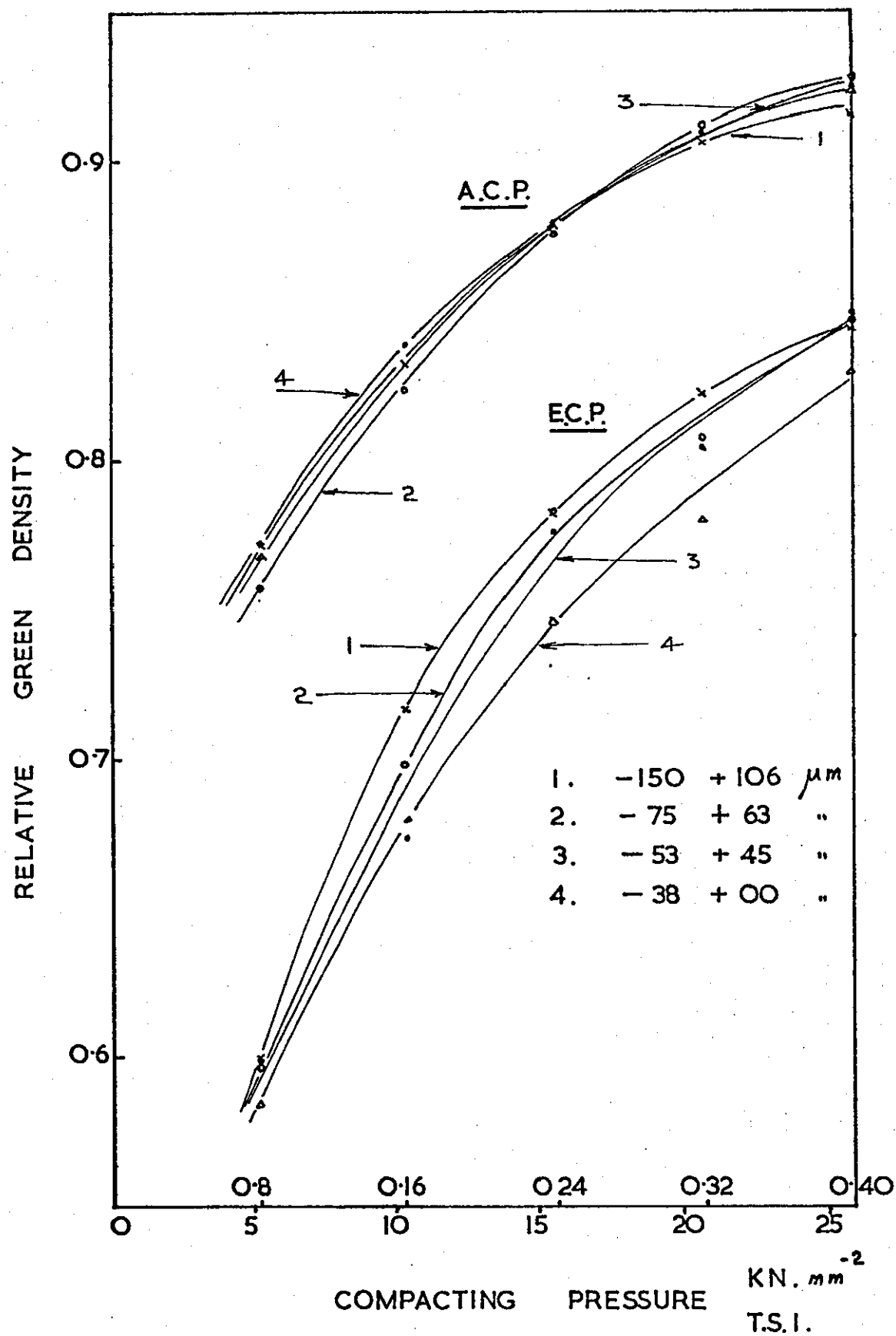


FIGURE 28

FIGURES 29 - 34

Comparisons of Density versus Pressure for a
monosize fraction ($-125 + 106\mu\text{m}$) of metal powders
in non de-aired (N.D.A) and de-aired (D.A) conditions.

FIGURE 29 S.G. Nickel

FIGURE 30 A.C.P.

FIGURE 31 Aluminium

FIGURE 32 Stainless Steel

FIGURE 33 Iron

FIGURE 34 Zinc

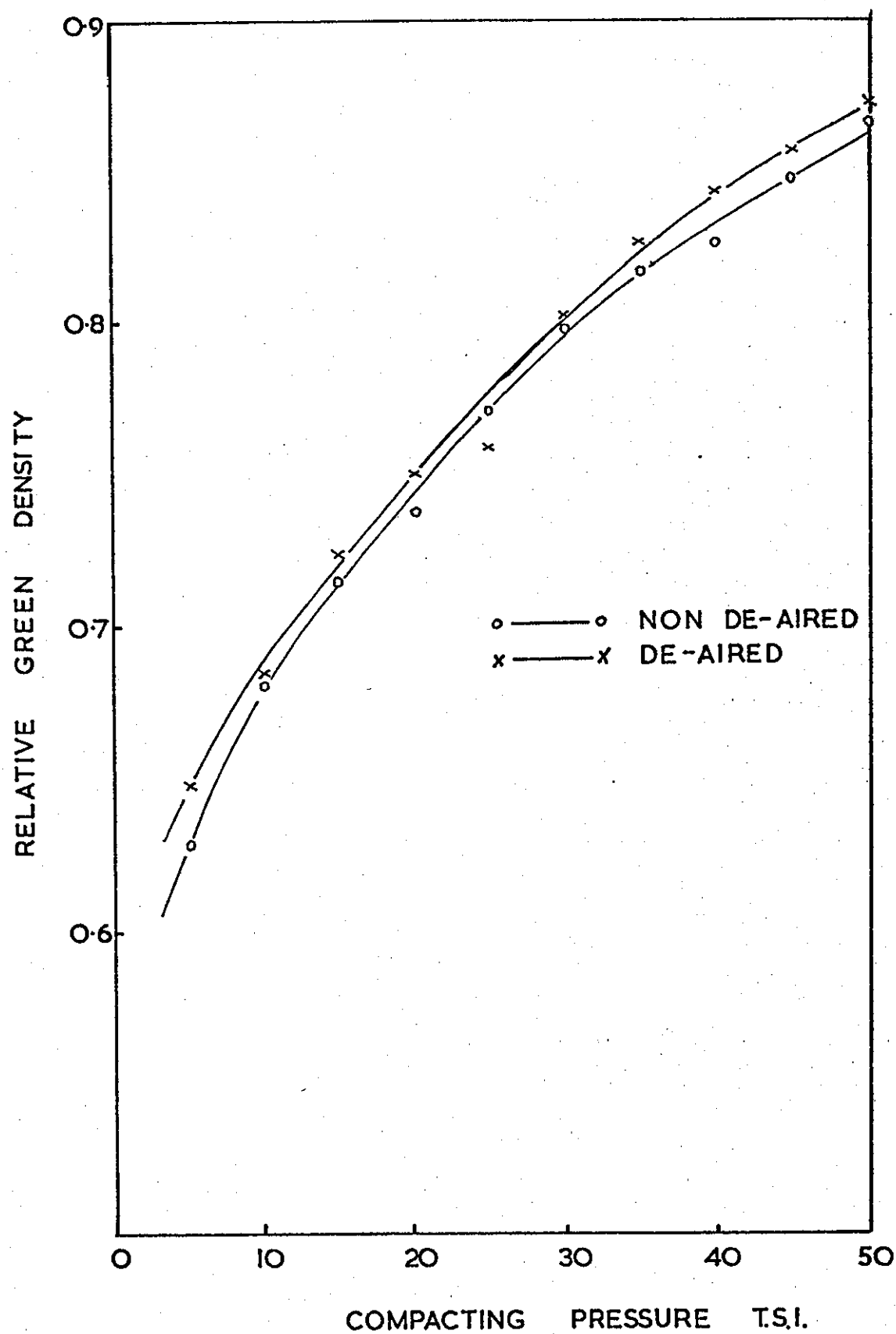


FIGURE 29

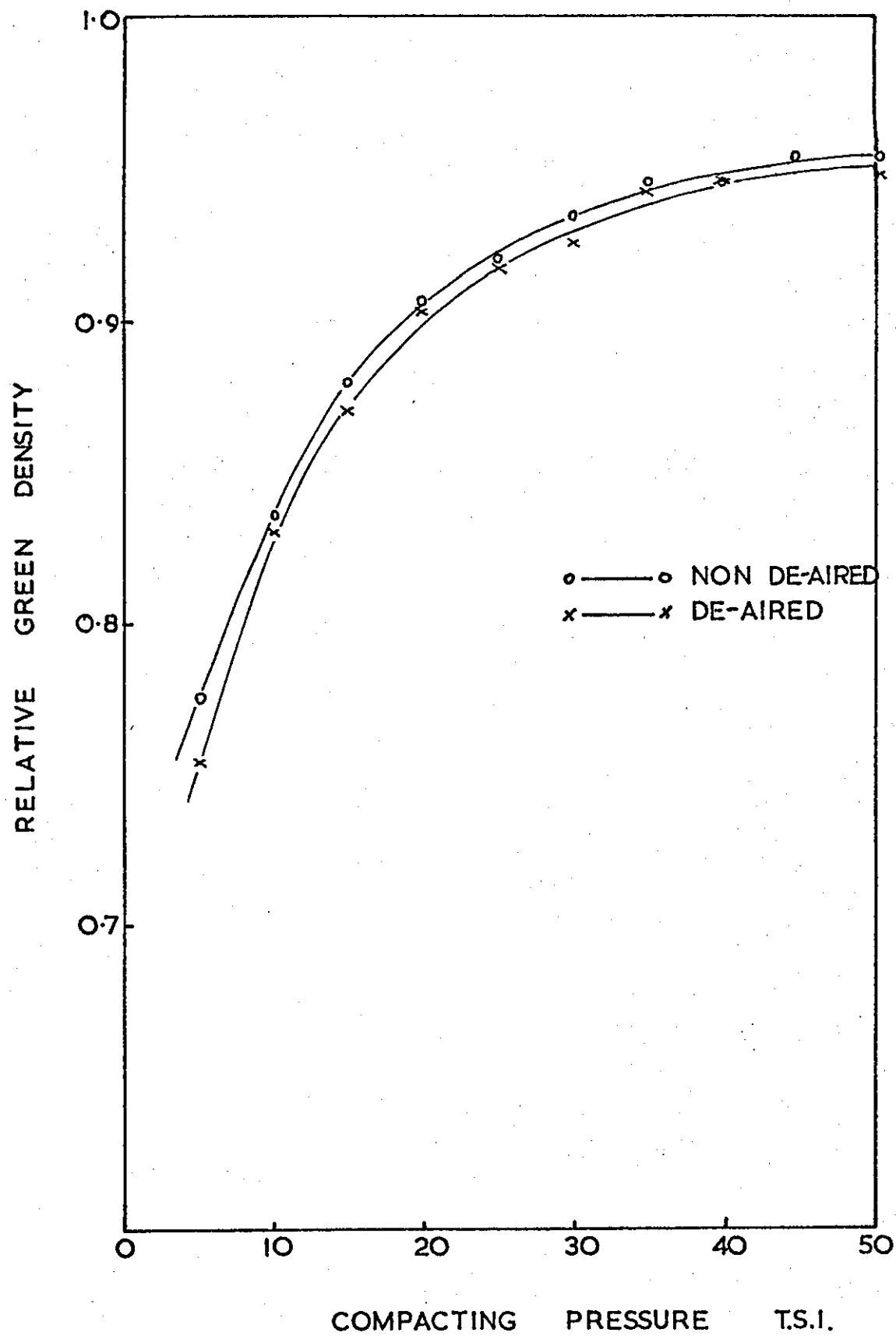


FIGURE 30

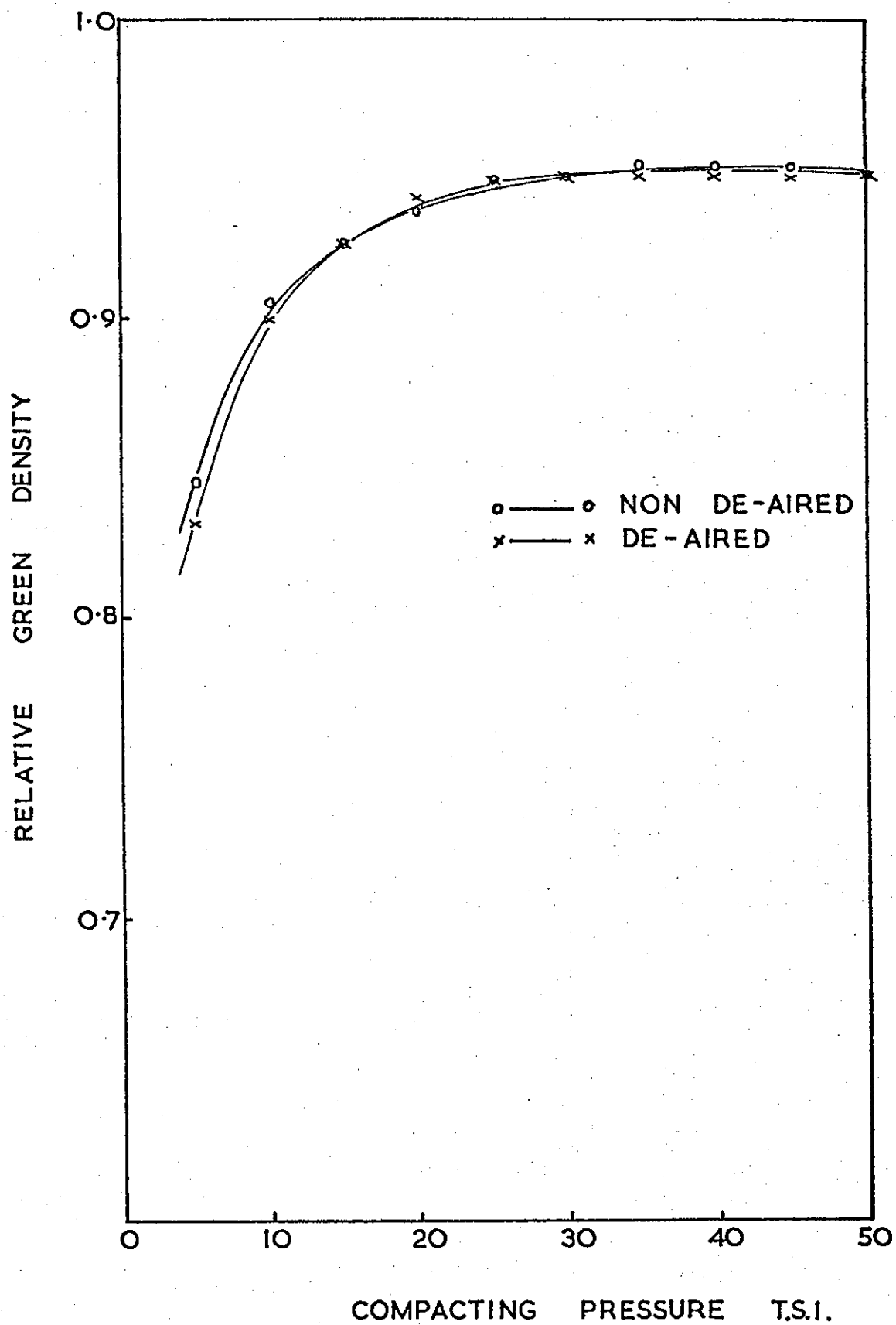


FIGURE 31

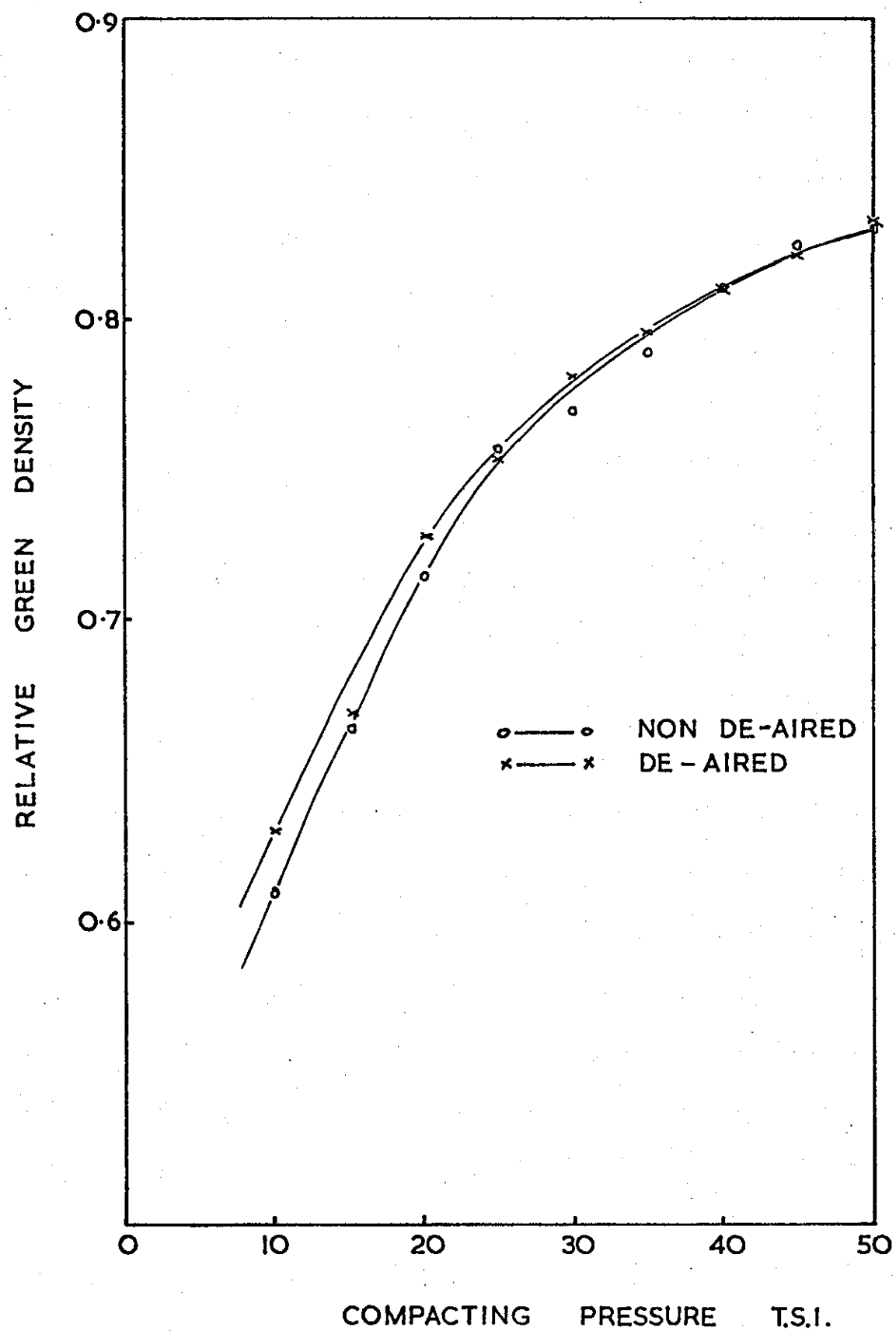


FIGURE 32

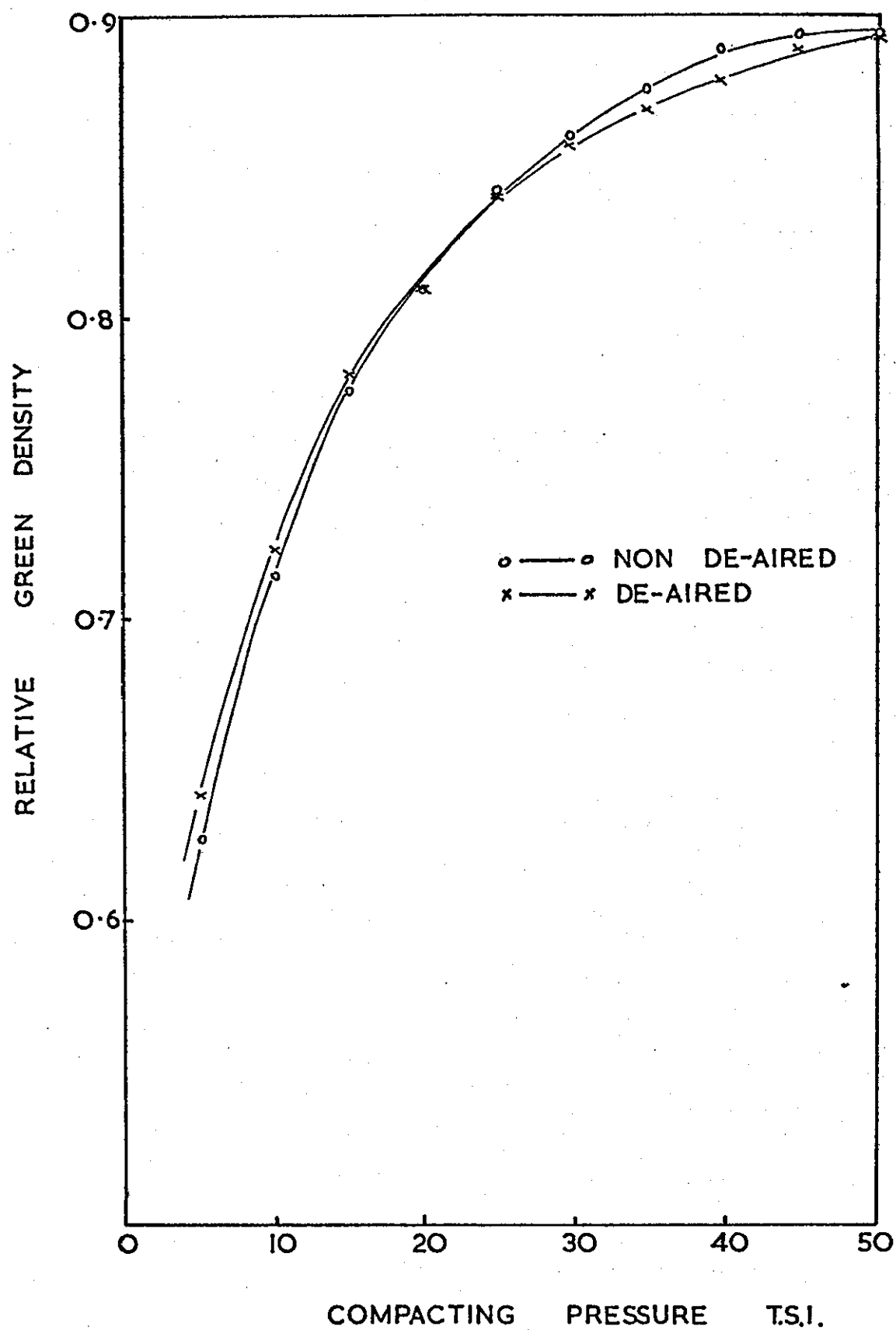


FIGURE 33

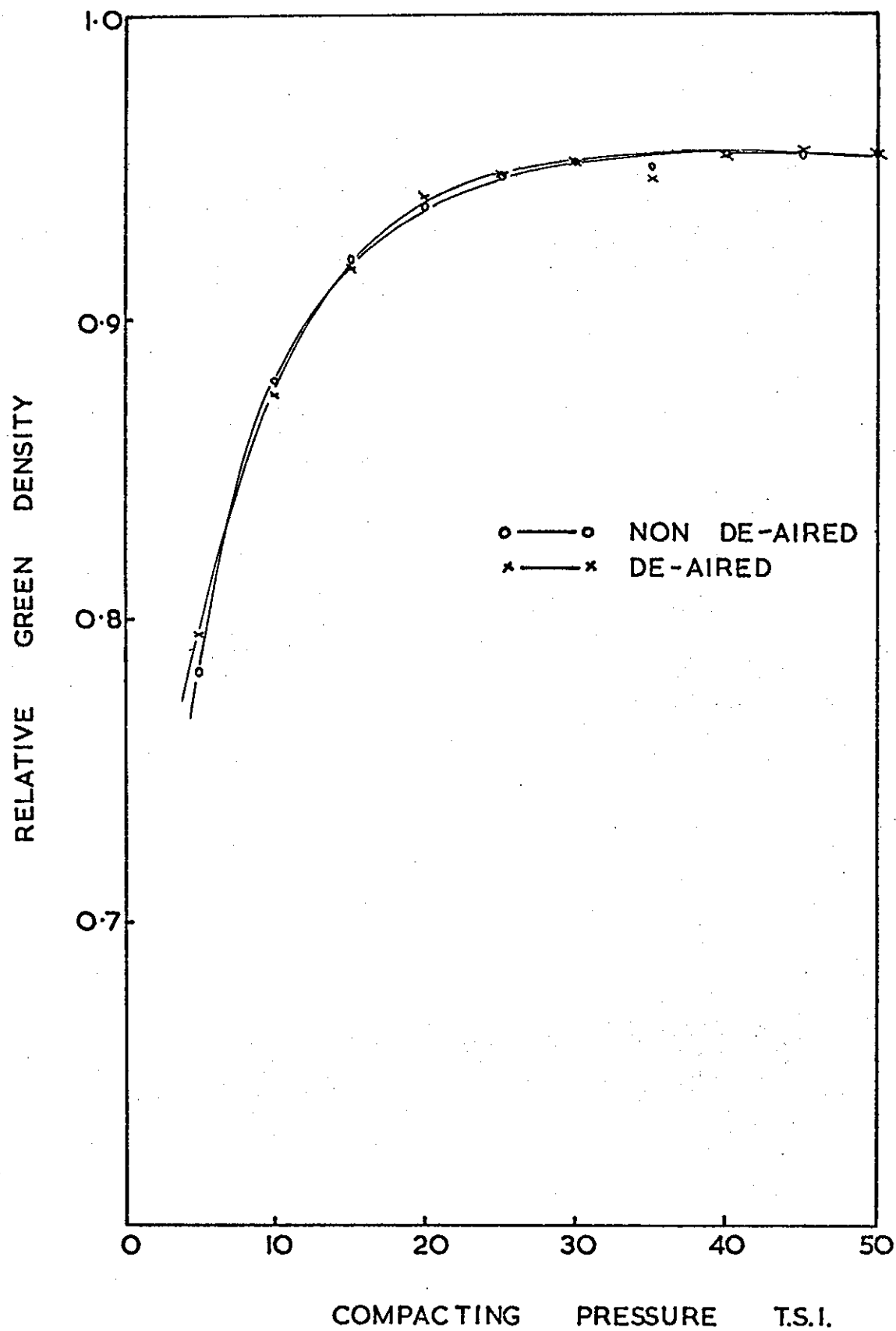


FIGURE 34

FIGURES 35 - 38

Kawakita relationships :

- FIGURE 35 Monosize fraction (-125 + 106 μ m)
 of powders
- FIGURE 36 Different size fractions of S.G.
 Nickel
- FIGURE 37 Different size fractions of Iron
- FIGURE 38 Different size fractions of E.C.P.

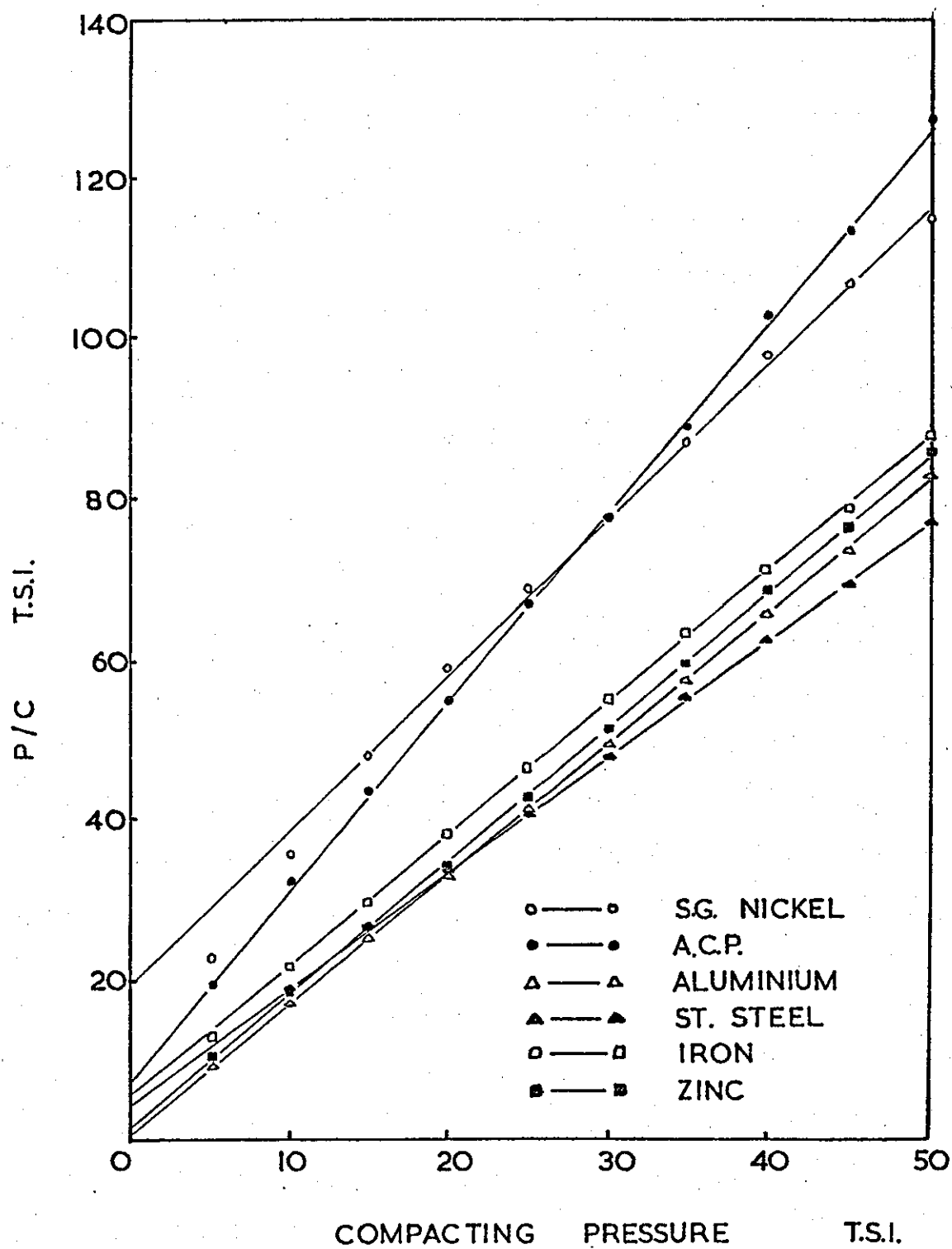


FIGURE 35

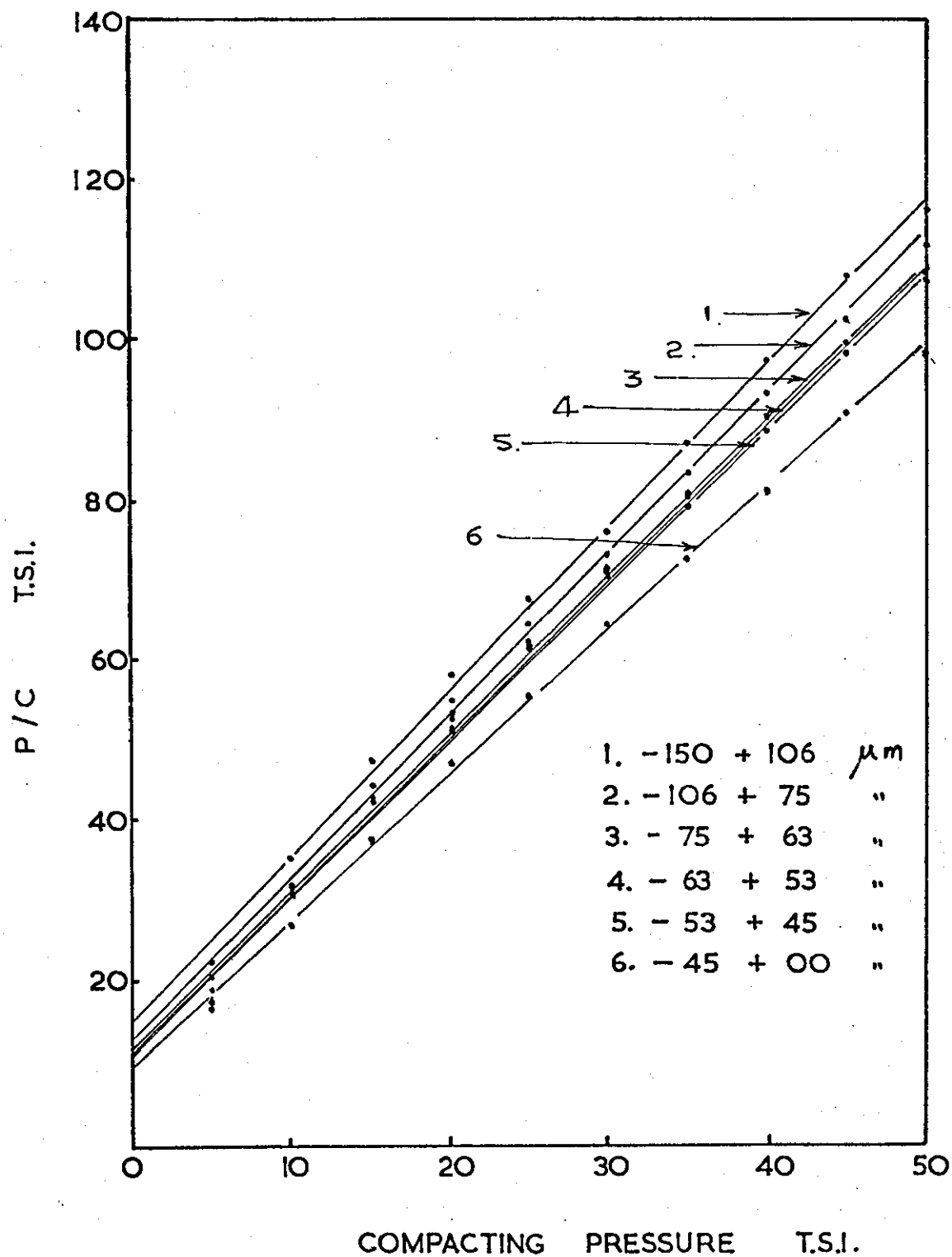


FIGURE 36

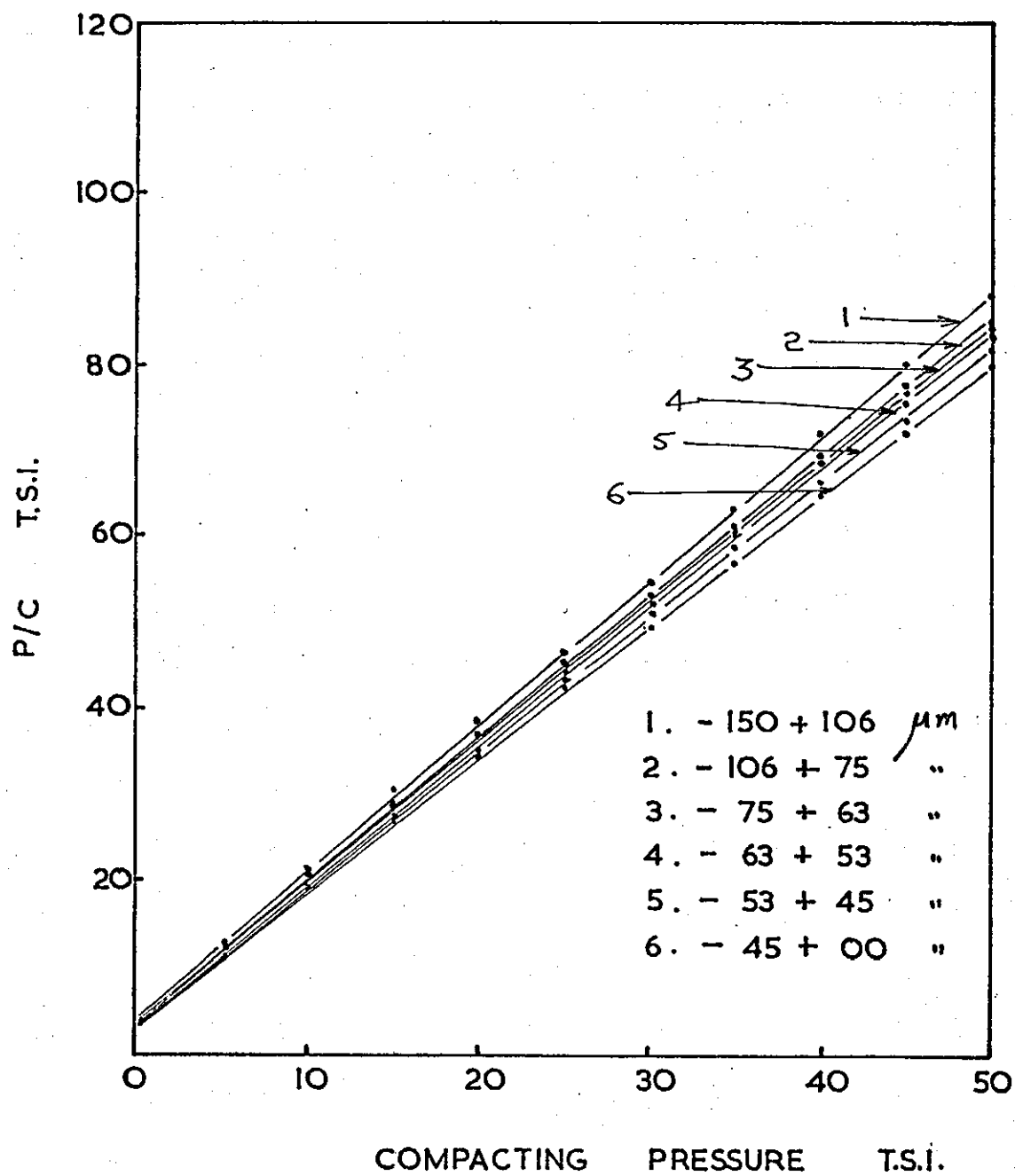


FIGURE 37

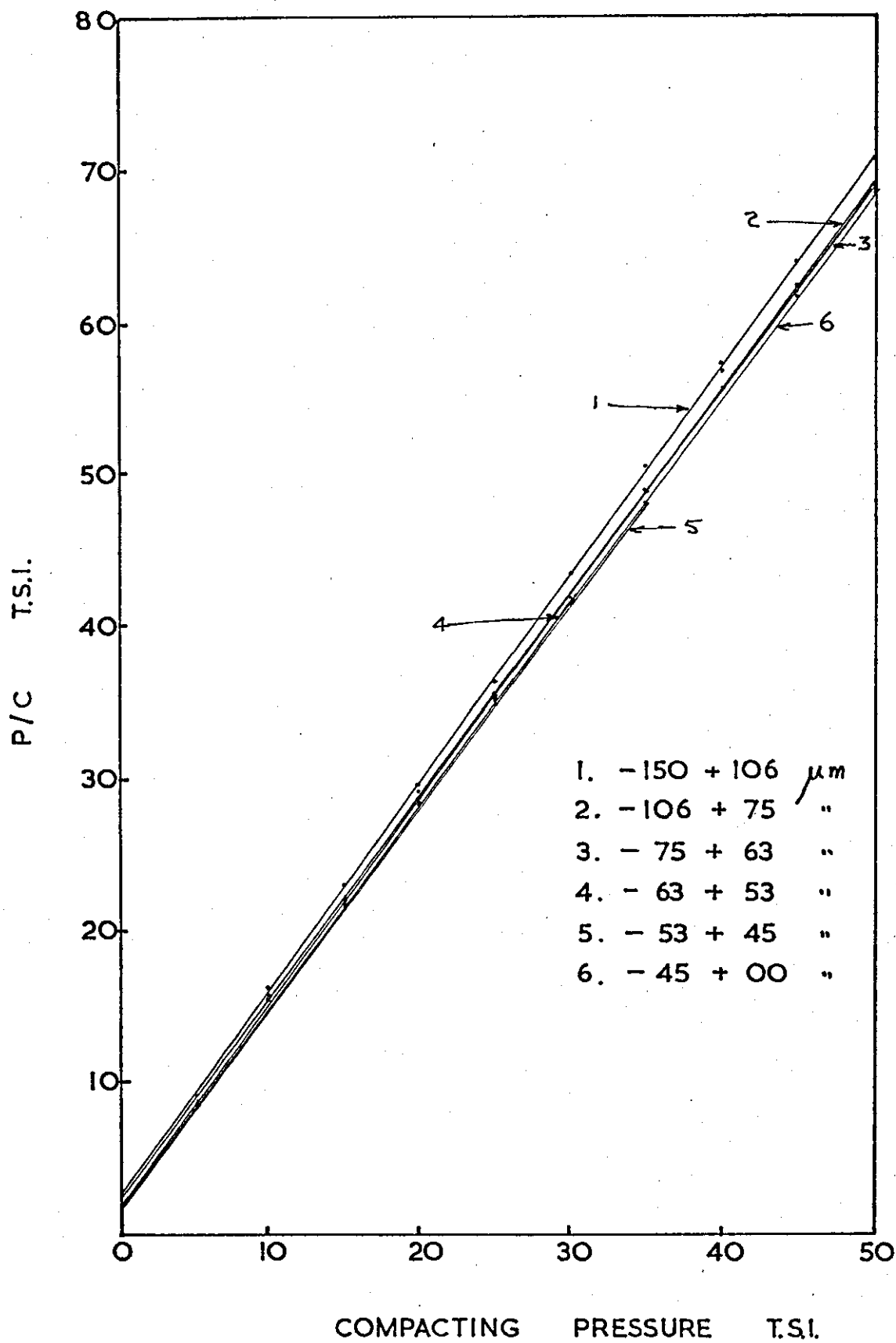


FIGURE 38

FIGURES 39 - 42

Konopicky - Shapiro relationships :

FIGURE 39 Monosize fraction ($-125 + 106\mu\text{m}$)
of powders

FIGURE 40 Different size fractions of S.G. Nickel

FIGURE 41 Different size fractions of Iron

FIGURE 42 Different size fractions of E.C.P.

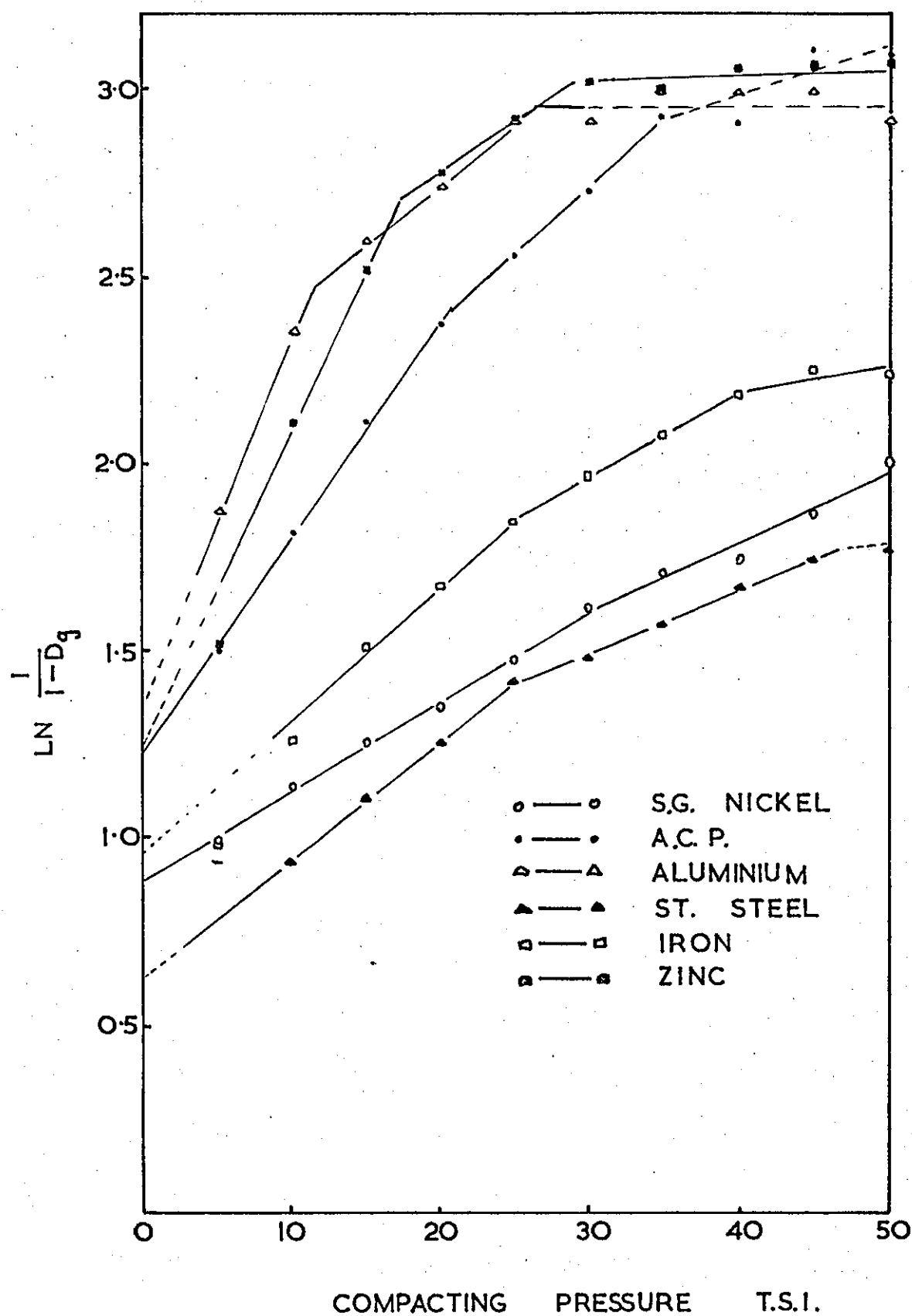


FIGURE 39

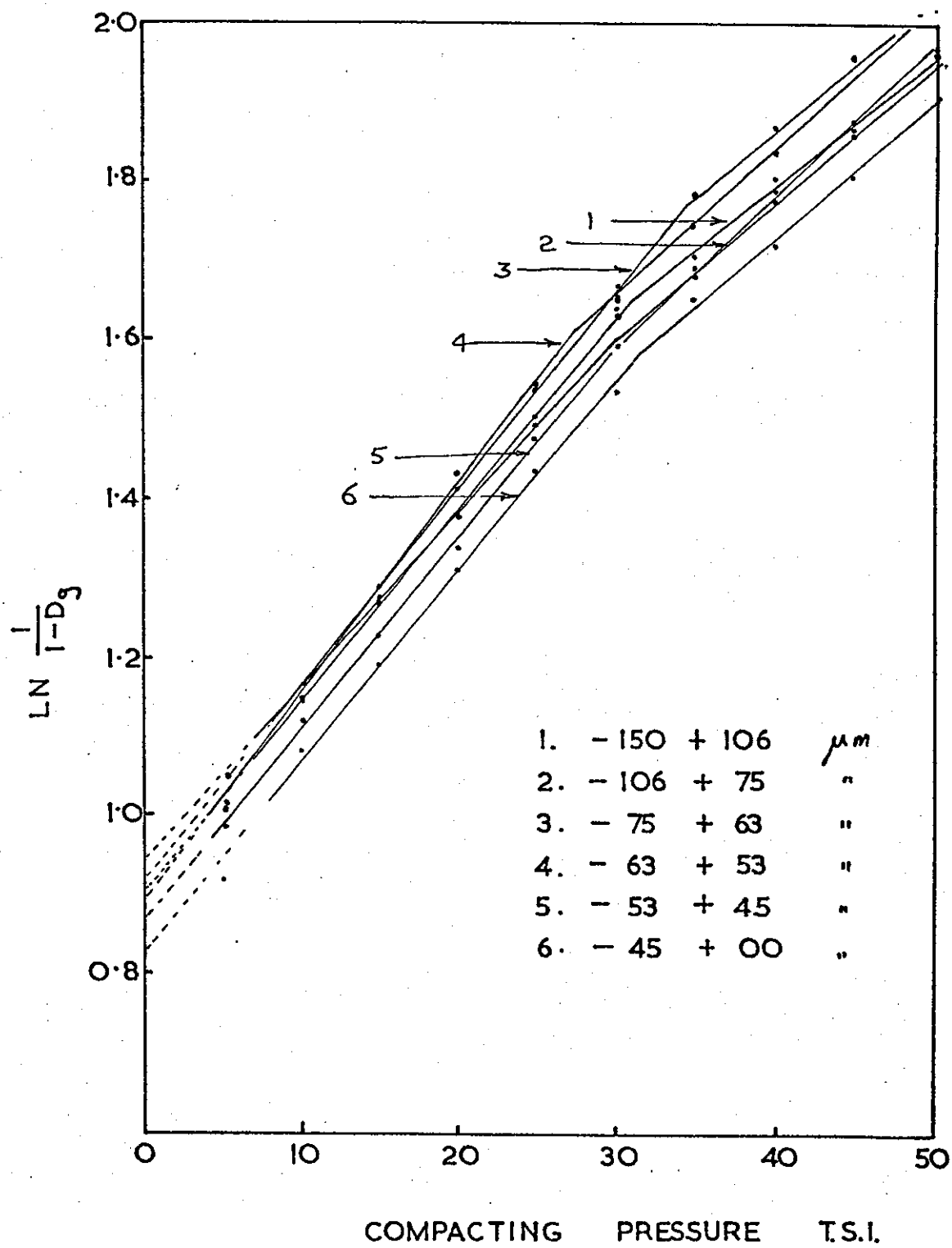


FIGURE 40

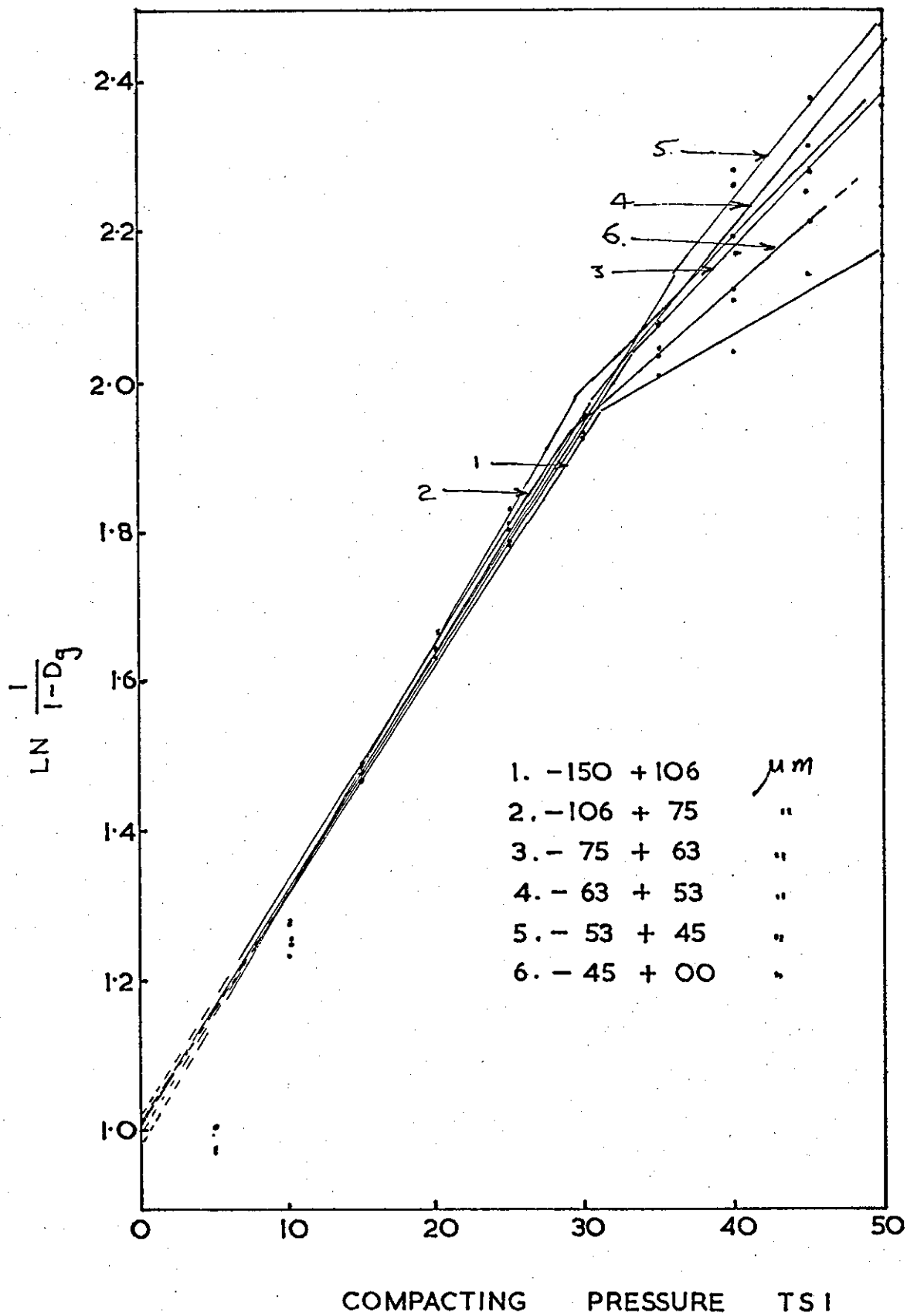


FIGURE 41

MR M J DRAPER
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If undelivered please return to:
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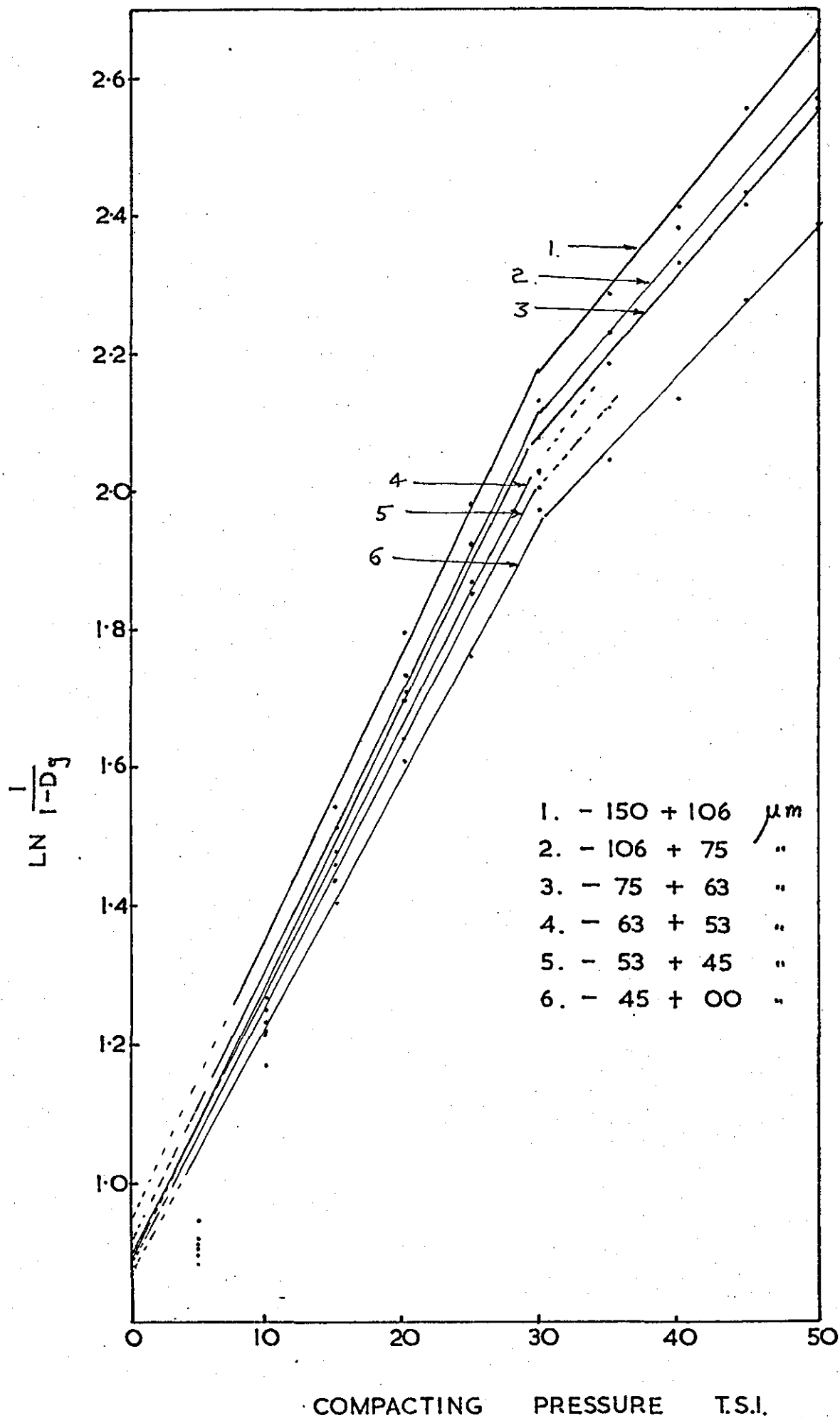


FIGURE 42

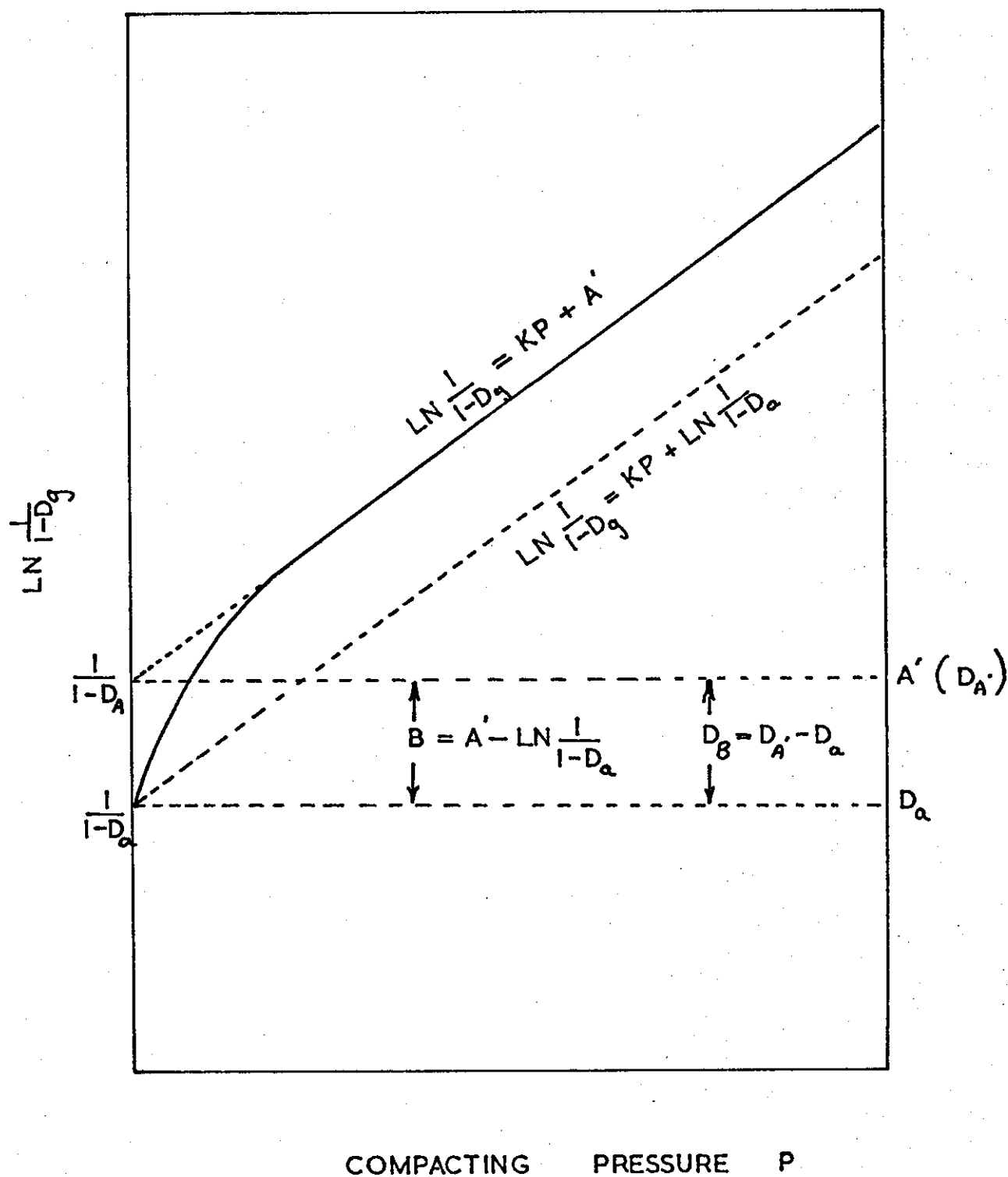


FIGURE 43

Displacement of constant A at zero pressure
in Konopicky - Shapiro relationship

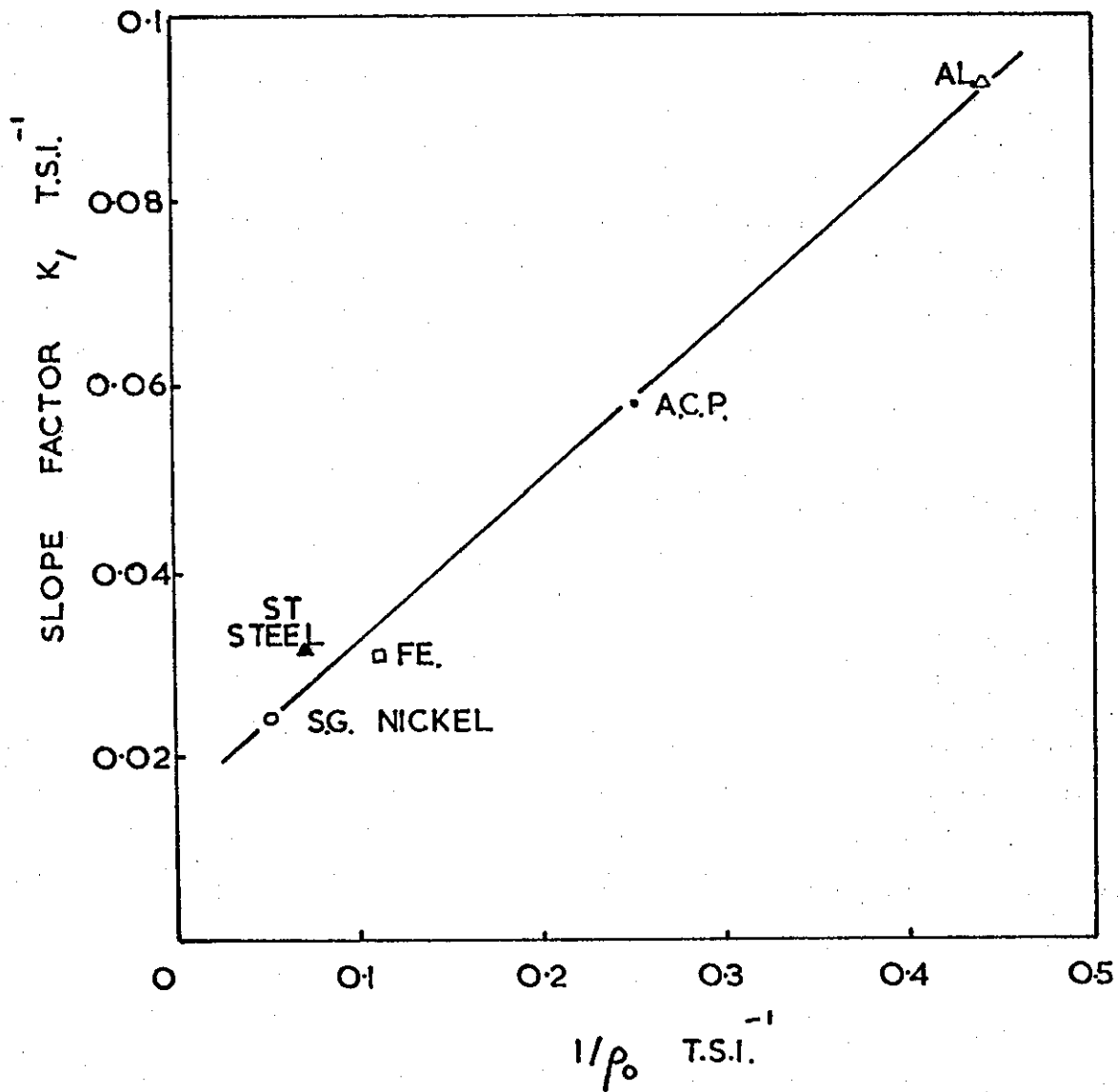


FIGURE 44

Konopicky - Shapiro slope factor K_1 versus
reciprocal of Yield Stress $1/\rho_0$

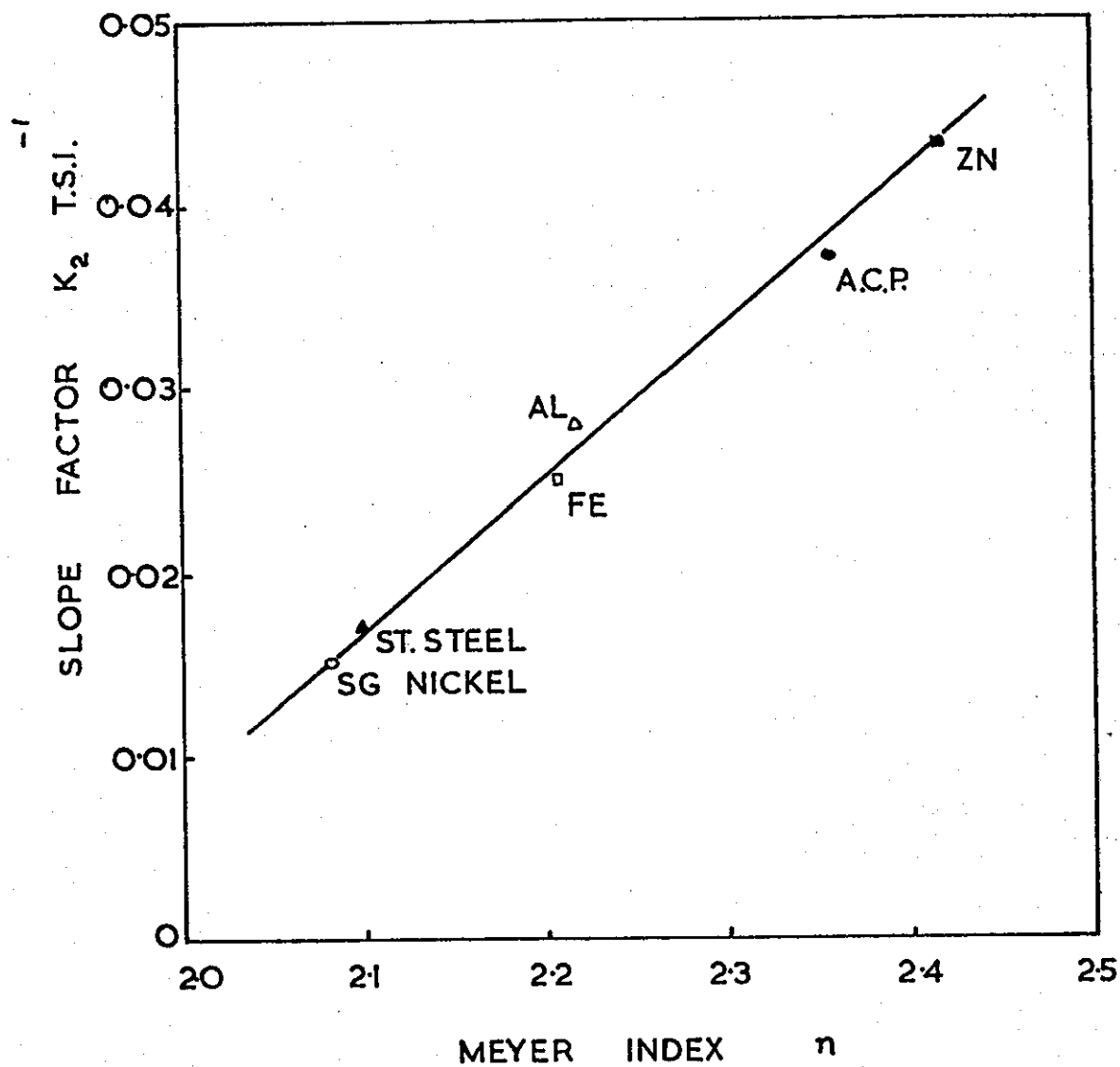


FIGURE 45
Konopicky - Shapiro slope factor K_2 versus
Meyer work-hardening index n .

FIGURE 46

Scanning Electron Micrographs of fracture
surfaces of a monosize fraction ($-125 + 106\mu\text{m}$)
of S.G. Nickel

- (a) compacting pressure 5 t.s.i.
- (b) compacting pressure 10 t.s.i.
- (c) compacting pressure 15 t.s.i.
- (d) compacting pressure 20 t.s.i.
- (e) compacting pressure 25 t.s.i.
- (f) compacting pressure 30 t.s.i.
- (g) compacting pressure 35 t.s.i.
- (h) compacting pressure 40 t.s.i.
- (i) compacting pressure 45 t.s.i.
- (j) compacting pressure 50 t.s.i.

All Magnifications X 600

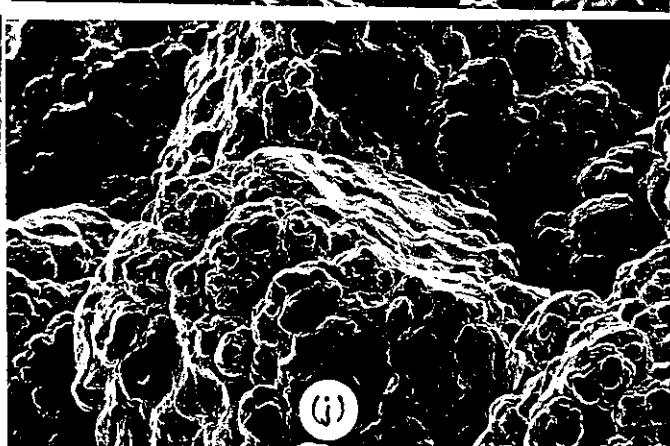
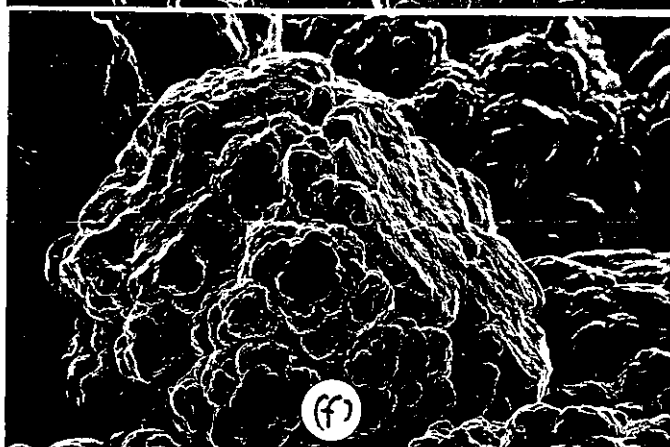
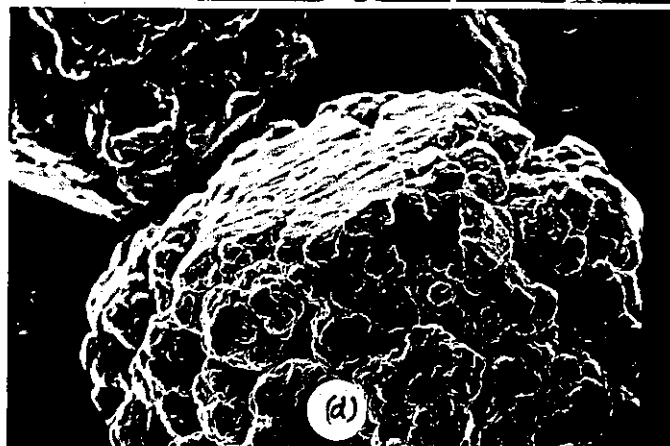
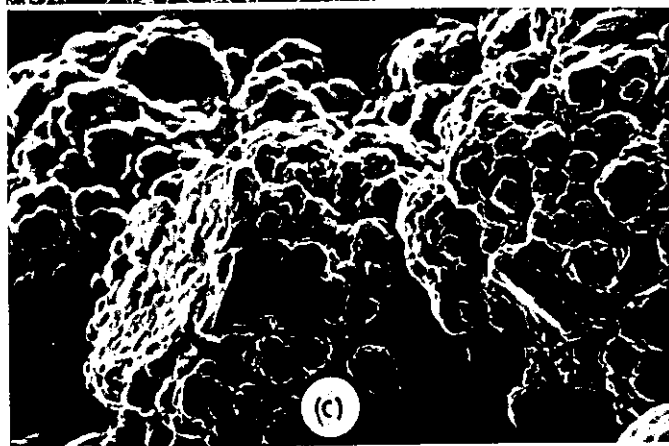
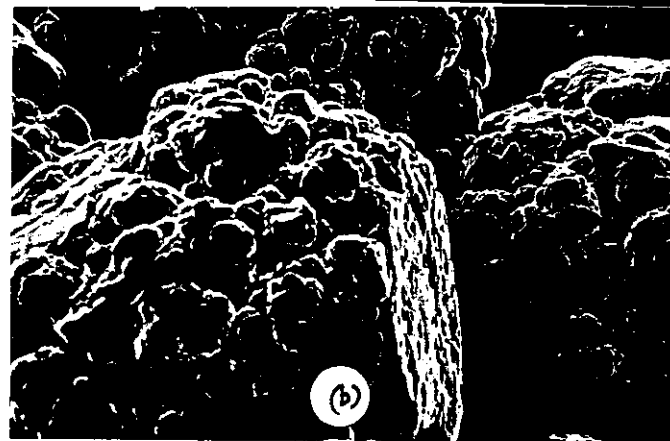
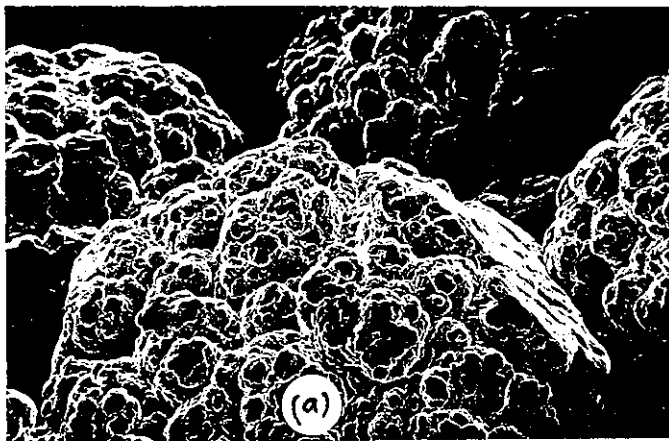


FIGURE 47

Scanning Electron Micrographs of fracture
surfaces of a monosize fraction ($-125 + 106 \mu\text{m}$)
of A.C.P.

- (a) compacting pressure 5 t.s.i.
- (b) compacting pressure 10 t.s.i.
- (c) compacting pressure 15 t.s.i.
- (d) compacting pressure 20 t.s.i.
- (e) compacting pressure 25 t.s.i.
- (f) compacting pressure 30 t.s.i.
- (g) compacting pressure 35 t.s.i.
- (h) compacting pressure 40 t.s.i.
- (i) compacting pressure 45 t.s.i.
- (j) compacting pressure 50 t.s.i.

All Magnifications X 600

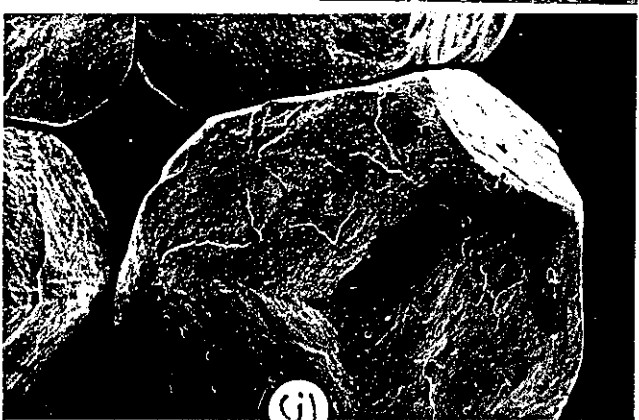
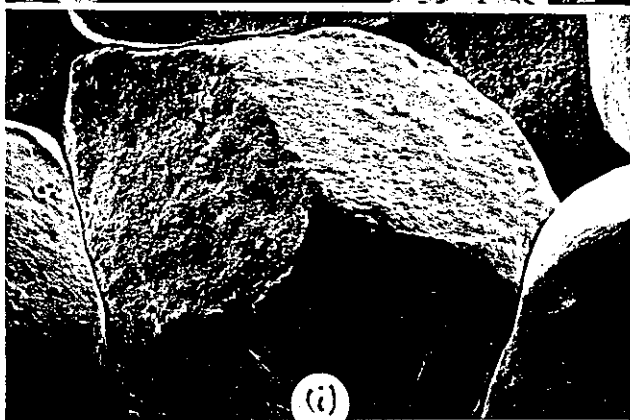
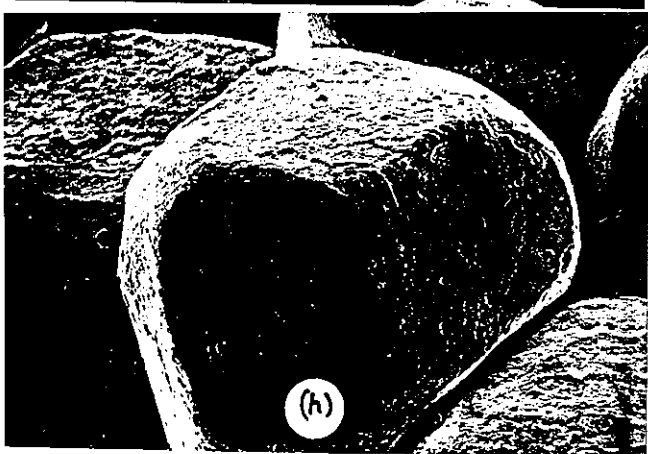
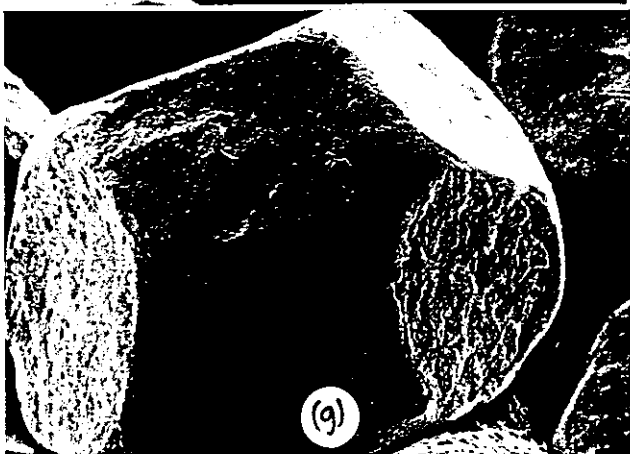
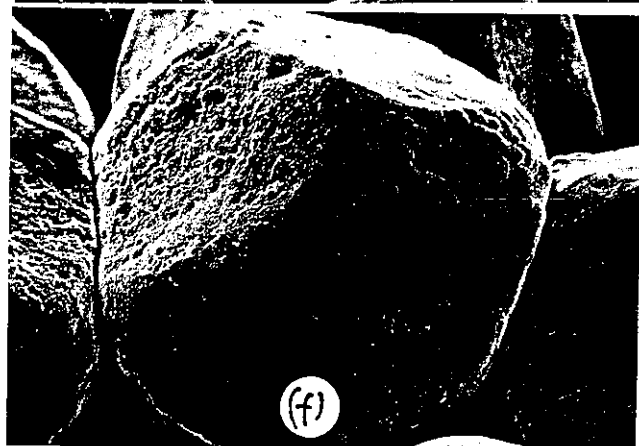
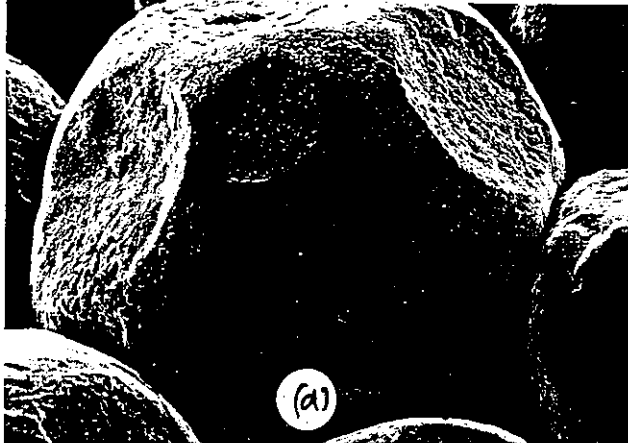
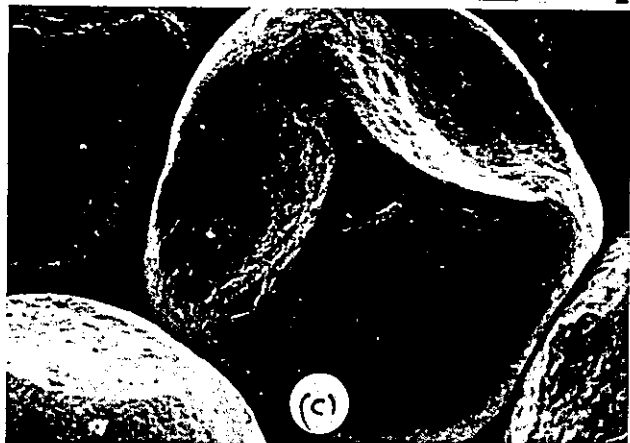
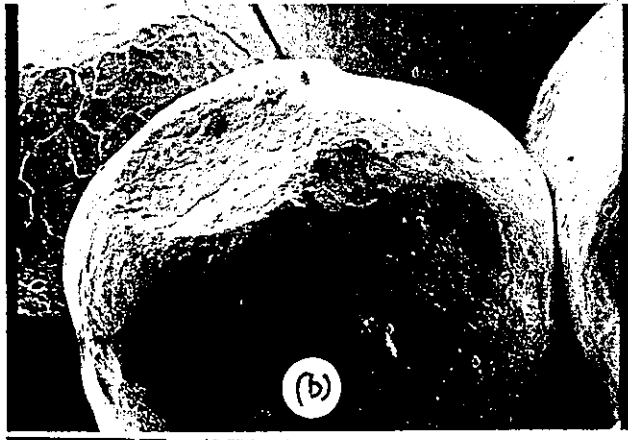
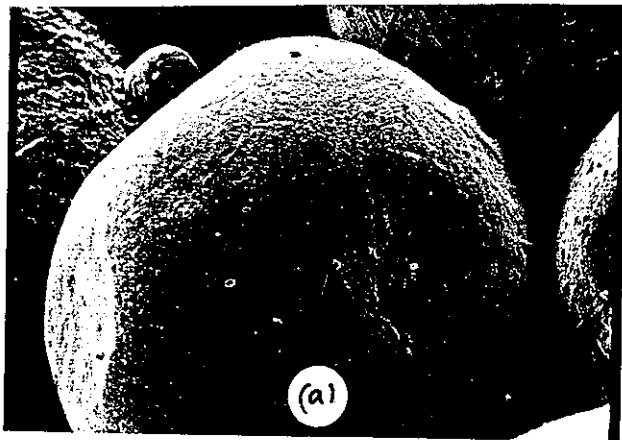


FIGURE 48

Scanning Electron Micrographs of fracture
surfaces of a de-aired monosize fraction
(-125 + 106 μ m) of A.C.P.

- (a) compacting pressure 5 t.s.i.
- (b) compacting pressure 10 t.s.i.
- (c) compacting pressure 20 t.s.i.
- (d) compacting pressure 25 t.s.i.
- (e) compacting pressure 30 t.s.i.
- (f) compacting pressure 35 t.s.i.
- (g) compacting pressure 40 t.s.i.
- (h) compacting pressure 50 t.s.i.

All Magnifications X 600

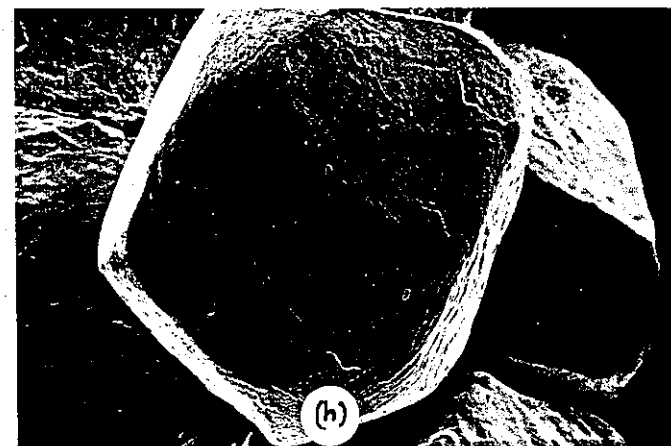
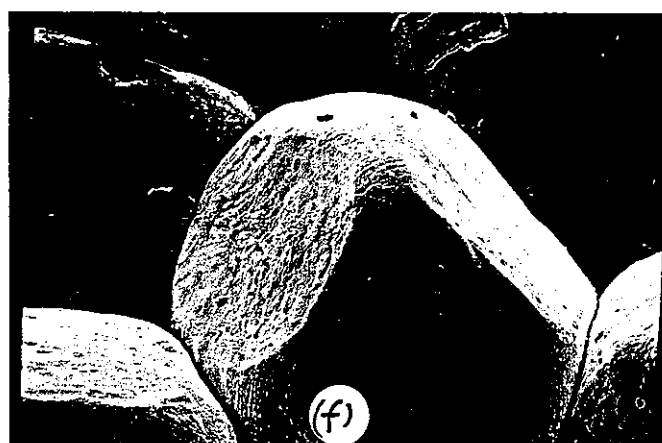
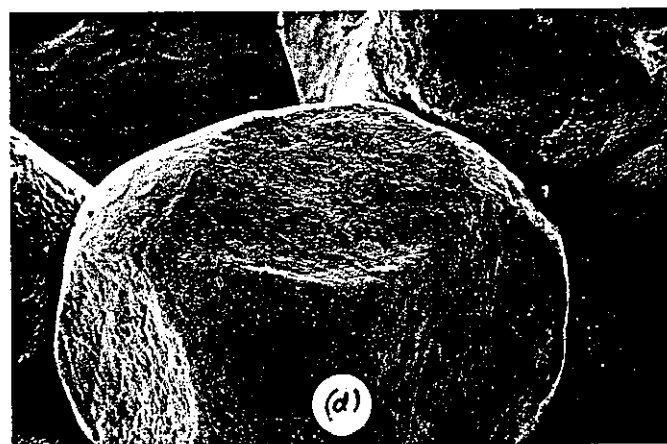
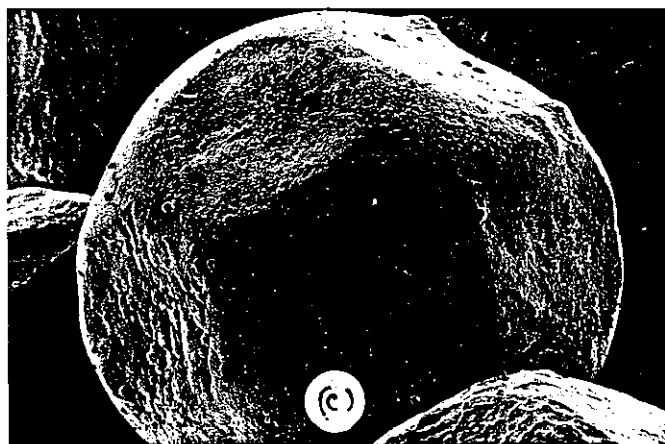
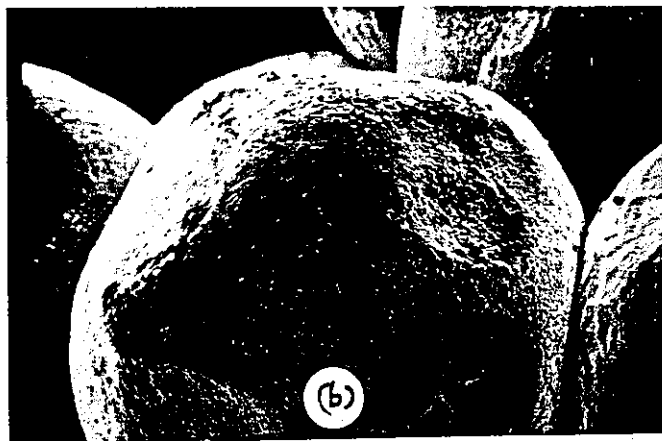
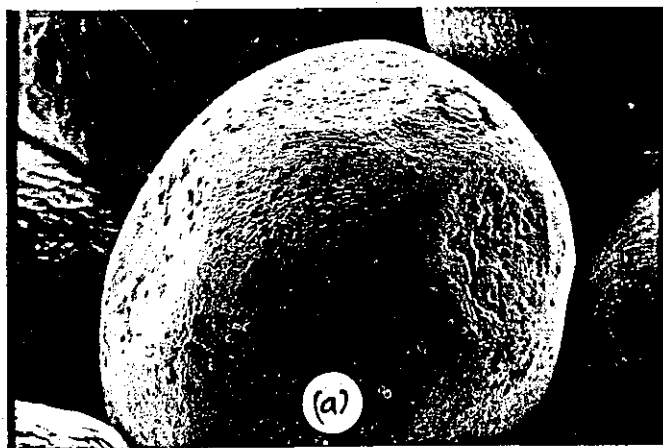


FIGURE 49

Scanning Electron Micrographs of fracture
surfaces of a monosize fraction ($-125 + 106 \mu\text{m}$)
of Aluminium

- (a) compacting pressure 5 t.s.i.
- (b) compacting pressure 10 t.s.i.
- (c) compacting pressure 15 t.s.i.
- (d) compacting pressure 20 t.s.i.
- (e) compacting pressure 25 t.s.i.
- (f) compacting pressure 30 t.s.i.
- (g) compacting pressure 35 t.s.i.
- (h) compacting pressure 40 t.s.i.
- (i) compacting pressure 45 t.s.i.
- (j) compacting pressure 50 t.s.i.

All Magnifications X 600

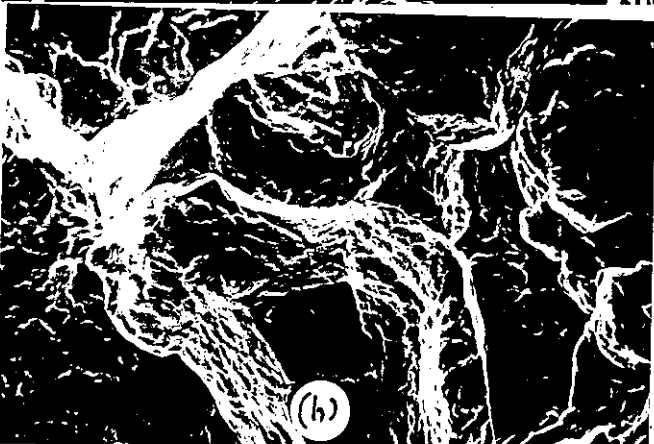
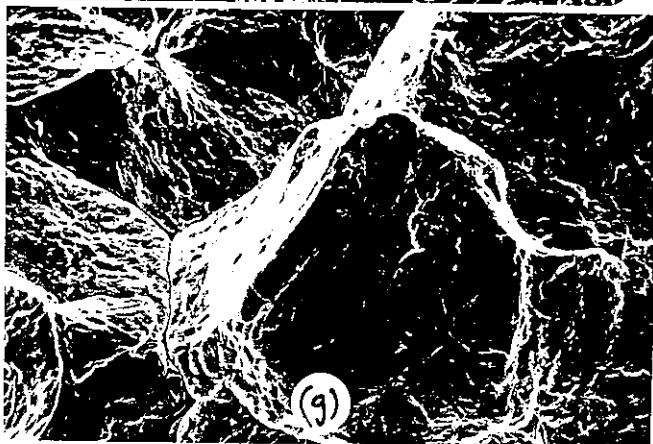
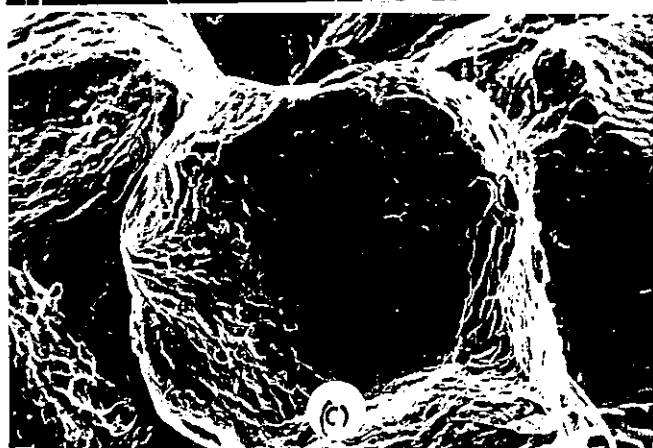
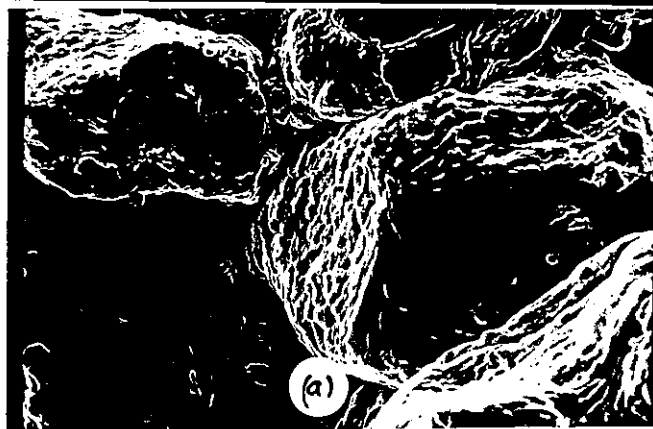


FIGURE 50

Scanning Electron Micrographs of fracture surfaces
of a monosize fraction ($-125 + 106\mu\text{m}$) of Stainless
Steel

- (a) compacting pressure 10 t.s.i.
- (b) compacting pressure 15 t.s.i.
- (c) compacting pressure 20 t.s.i.
- (d) compacting pressure 25 t.s.i.
- (e) compacting pressure 30 t.s.i.
- (f) compacting pressure 35 t.s.i.
- (g) compacting pressure 40 t.s.i.
- (h) compacting pressure 45 t.s.i.
- (i) compacting pressure 50 t.s.i.

All Magnifications X 600

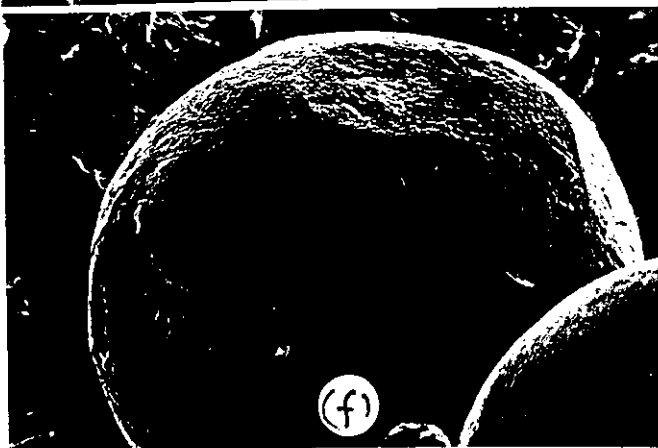
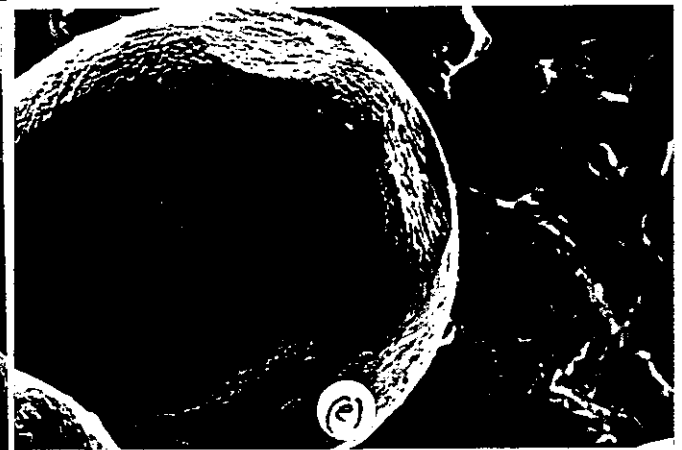
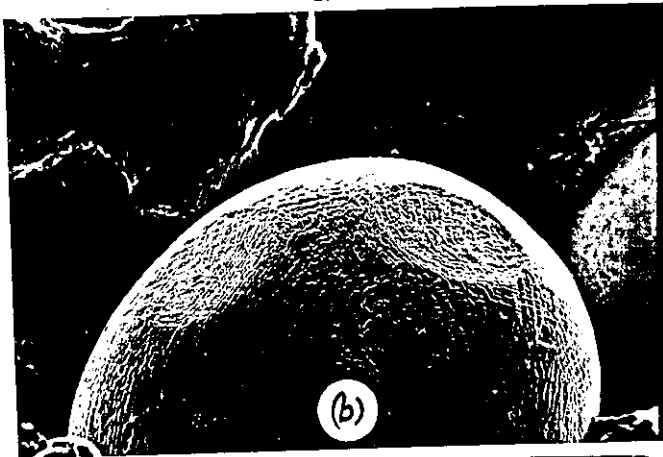
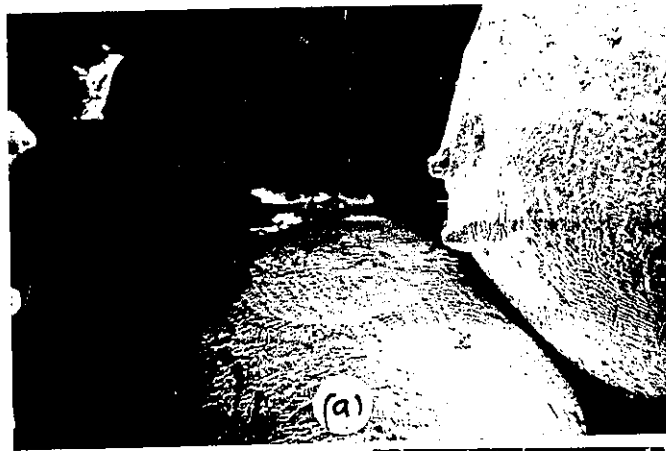


FIGURE 51

Scanning Electron Micrographs of fracture
surfaces of a monosize fraction ($-125 + 106\mu\text{m}$)
of Iron

- (a) compacting pressure 5 t.s.i.
- (b) compacting pressure 10 t.s.i.
- (c) compacting pressure 15 t.s.i.
- (d) compacting pressure 20 t.s.i.
- (e) compacting pressure 25 t.s.i.
- (f) compacting pressure 30 t.s.i.
- (g) compacting pressure 35 t.s.i.
- (h) compacting pressure 40 t.s.i.
- (i) compacting pressure 45 t.s.i.
- (j) compacting pressure 50 t.s.i.

All Magnifications X 600

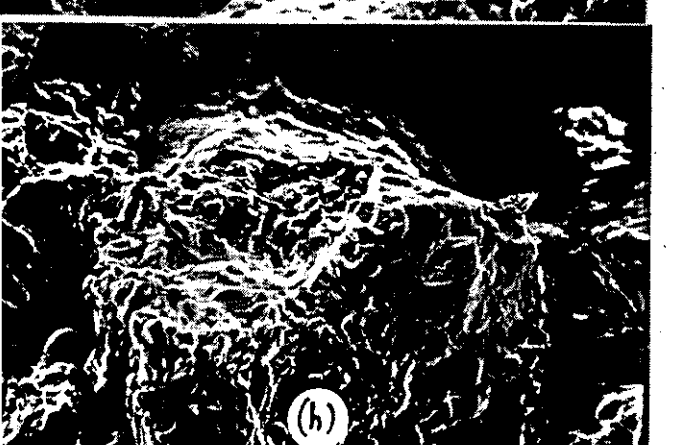
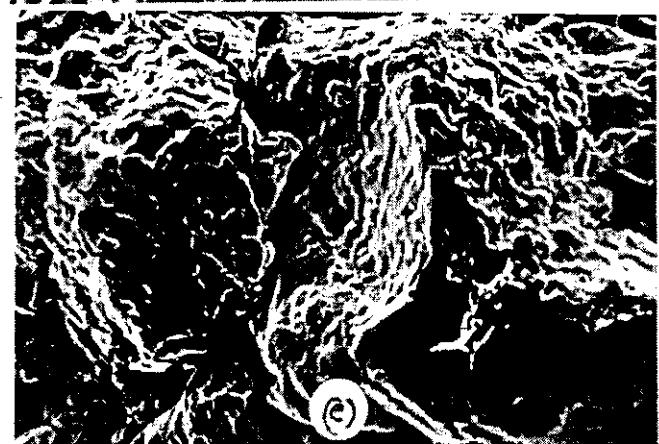
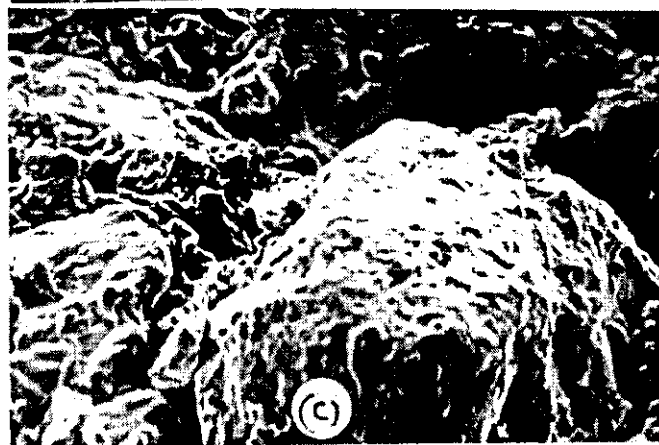
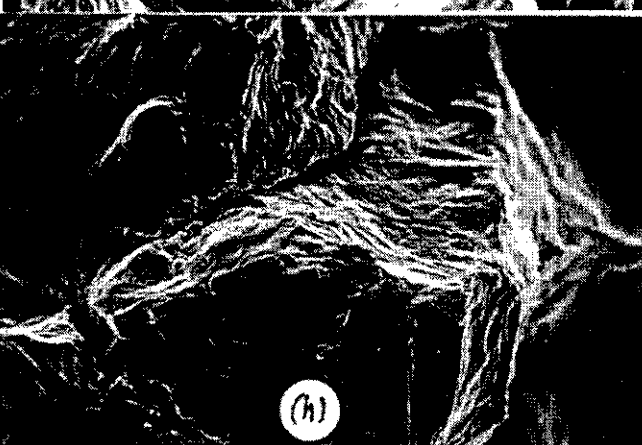
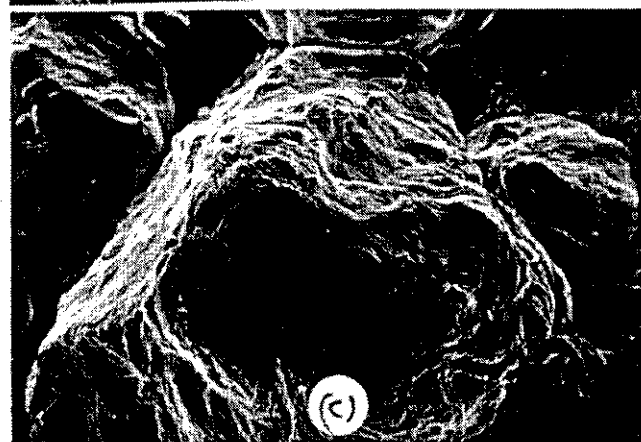


FIGURE 52

Scanning Electron Micrographs of fracture
surfaces of a monosize fraction ($-125 + 106\mu\text{m}$)
of Zinc

- (a) compacting pressure 5 t.s.i.
- (b) compacting pressure 10 t.s.i.
- (c) compacting pressure 15 t.s.i.
- (d) compacting pressure 20 t.s.i.
- (e) compacting pressure 25 t.s.i.
- (f) compacting pressure 30 t.s.i.
- (g) compacting pressure 35 t.s.i.
- (h) compacting pressure 40 t.s.i.
- (i) compacting pressure 45 t.s.i.
- (j) compacting pressure 50 t.s.i.

All Magnifications X 600



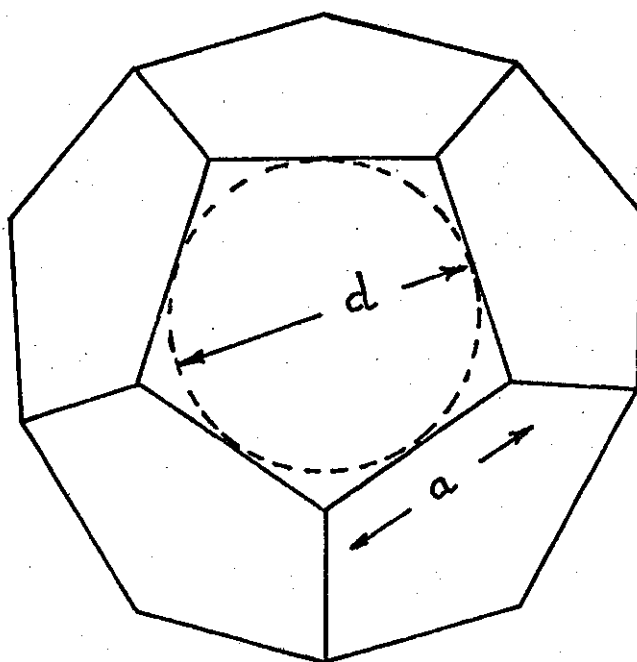
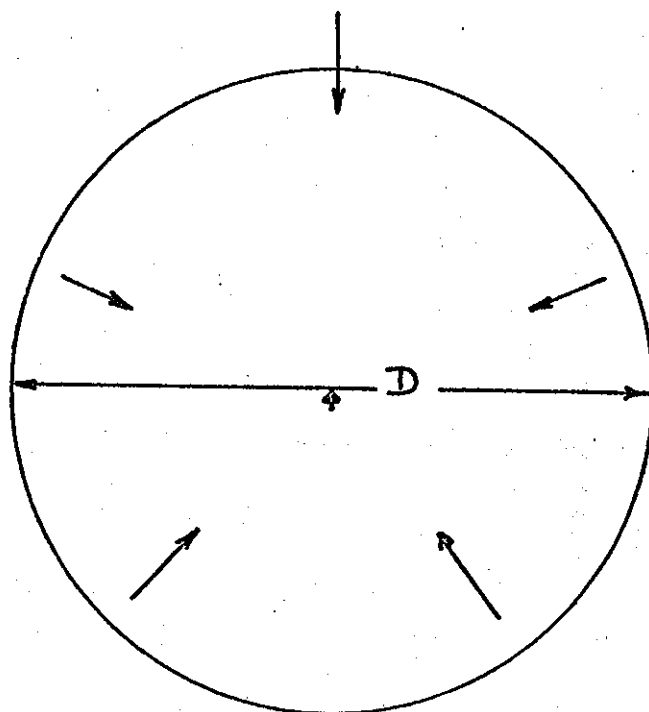


FIGURE 53

Model conversion of a sphere to a dodecahedron

FIGURES 54 - 59

Changes in contact area d/d_m and particle micro-hardness M.H. with compacting pressure for a monosize fraction ($-125 + 106\mu\text{m}$) of powders.

- | | |
|-----------|---|
| FIGURE 54 | S.G. Nickel |
| FIGURE 55 | A.C.P. non de-aired (NDA) & de-aired (DA) |
| FIGURE 56 | Aluminium |
| FIGURE 57 | Stainless Steel |
| FIGURE 58 | Iron |
| FIGURE 59 | Zinc |

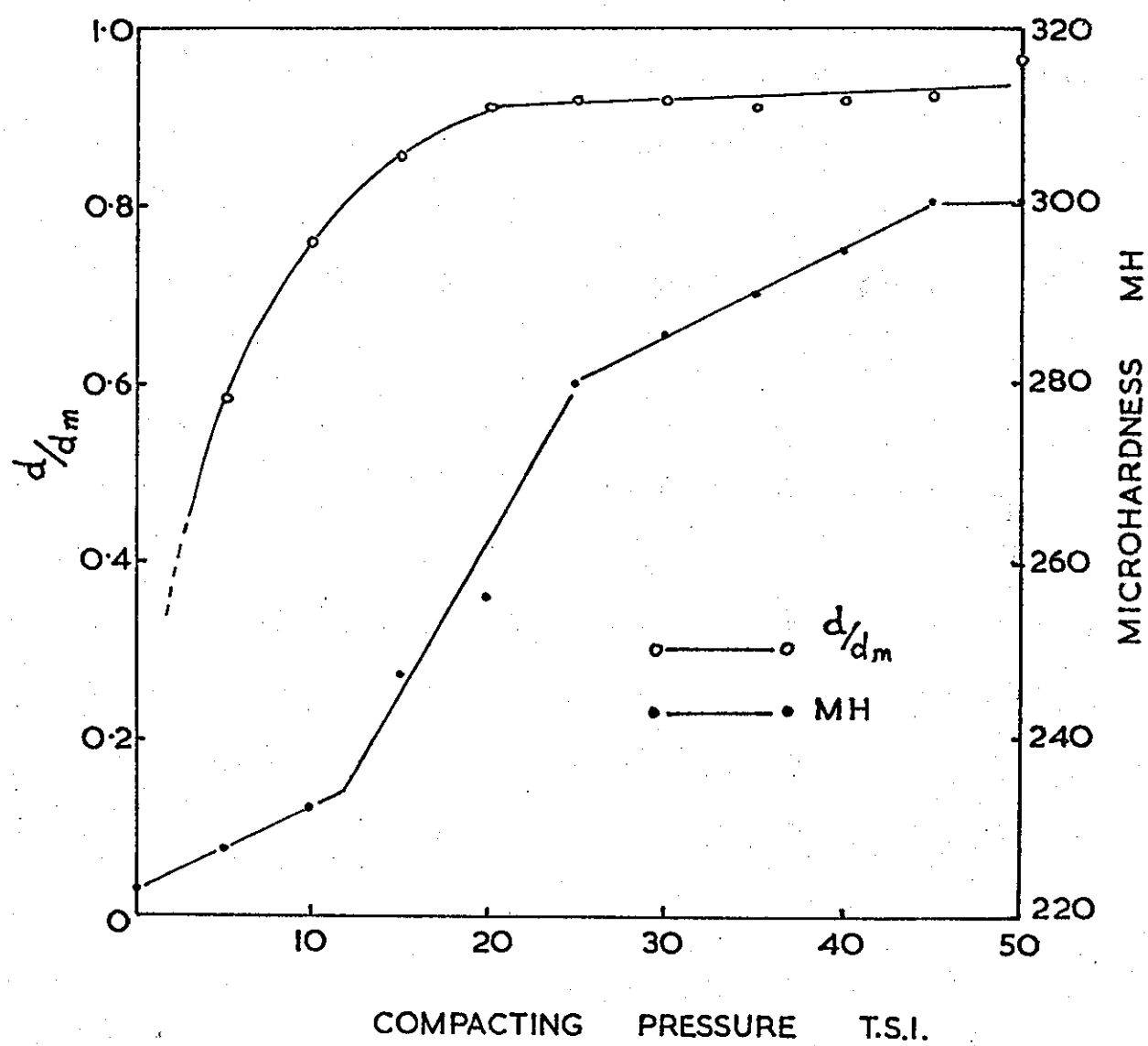


FIGURE 54

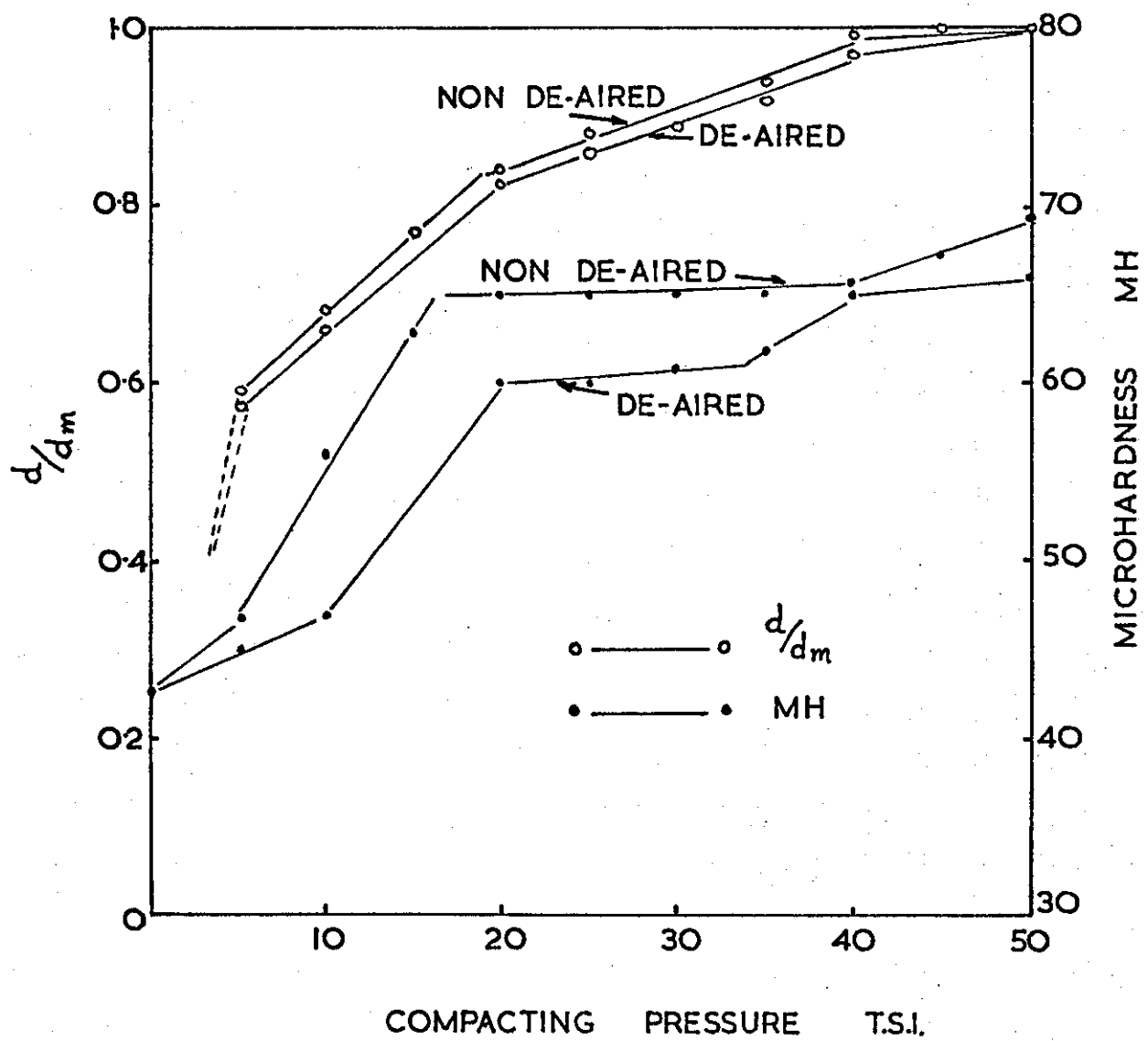


FIGURE 55

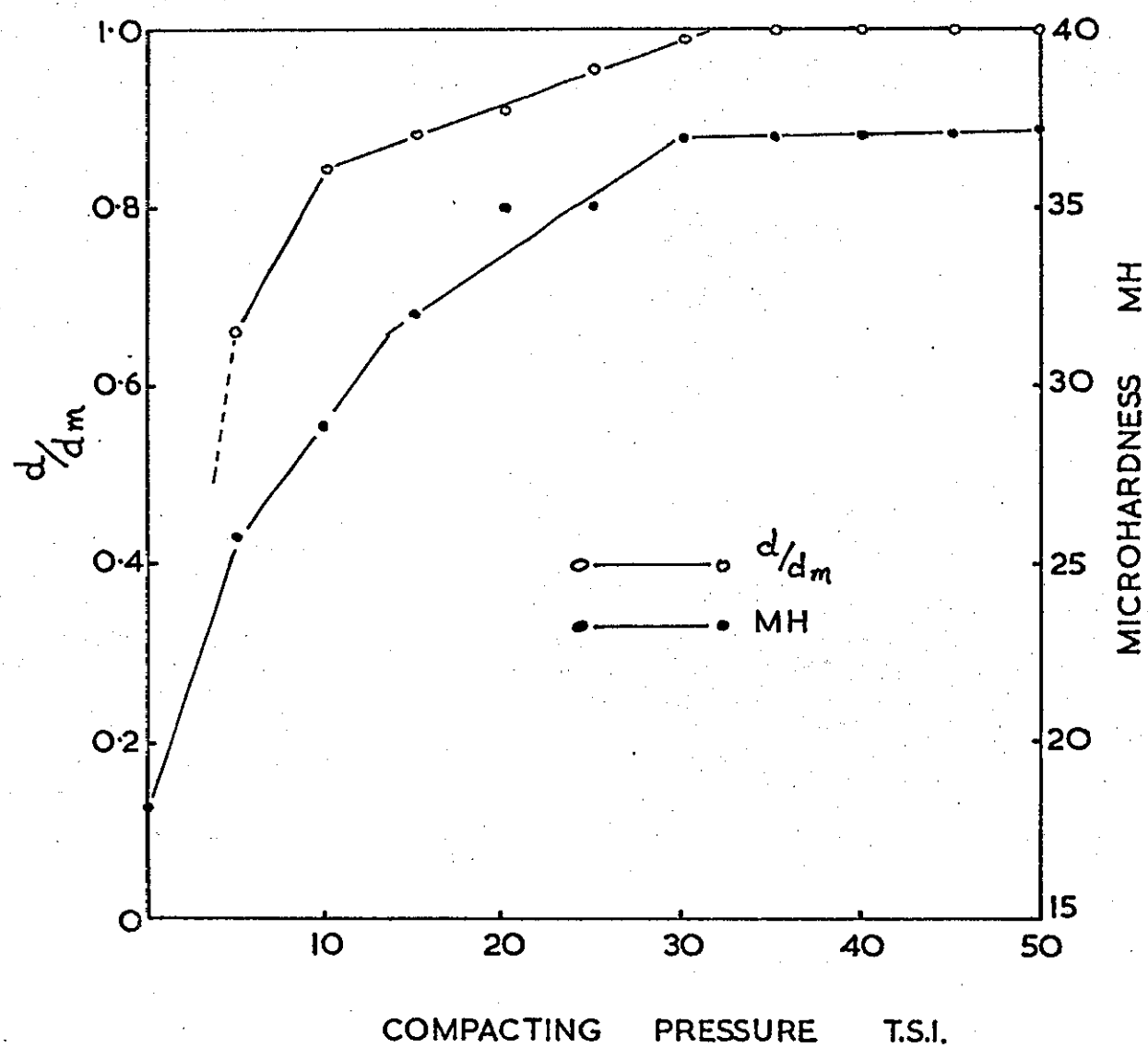


FIGURE 56

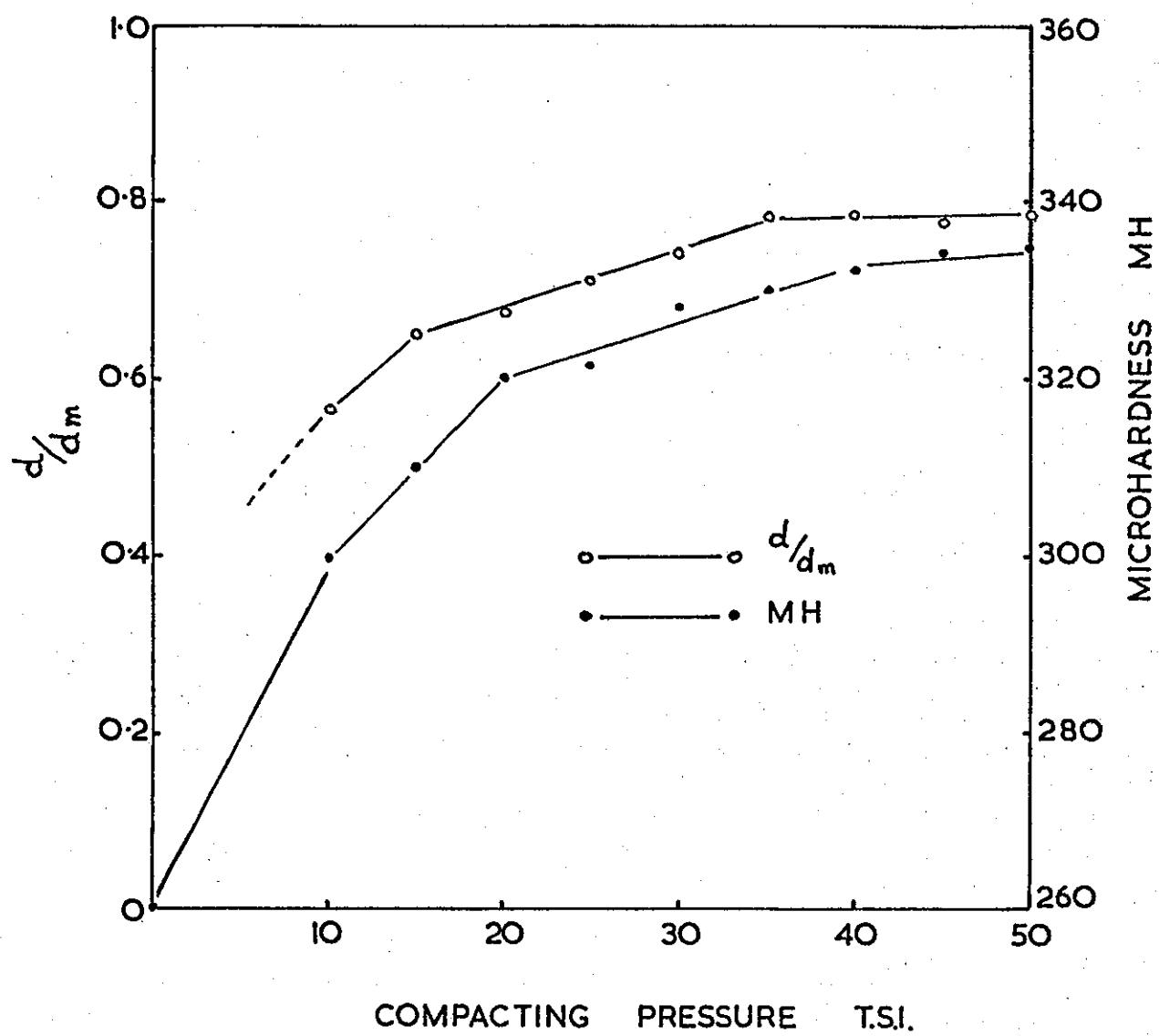


FIGURE 57

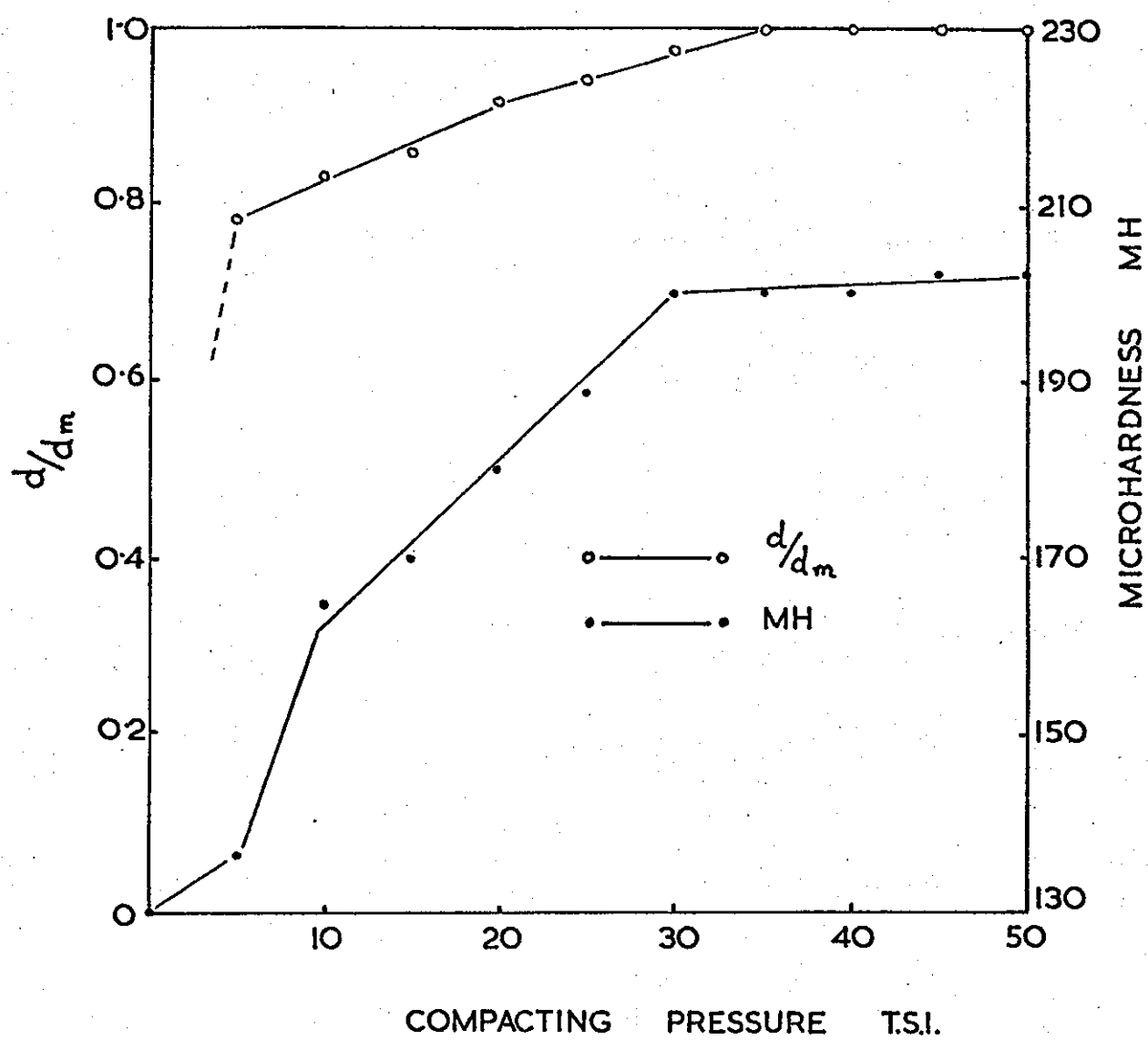


FIGURE 58

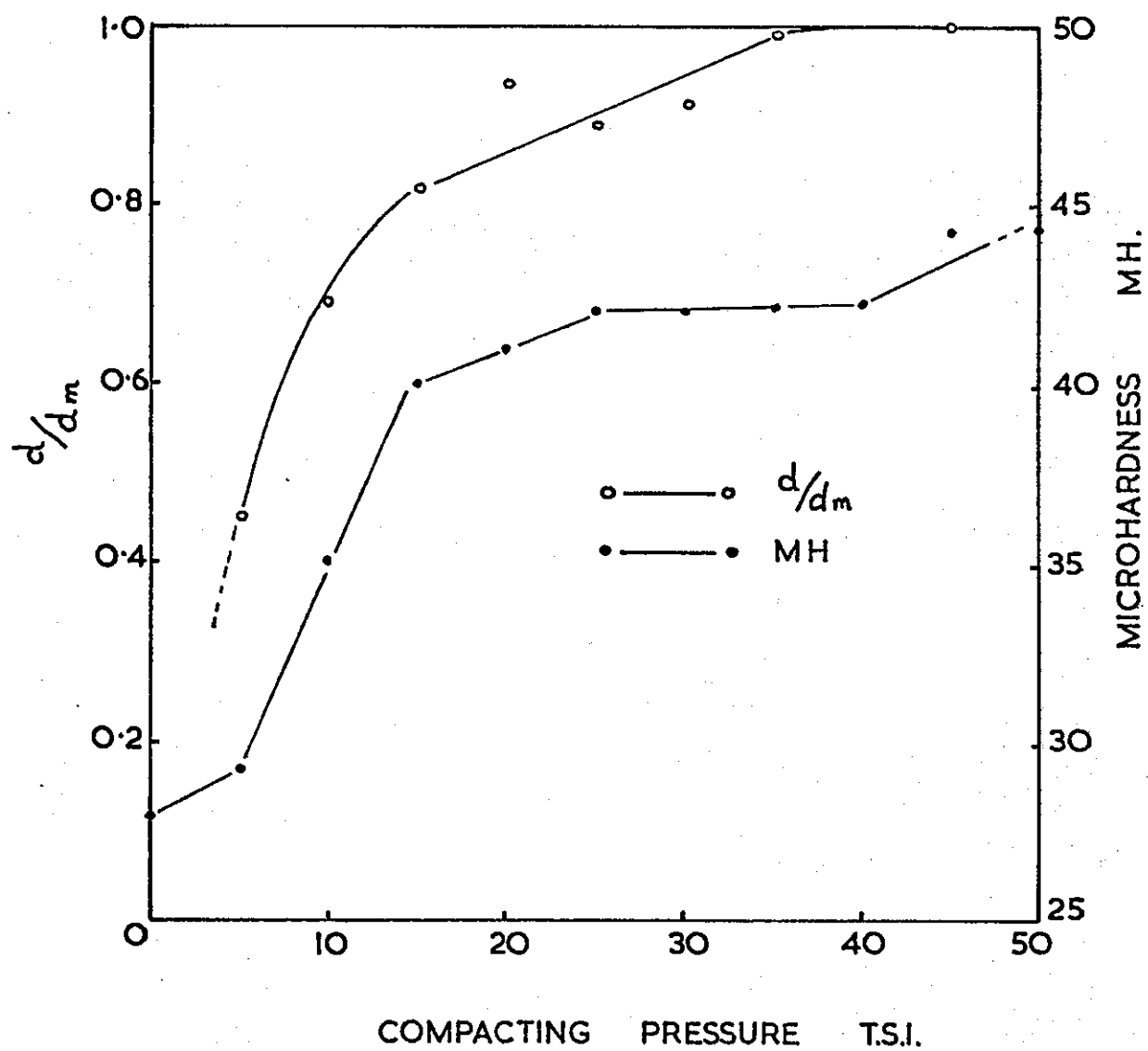


FIGURE 59

FIGURE 60

Photomicrographs of impregnated compacts of a
monosize fraction ($-125 + 106 \mu\text{m}$) of S.G.

Nickel

- (a) compacting pressure 5 t.s.i.
- (b) compacting pressure 10 t.s.i.
- (c) compacting pressure 15 t.s.i.
- (d) compacting pressure 20 t.s.i.
- (e) compacting pressure 25 t.s.i.
- (f) compacting pressure 30 t.s.i.
- (g) compacting pressure 35 t.s.i.
- (h) compacting pressure 40 t.s.i.
- (i) compacting pressure 45 t.s.i.
- (j) compacting pressure 50 t.s.i.

All Magnifications X 200

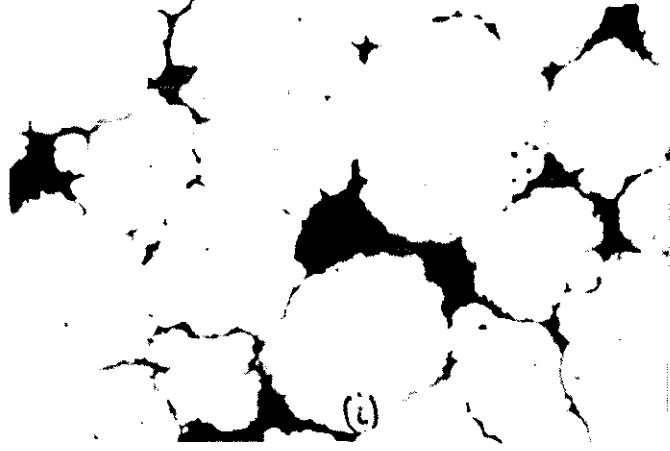
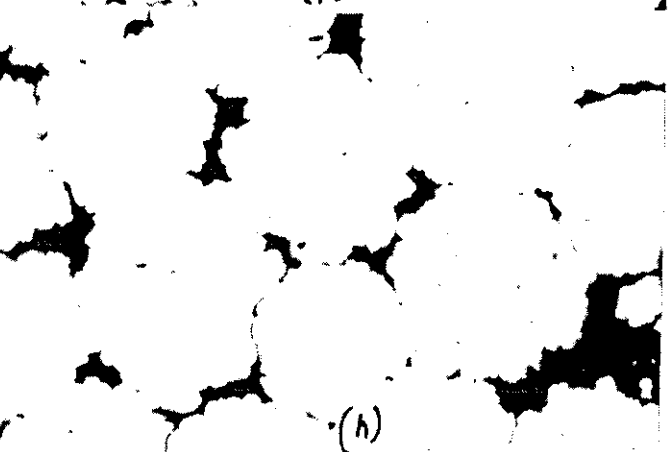
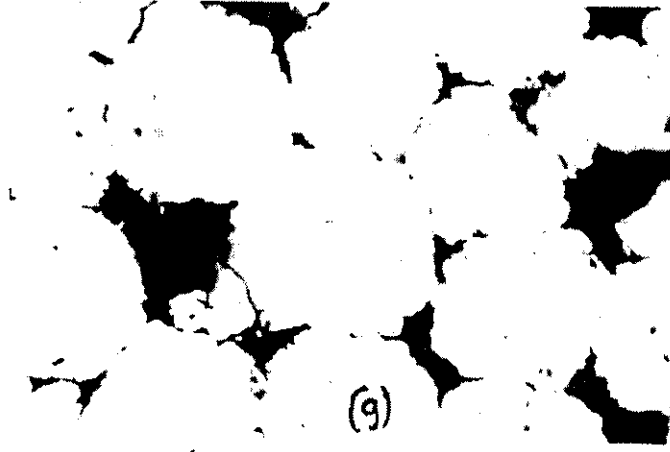
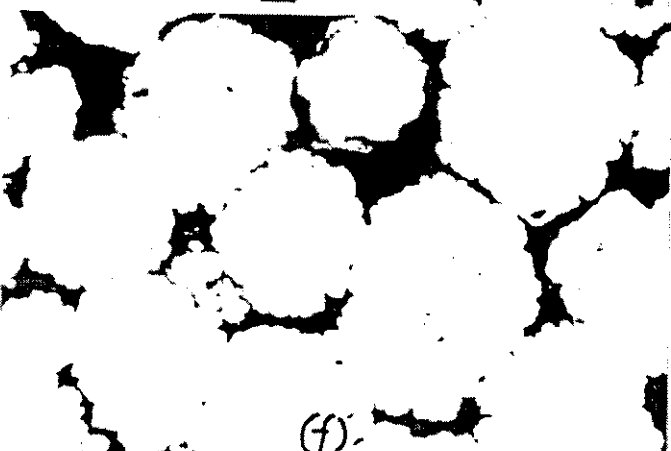
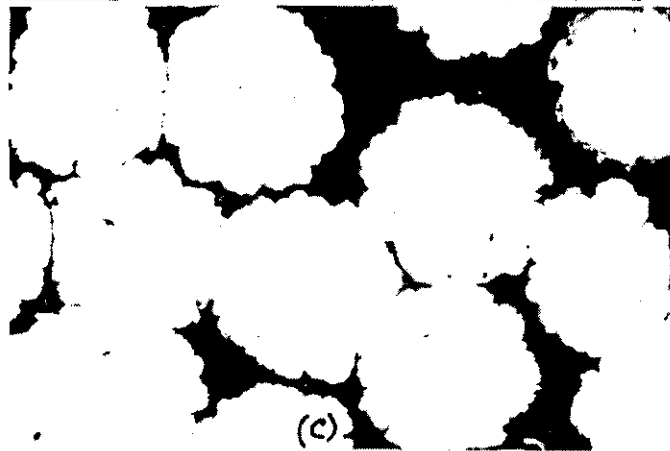
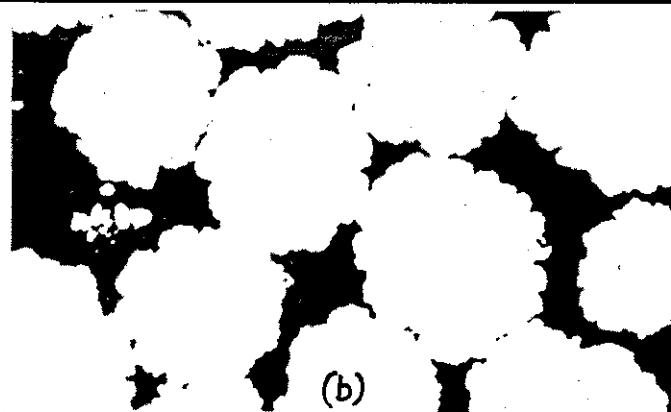
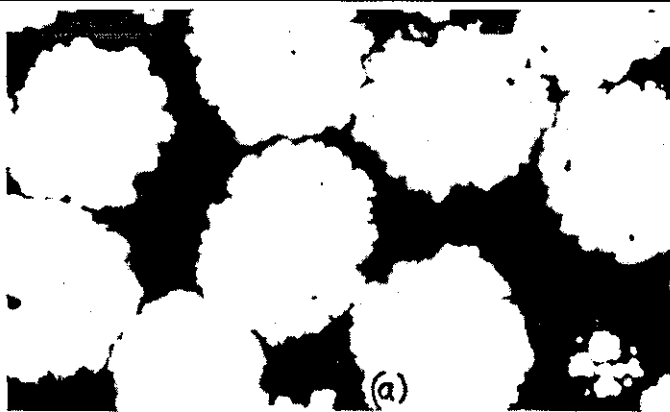
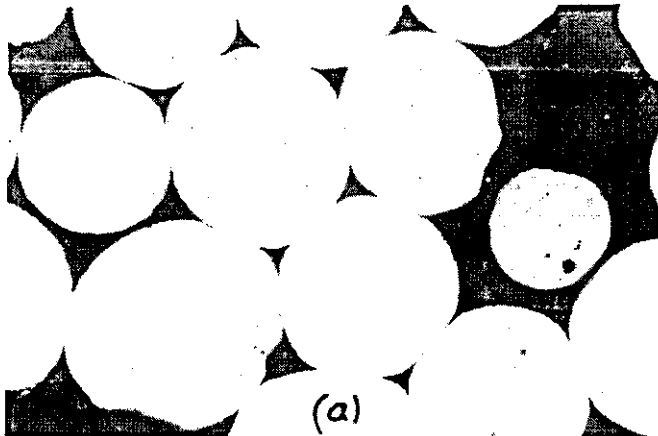


FIGURE 61

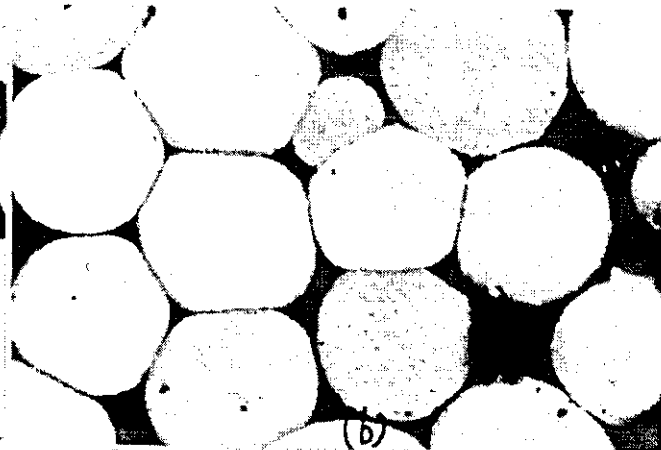
Photomicrographs of impregnated compacts
of a monosize fraction ($-125 + 106 \mu\text{m}$) of
A.C.P.

- (a) compacting pressure 5 t.s.i.
- (b) compacting pressure 10 t.s.i.
- (c) compacting pressure 15 t.s.i.
- (d) compacting pressure 20 t.s.i.
- (e) compacting pressure 25 t.s.i.
- (f) compacting pressure 30 t.s.i.
- (g) compacting pressure 35 t.s.i.
- (h) compacting pressure 40 t.s.i.
- (i) compacting pressure 45 t.s.i.
- (j) compacting pressure 50 t.s.i.

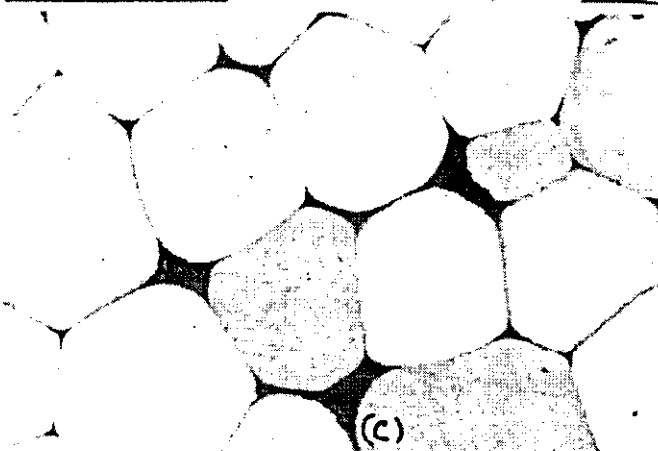
All Magnifications X 200



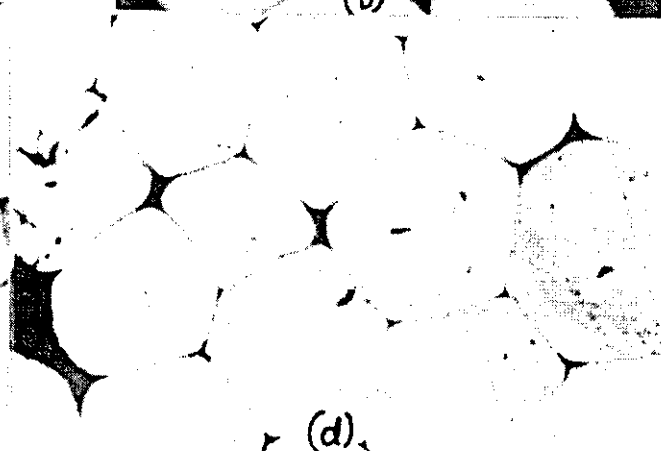
(a)



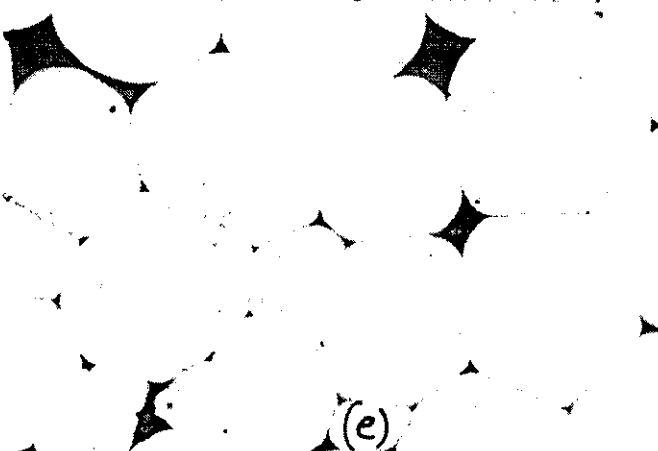
(b)



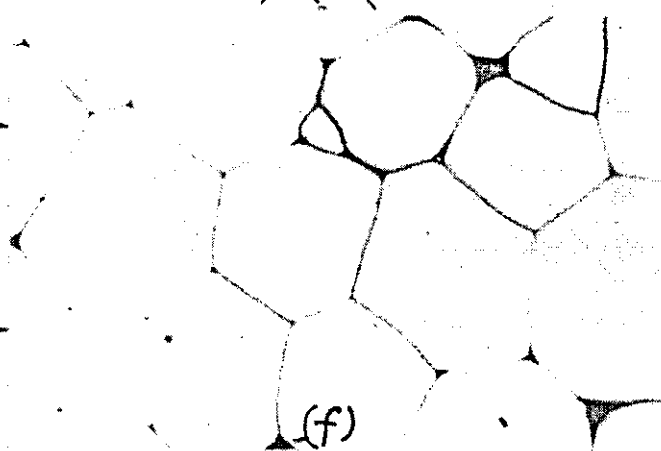
(c)



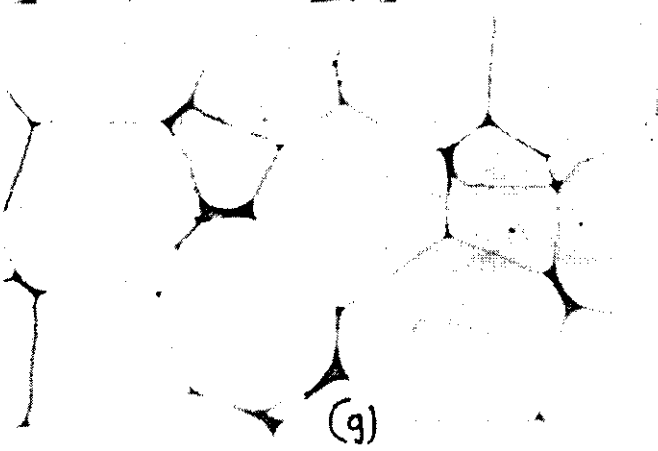
(d)



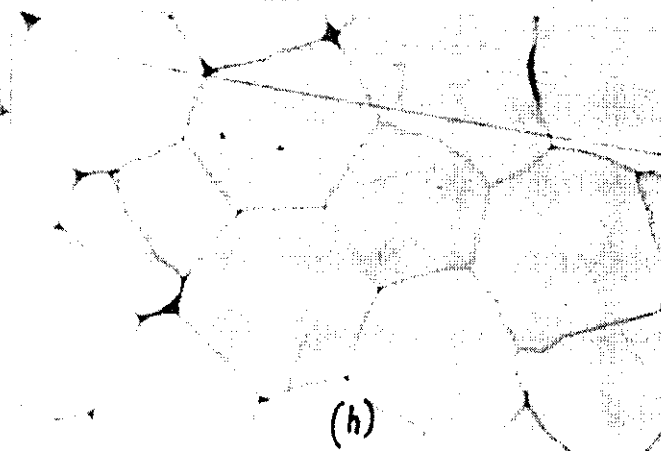
(e)



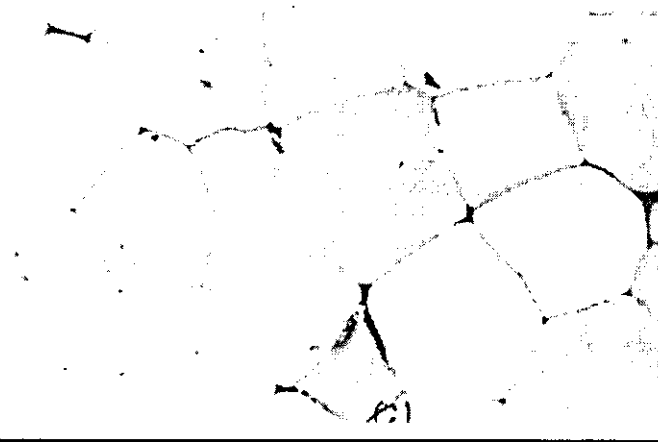
(f)



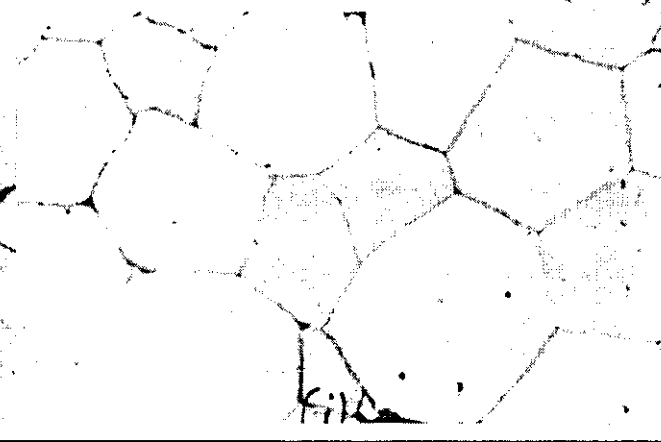
(g)



(h)



(i)



(j)

FIGURE 62

Photomicrographs of impregnated compacts of
a de-aired monosize fraction ($-125 + 106 \mu\text{m}$)
of A.C.P.

- (a) compacting pressure 5 t.s.i.
- (b) compacting pressure 10 t.s.i.
- (c) compacting pressure 20 t.s.i.
- (d) compacting pressure 25 t.s.i.
- (e) compacting pressure 30 t.s.i.
- (f) compacting pressure 35 t.s.i.
- (g) compacting pressure 40 t.s.i.
- (h) compacting pressure 50 t.s.i.

All Magnifications X 200

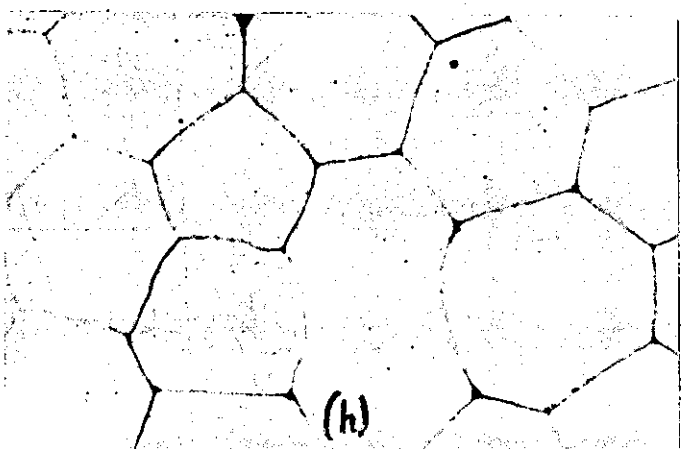
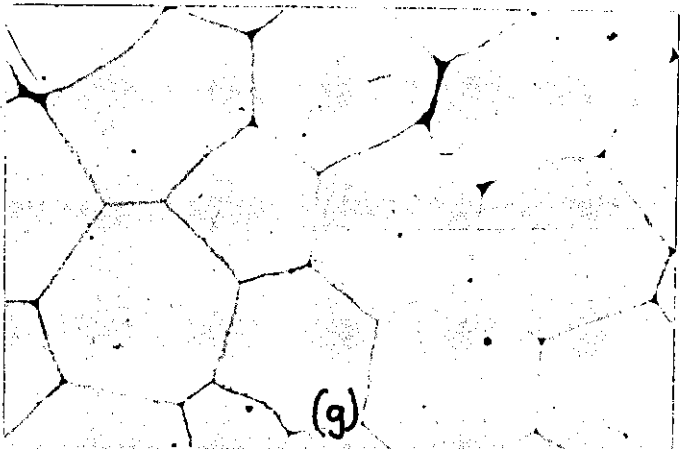
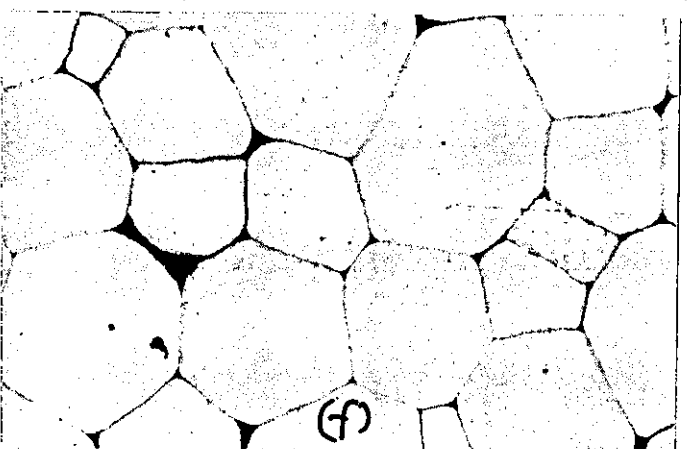
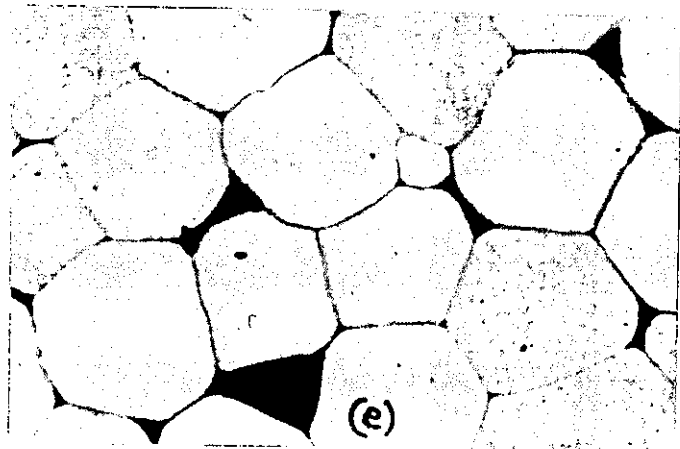
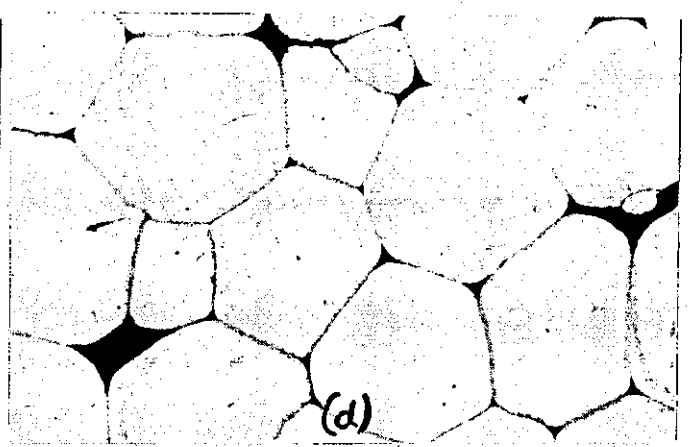
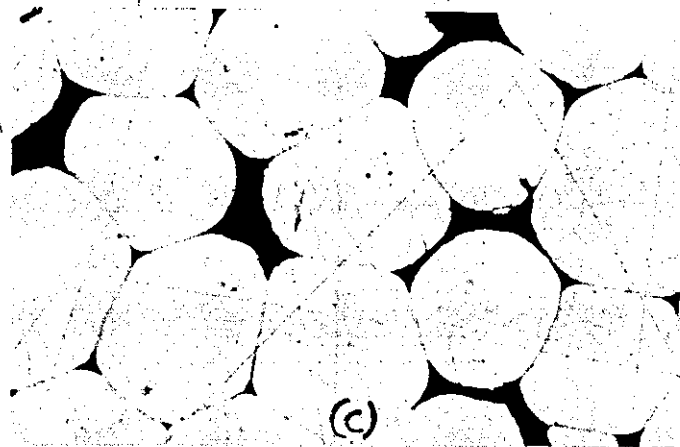
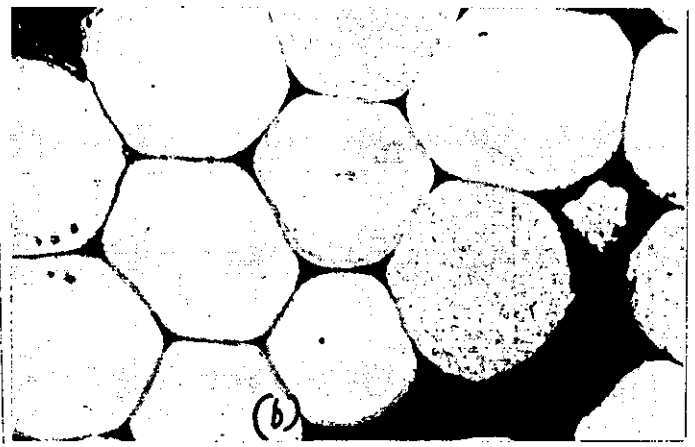
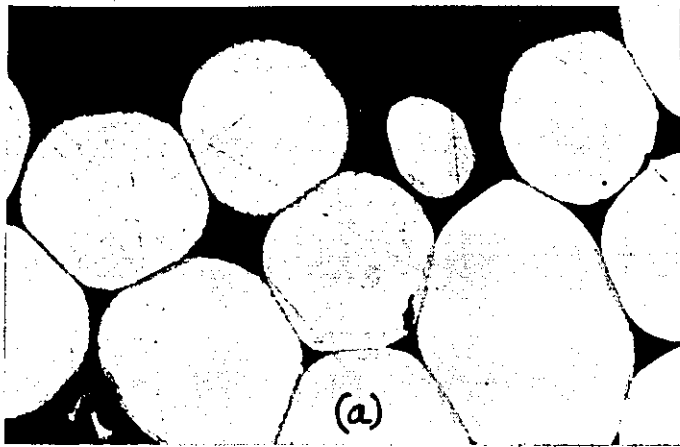


FIGURE 63

Photomicrographs of impregnated compacts of
a monosize fraction ($-125 + 106 \mu\text{m}$) of
Aluminium

- (a) compacting pressure 5 t.s.i.
- (b) compacting pressure 10 t.s.i.
- (c) compacting pressure 15 t.s.i.
- (d) compacting pressure 20 t.s.i.
- (e) compacting pressure 25 t.s.i.
- (f) compacting pressure 30 t.s.i.
- (g) compacting pressure 35 t.s.i.
- (h) compacting pressure 40 t.s.i.
- (i) compacting pressure 45 t.s.i.
- (j) compacting pressure 50 t.s.i.

All Magnifications X 200

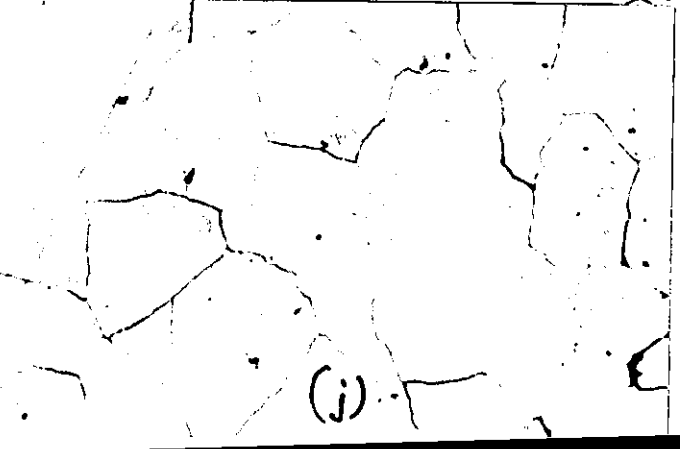
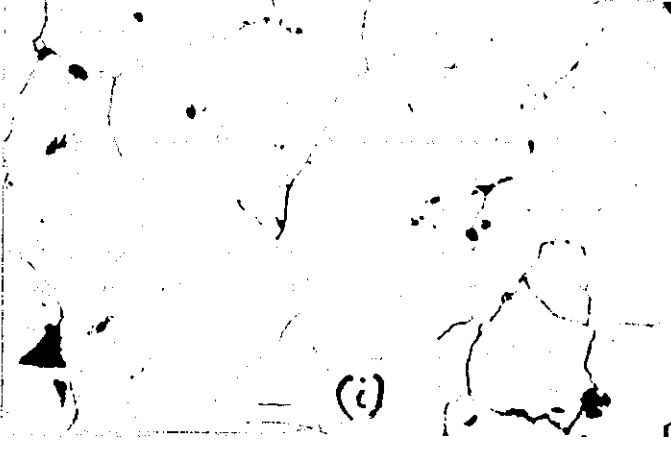
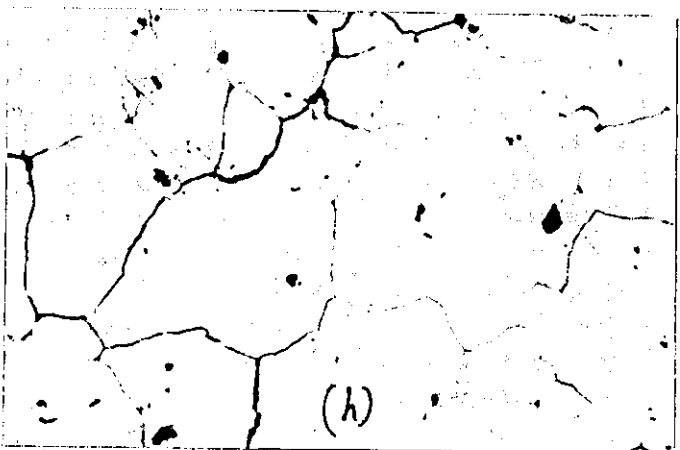
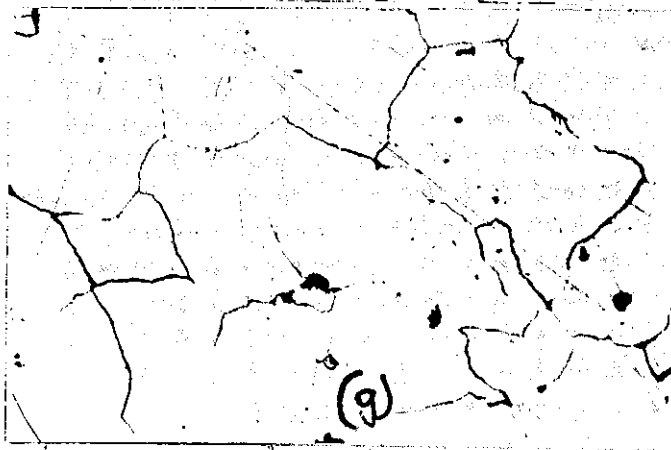
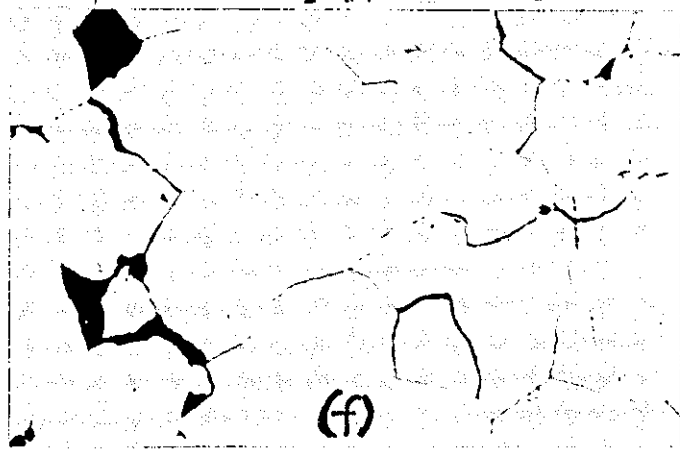
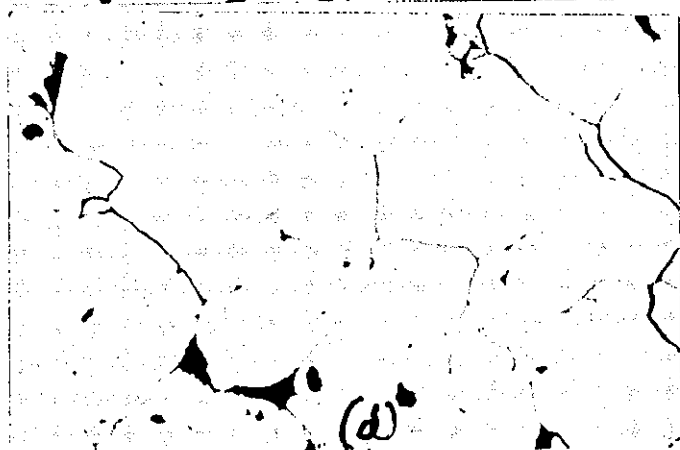
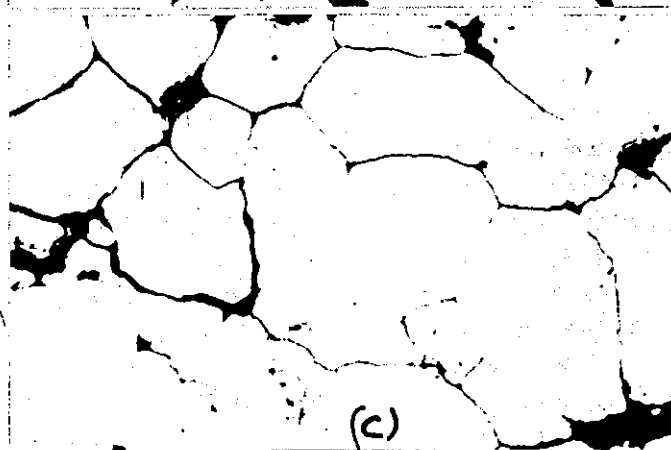
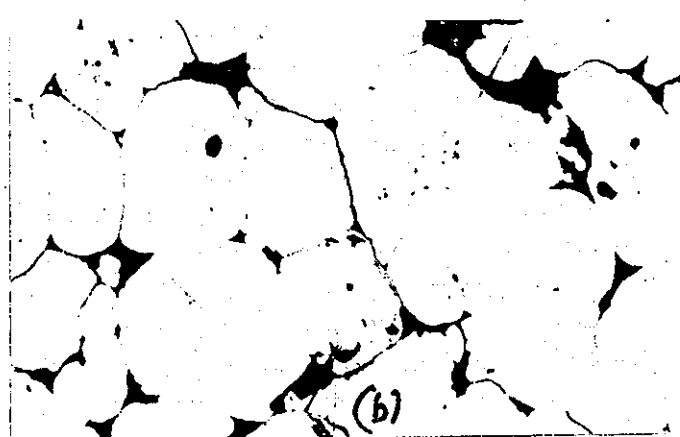
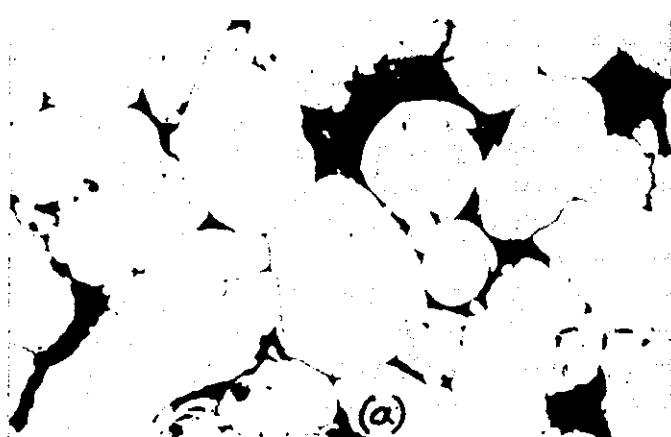


FIGURE 64

Photomicrographs of impregnated compacts
of a monosize fraction ($-125 + 106\mu\text{m}$) of
Stainless Steel

- (a) compacting pressure 10 t.s.i.
- (b) compacting pressure 15 t.s.i.
- (c) compacting pressure 20 t.s.i.
- (d) compacting pressure 25 t.s.i.
- (e) compacting pressure 30 t.s.i.
- (f) compacting pressure 35 t.s.i.
- (g) compacting pressure 40 t.s.i.
- (h) compacting pressure 45 t.s.i.
- (i) compacting pressure 50 t.s.i.

All Magnifications X 200

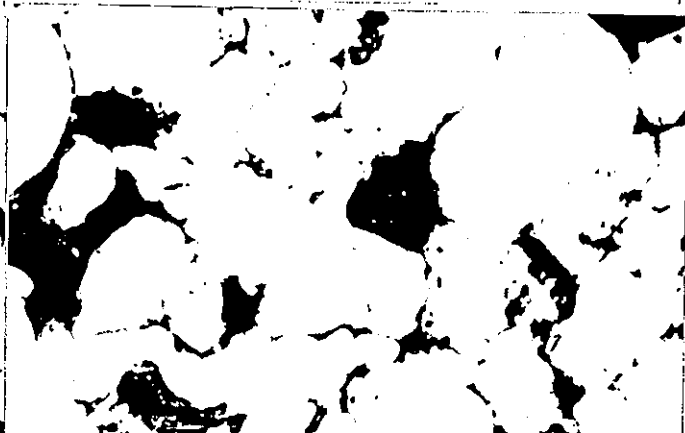
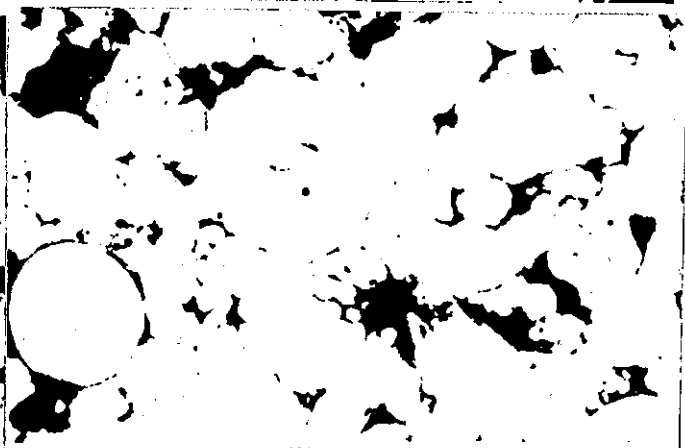
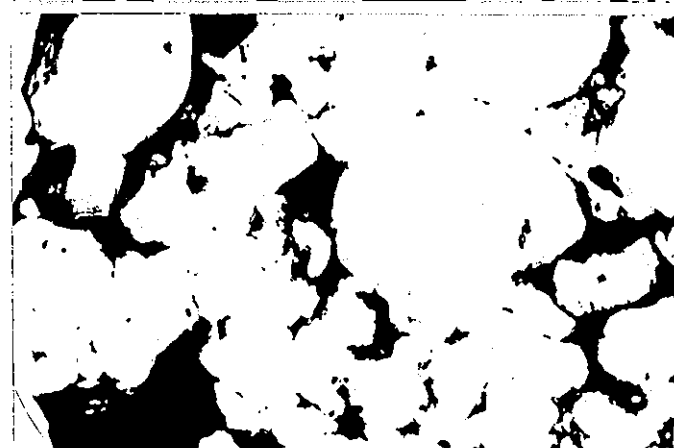
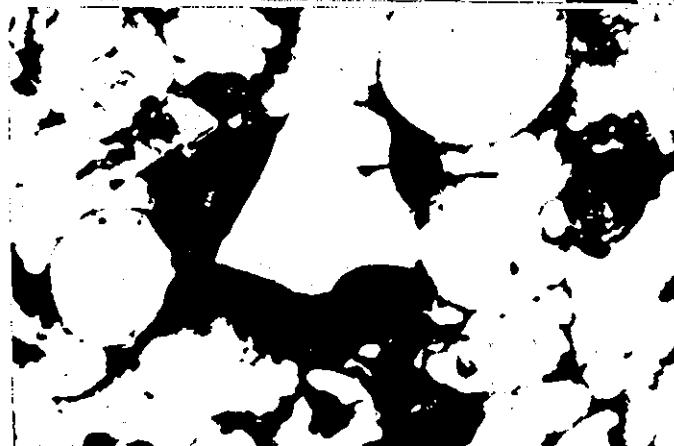
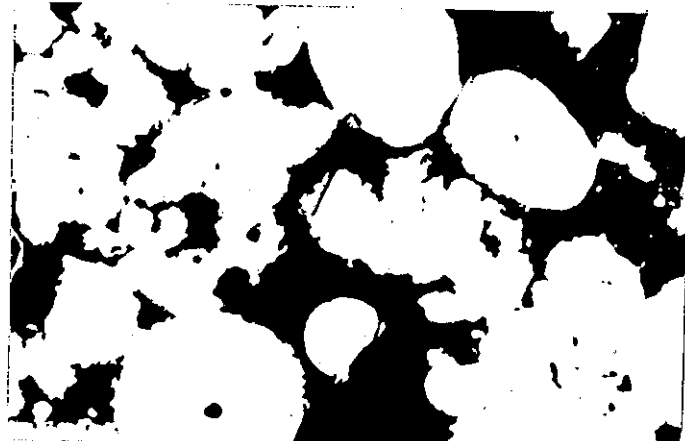
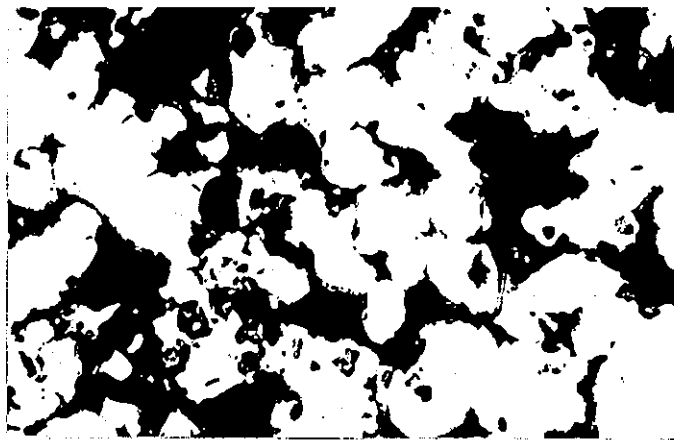


FIGURE 65

Photomicrographs of impregnated compacts of
a monosize fraction ($-125 + 106\mu\text{m}$) of Iron

- (a) compacting pressure 5 t.s.i.
- (b) compacting pressure 10 t.s.i.
- (c) compacting pressure 15 t.s.i.
- (d) compacting pressure 20 t.s.i.
- (e) compacting pressure 25 t.s.i.
- (f) compacting pressure 30 t.s.i.
- (g) compacting pressure 35 t.s.i.
- (h) compacting pressure 40 t.s.i.
- (i) compacting pressure 45 t.s.i.
- (j) compacting pressure 50 t.s.i.

All Magnifications X 200

FIGURE 65

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- (a) compacting pressure 5 t.s.i.
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- (g) compacting pressure 35 t.s.i.
- (h) compacting pressure 40 t.s.i.
- (i) compacting pressure 45 t.s.i.
- (j) compacting pressure 50 t.s.i.

All Magnifications X 200

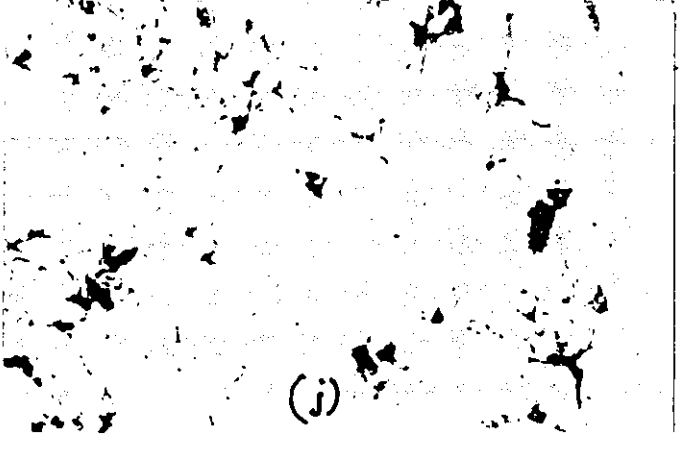
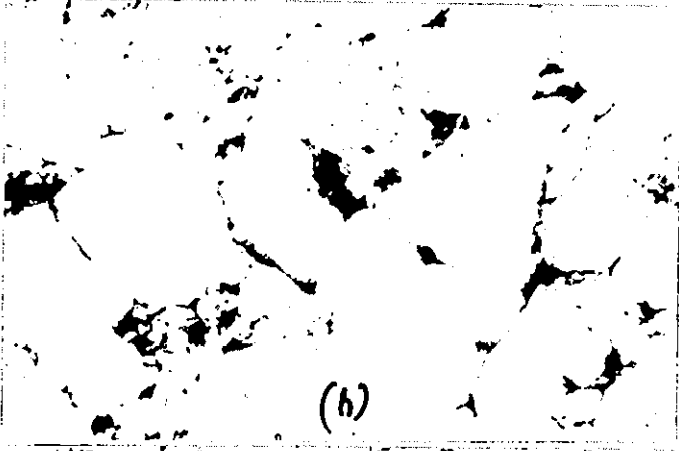
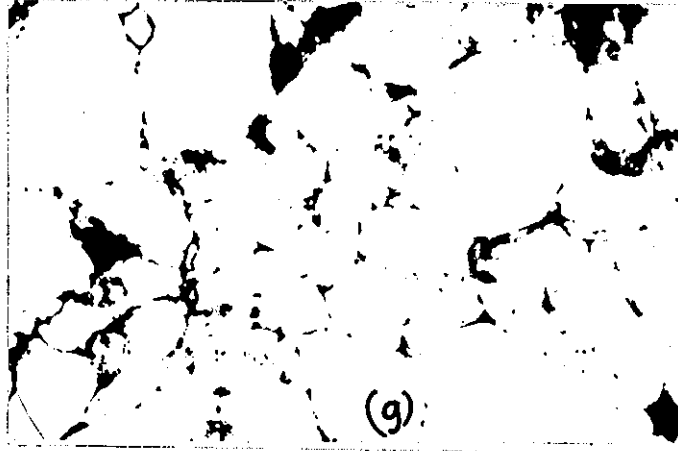
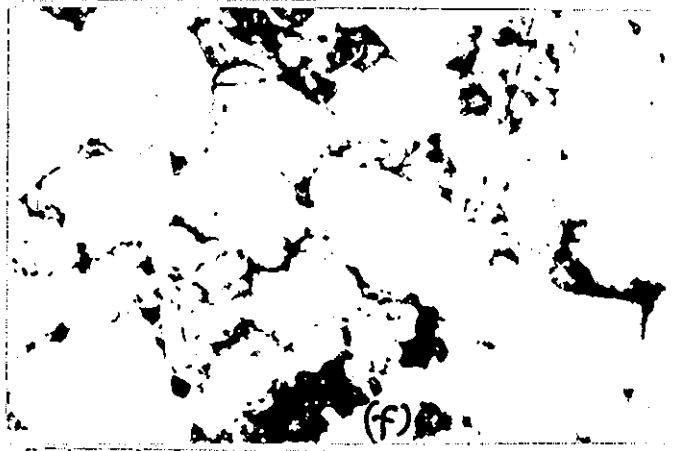
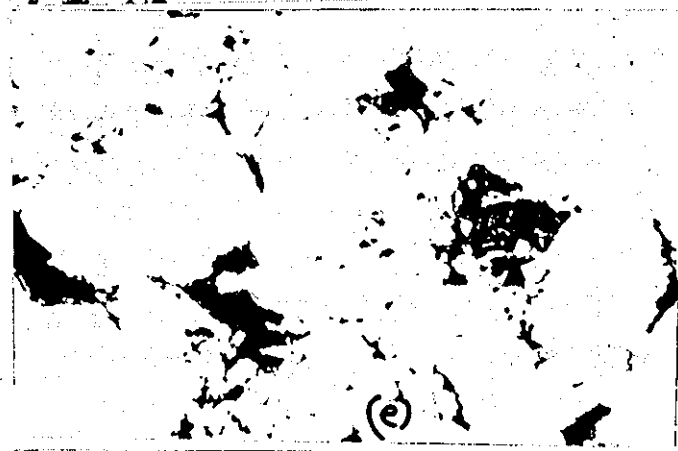
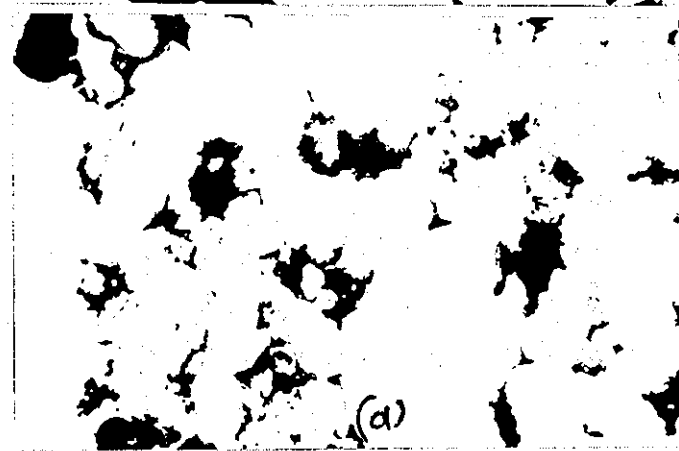
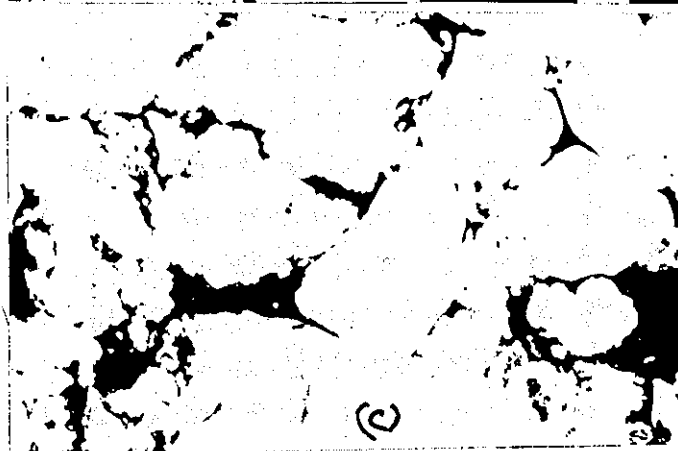
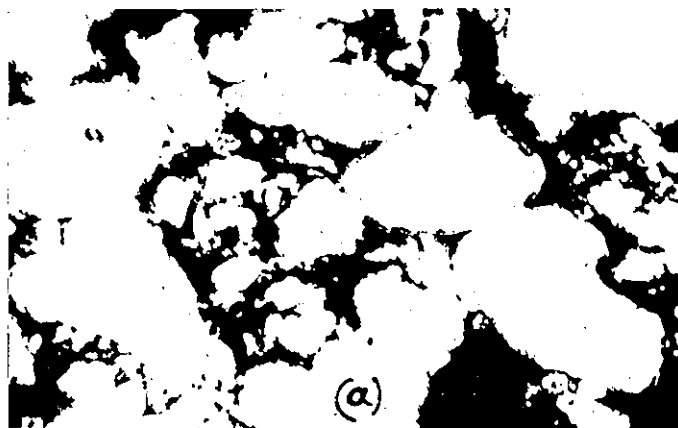
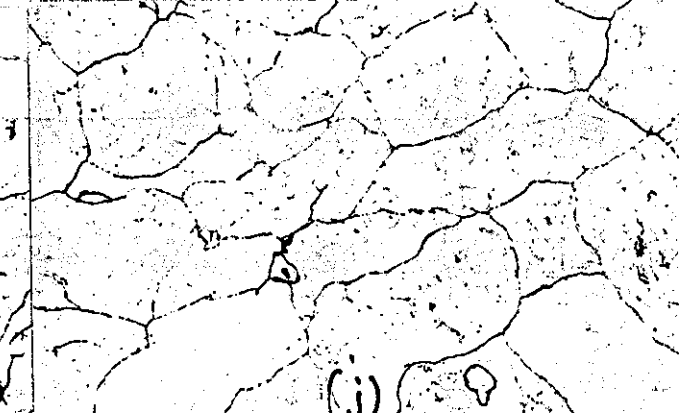
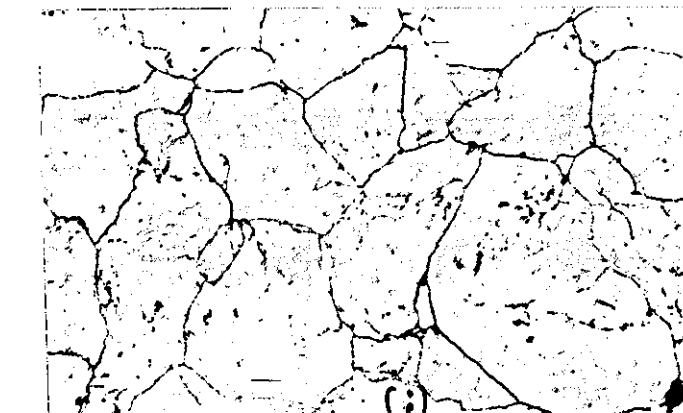
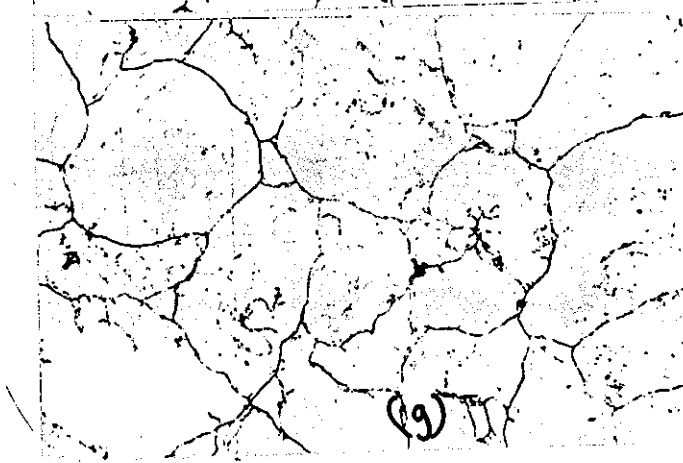
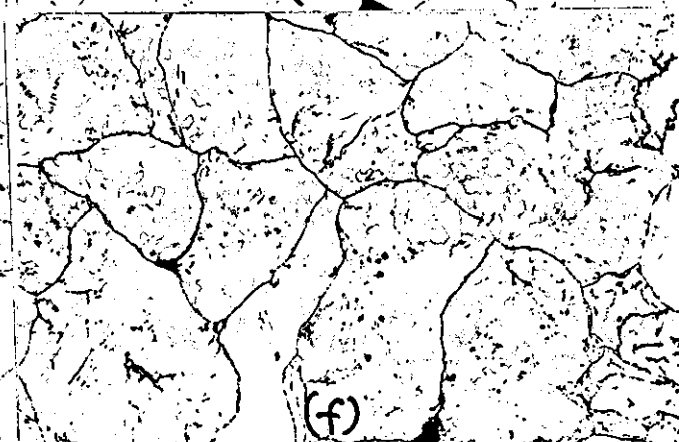
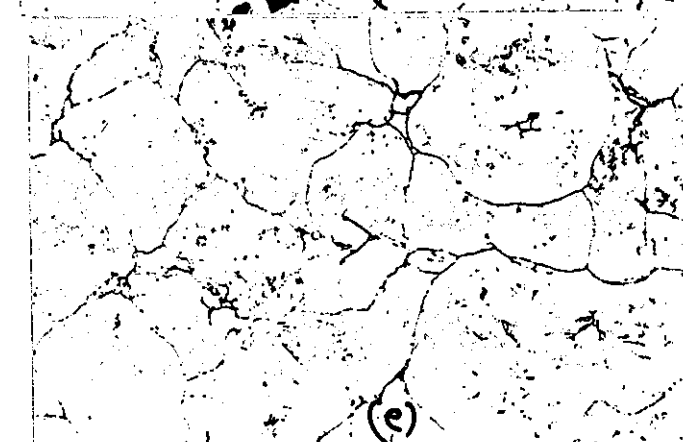
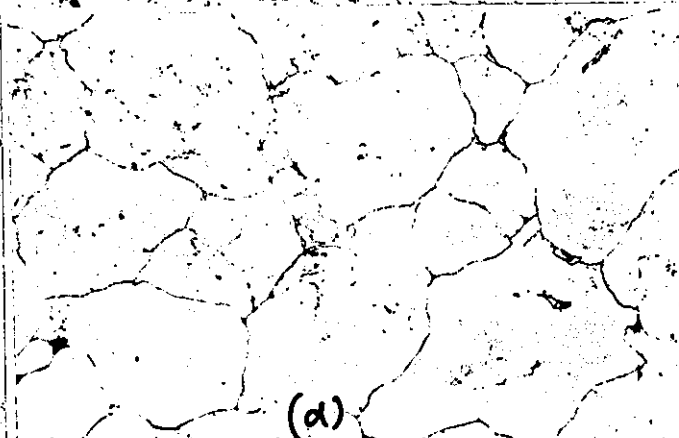
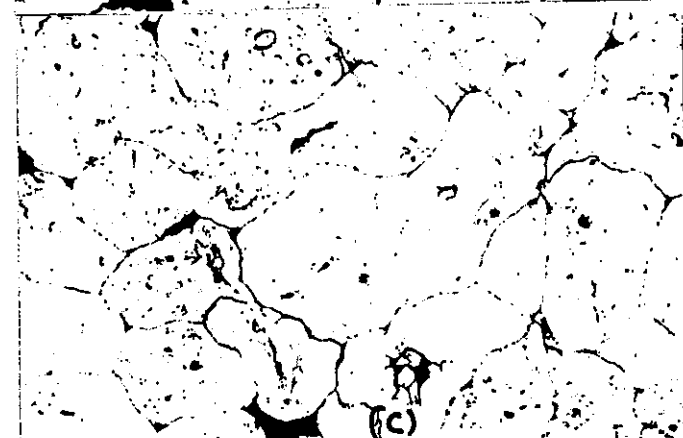
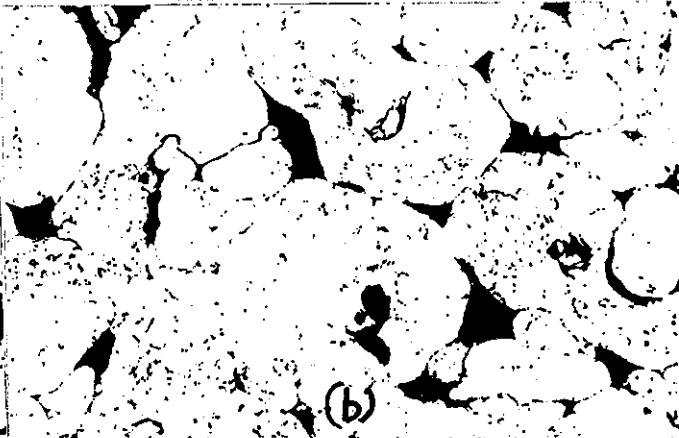


FIGURE 66

Photomicrographs of impregnated compacts of
a monosize fraction ($-125 + 106\mu\text{m}$) of Zinc

- (a) compacting pressure 5 t.s.i.
- (b) compacting pressure 10 t.s.i.
- (c) compacting pressure 15 t.s.i.
- (d) compacting pressure 20 t.s.i.
- (e) compacting pressure 25 t.s.i.
- (f) compacting pressure 30 t.s.i.
- (g) compacting pressure 35 t.s.i.
- (h) compacting pressure 40 t.s.i.
- (i) compacting pressure 45 t.s.i.
- (j) compacting pressure 50 t.s.i.

All Magnifications X 200



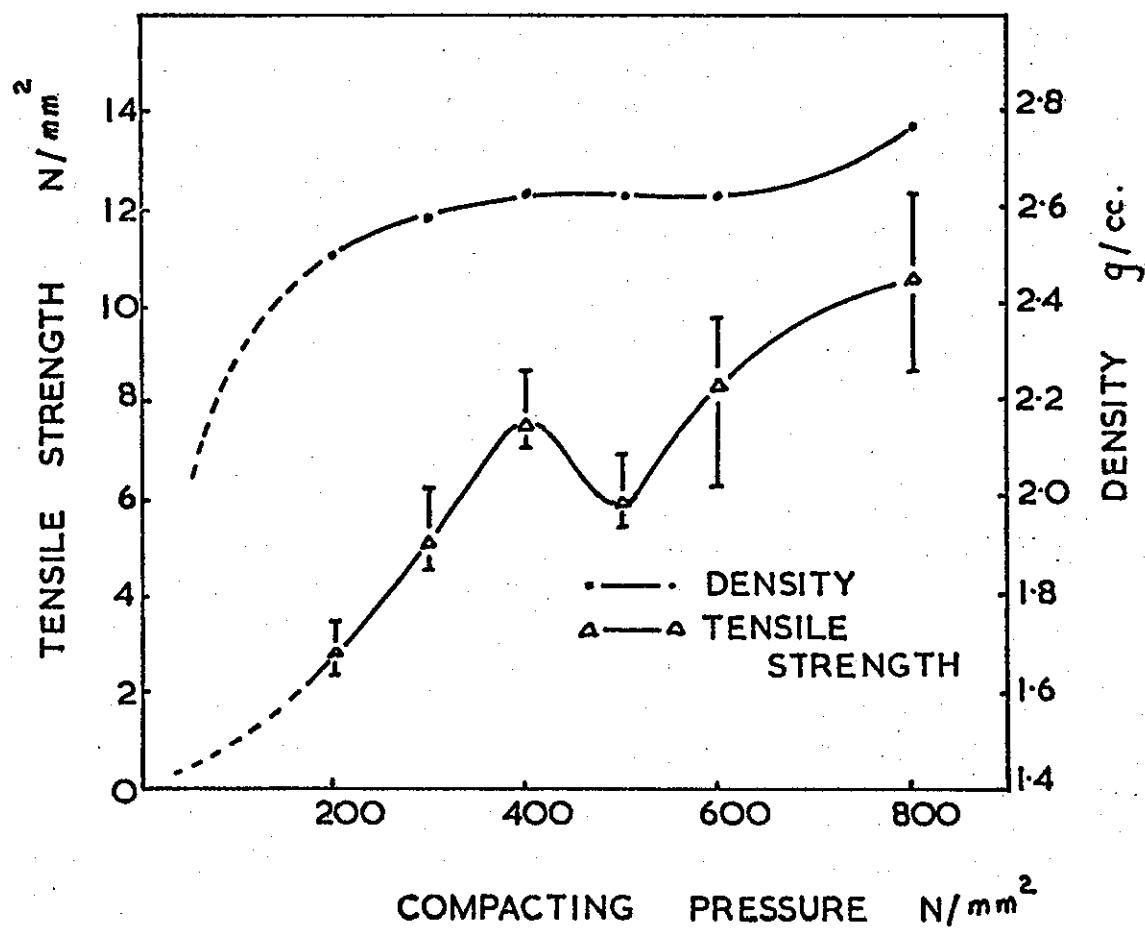


FIGURE 67

Changes in tensile strength and density of a monosize fraction (75 μm) of spherical aluminium powder (Stromgren et al ref. 53)

FIGURES 68 - 74

Changes in tensile strength and density of
multisize fractions of zinc powder with
compacting pressure

FIGURE 68	-125 + 106 μ m
FIGURE 69	-106 + 90 μ m
FIGURE 70	- 90 + 75 μ m
FIGURE 71	- 75 + 63 μ m
FIGURE 72	- 63 + 53 μ m
FIGURE 73	- 45 + 38 μ m
FIGURE 74	-38 + 00 μ m

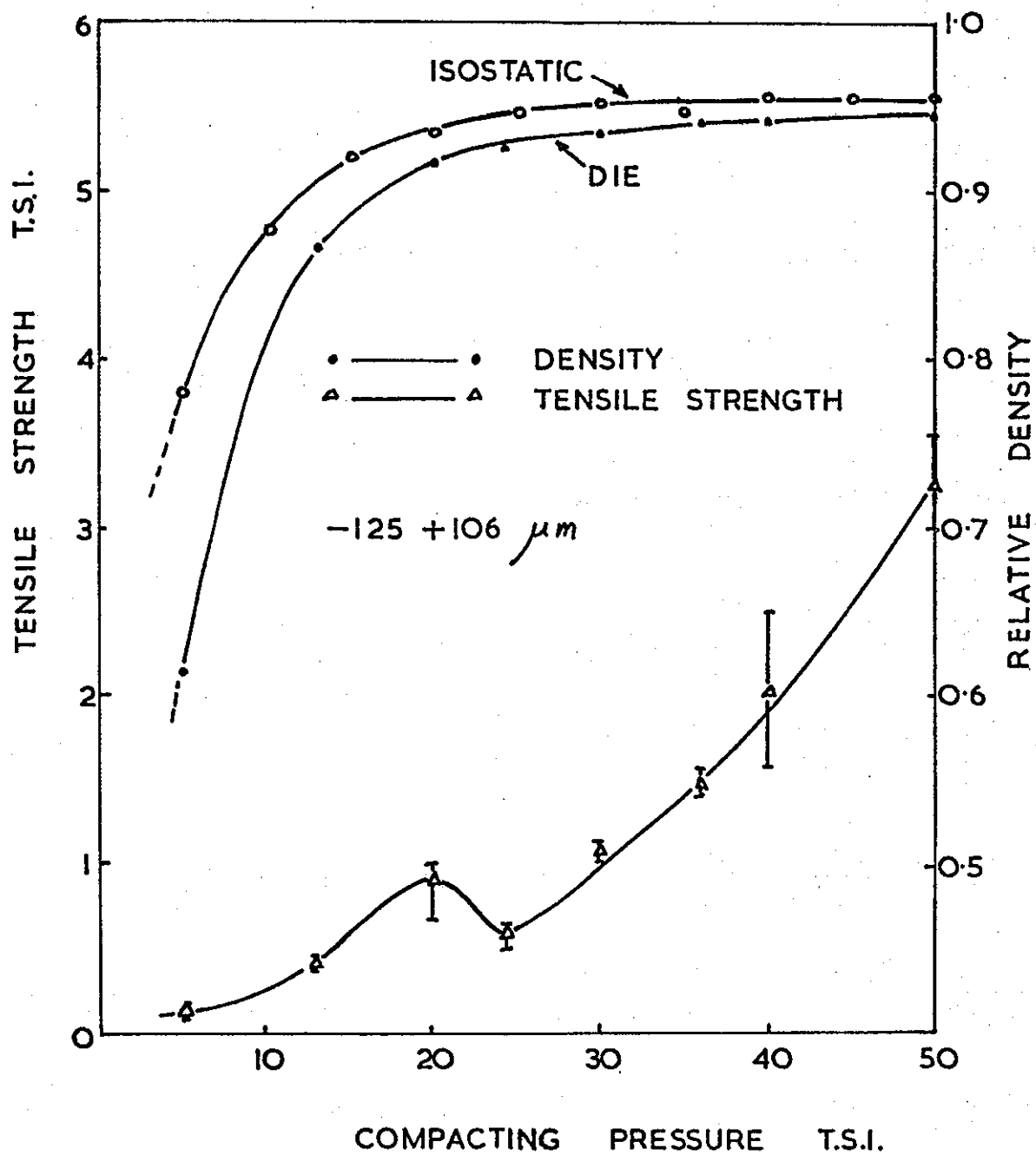


FIGURE 68

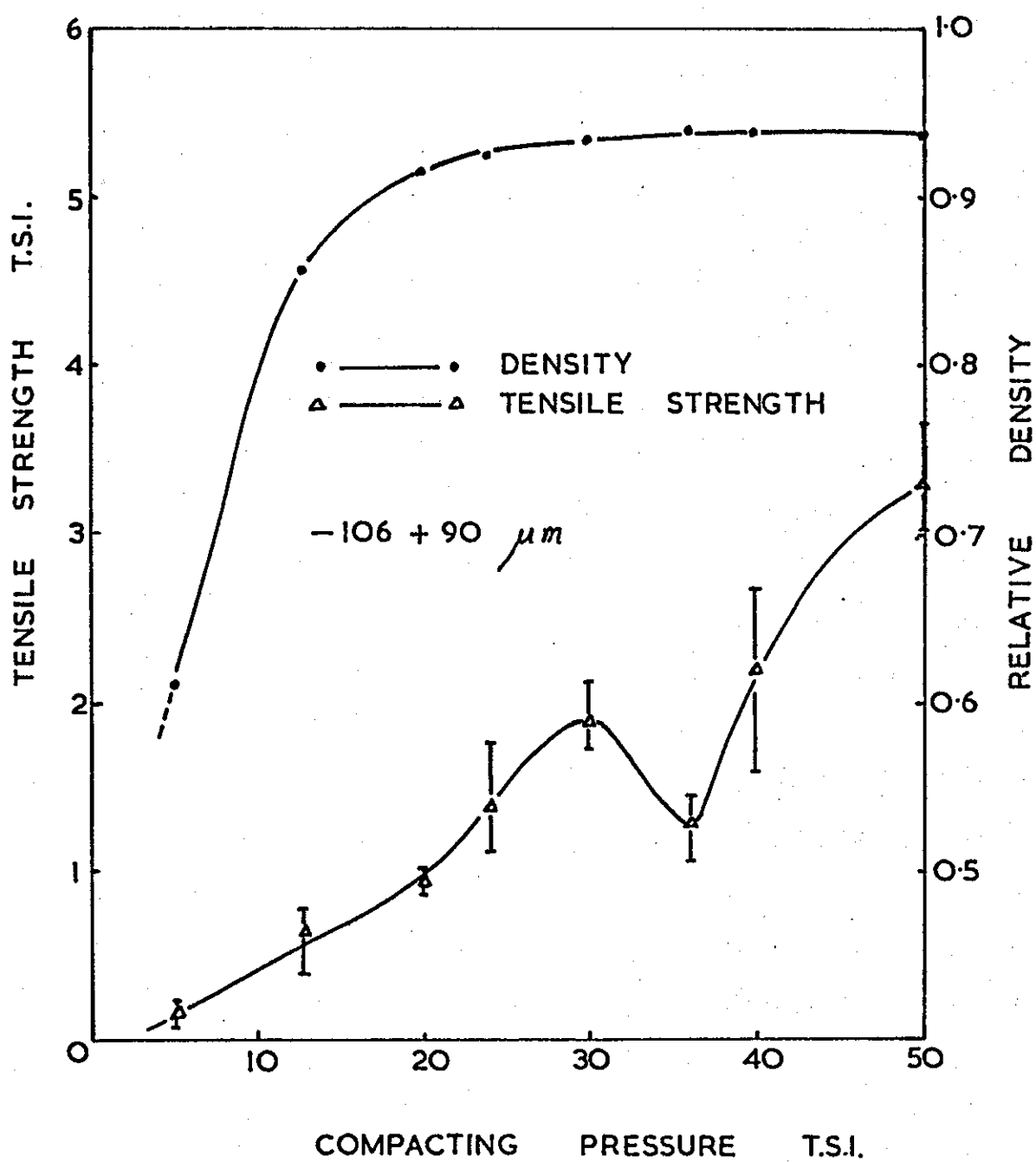


FIGURE 69

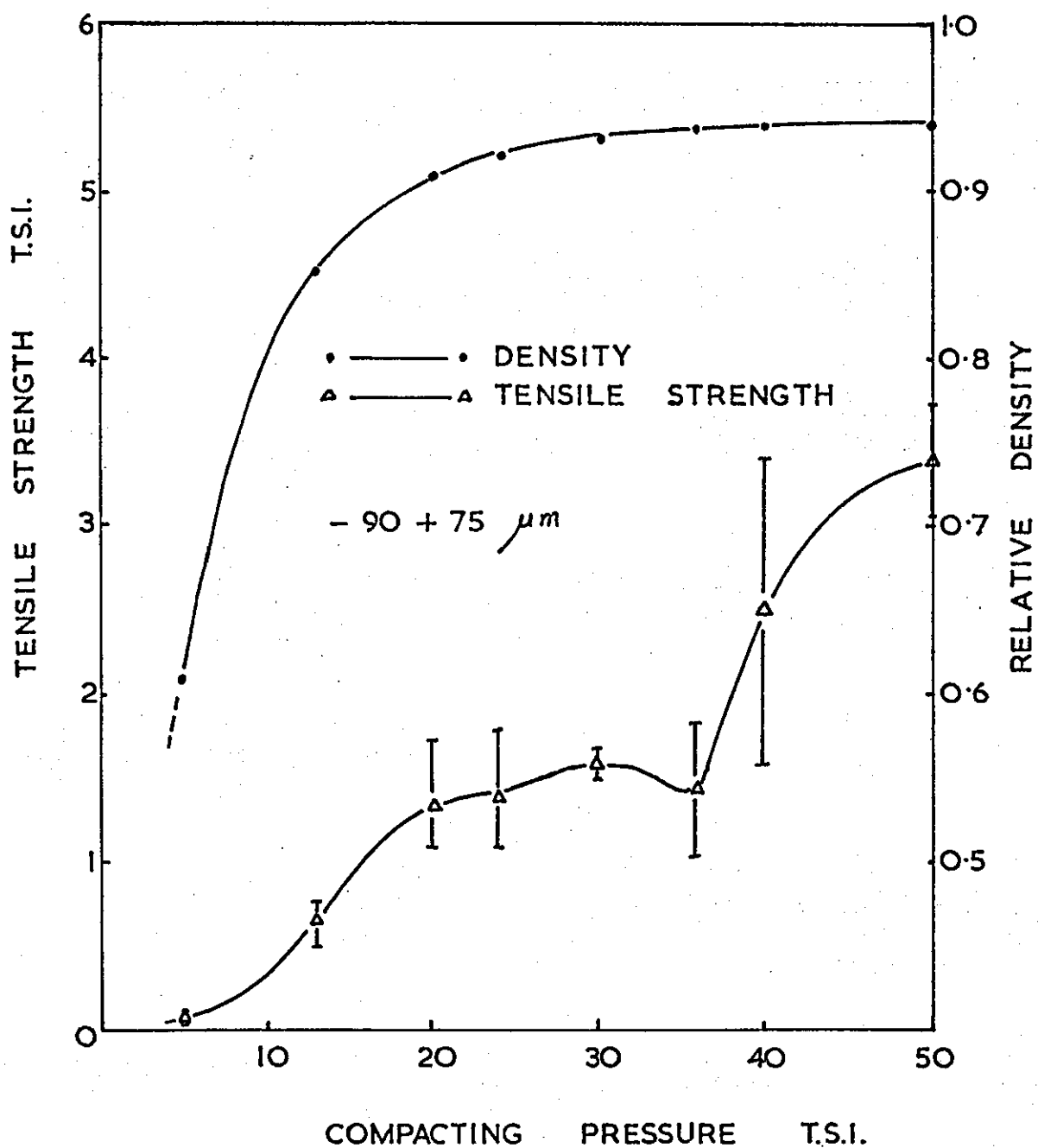


FIGURE 70

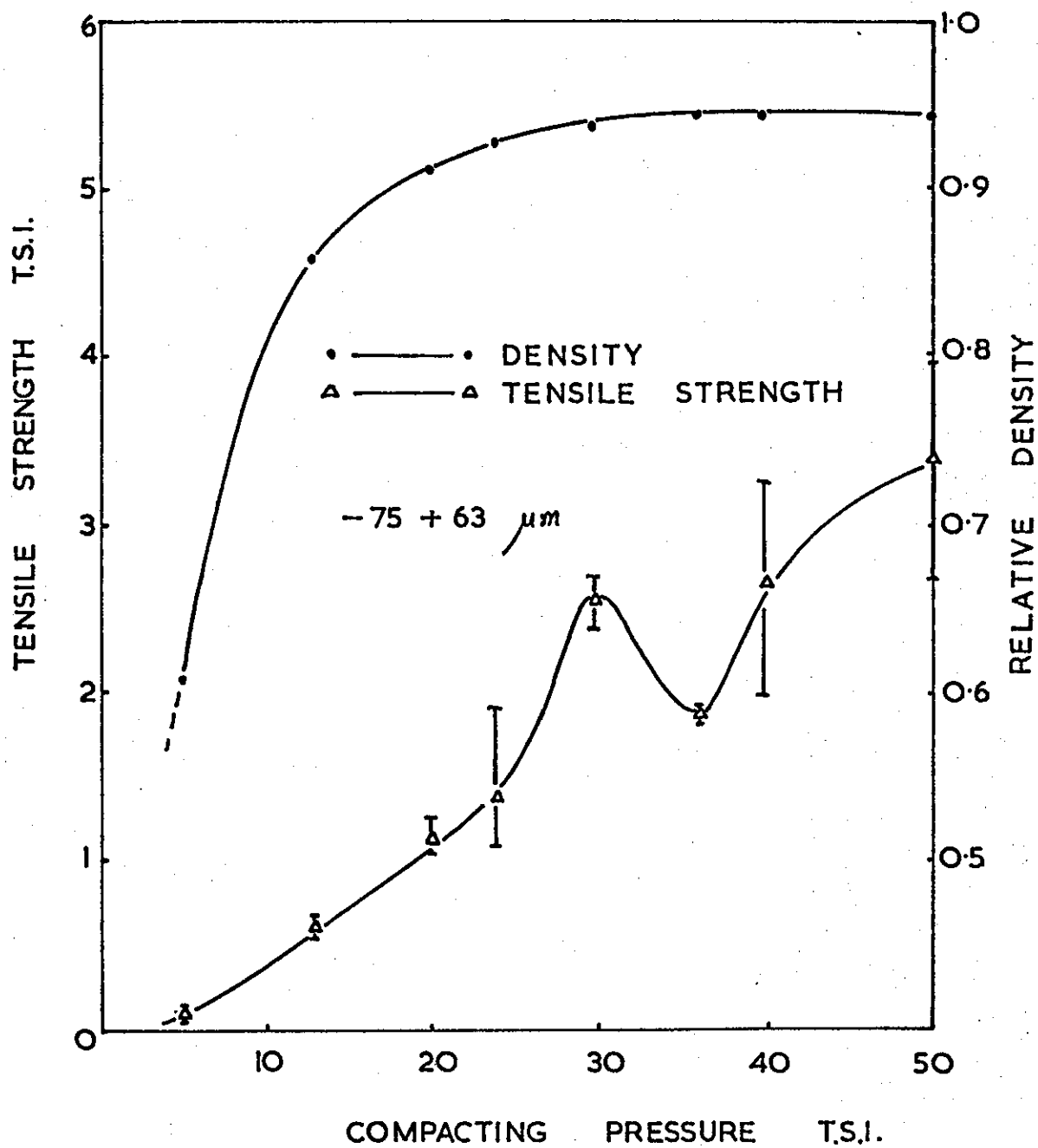


FIGURE 71

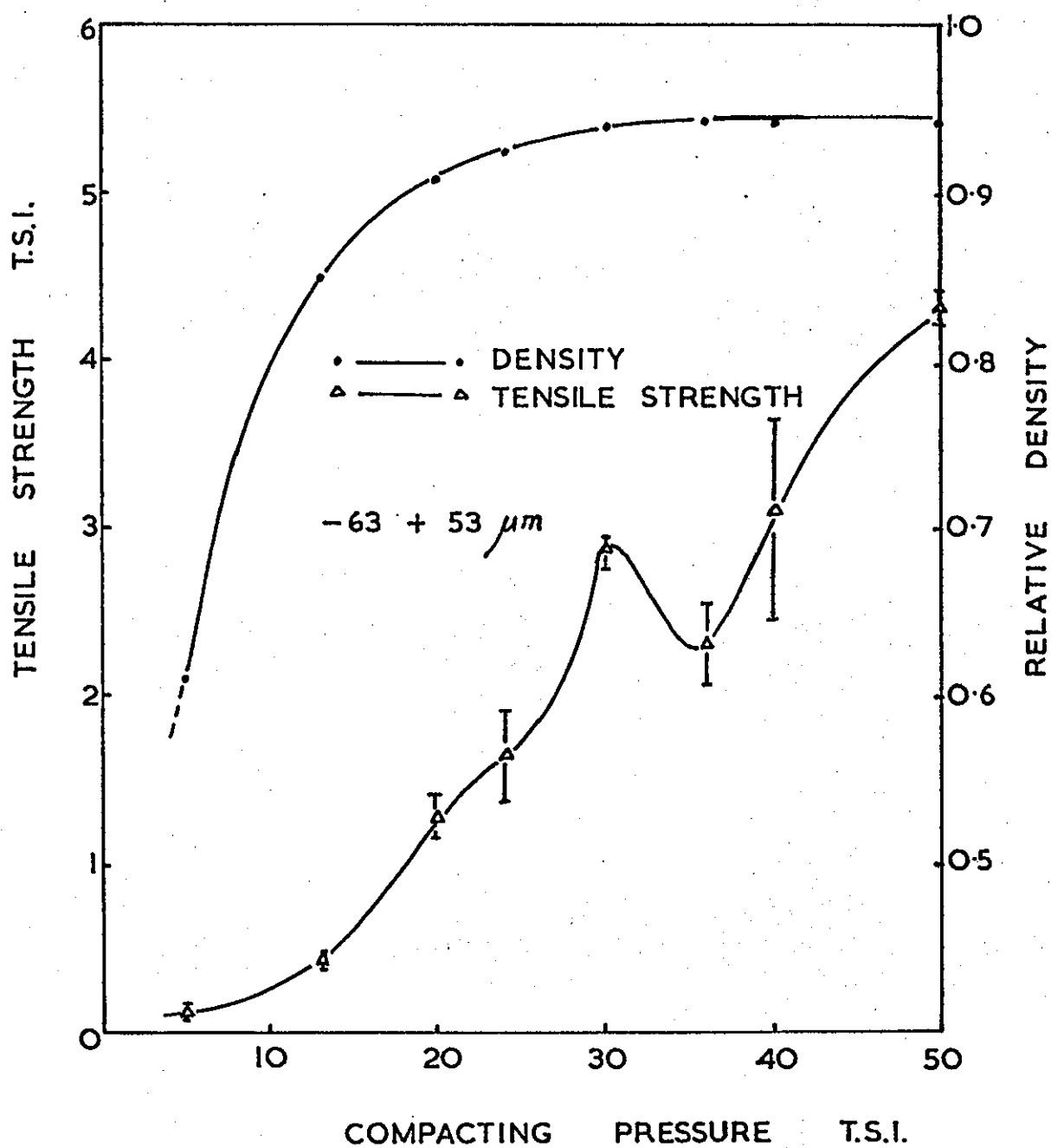


FIGURE 72

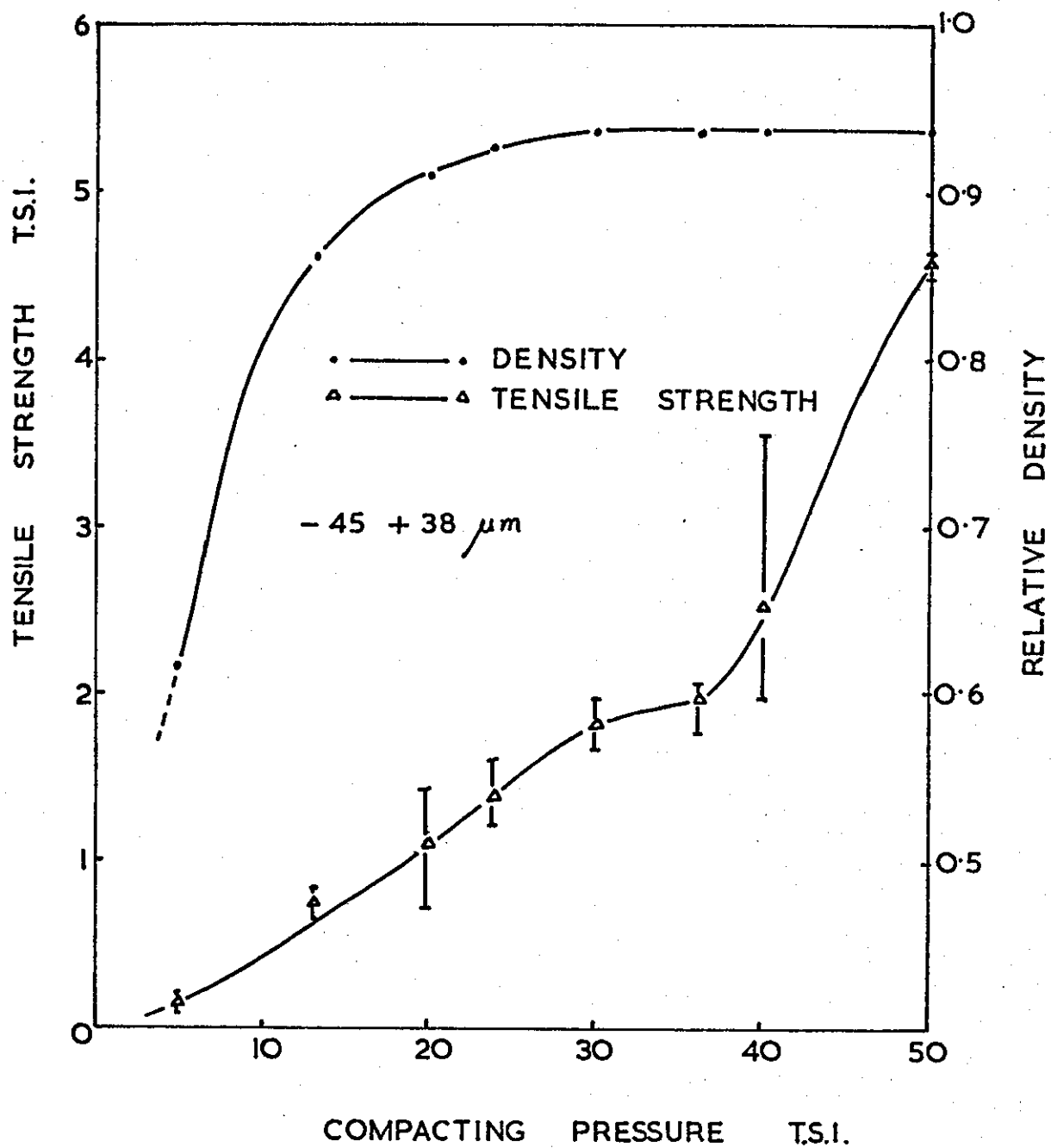


FIGURE 73

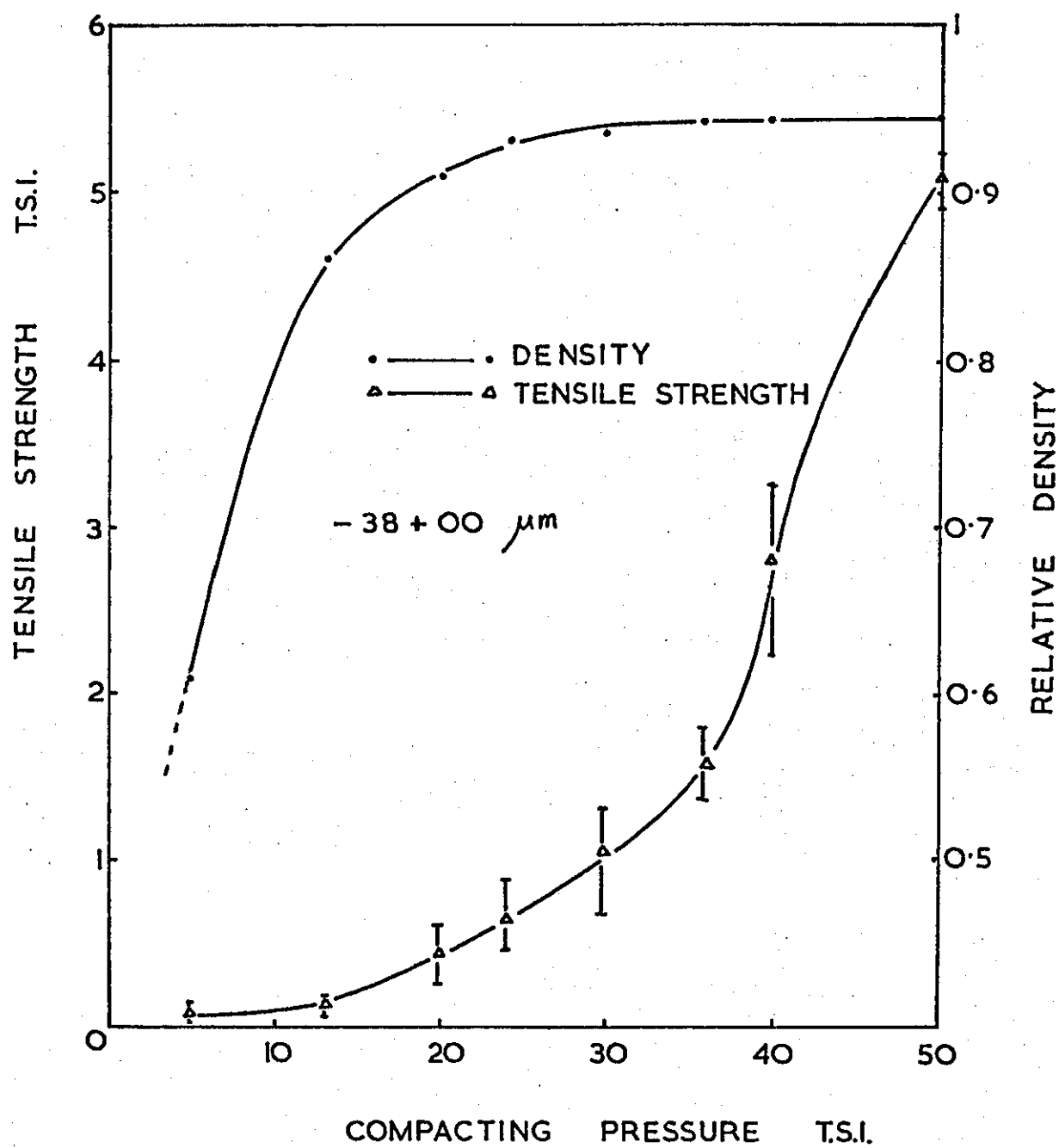


FIGURE 74

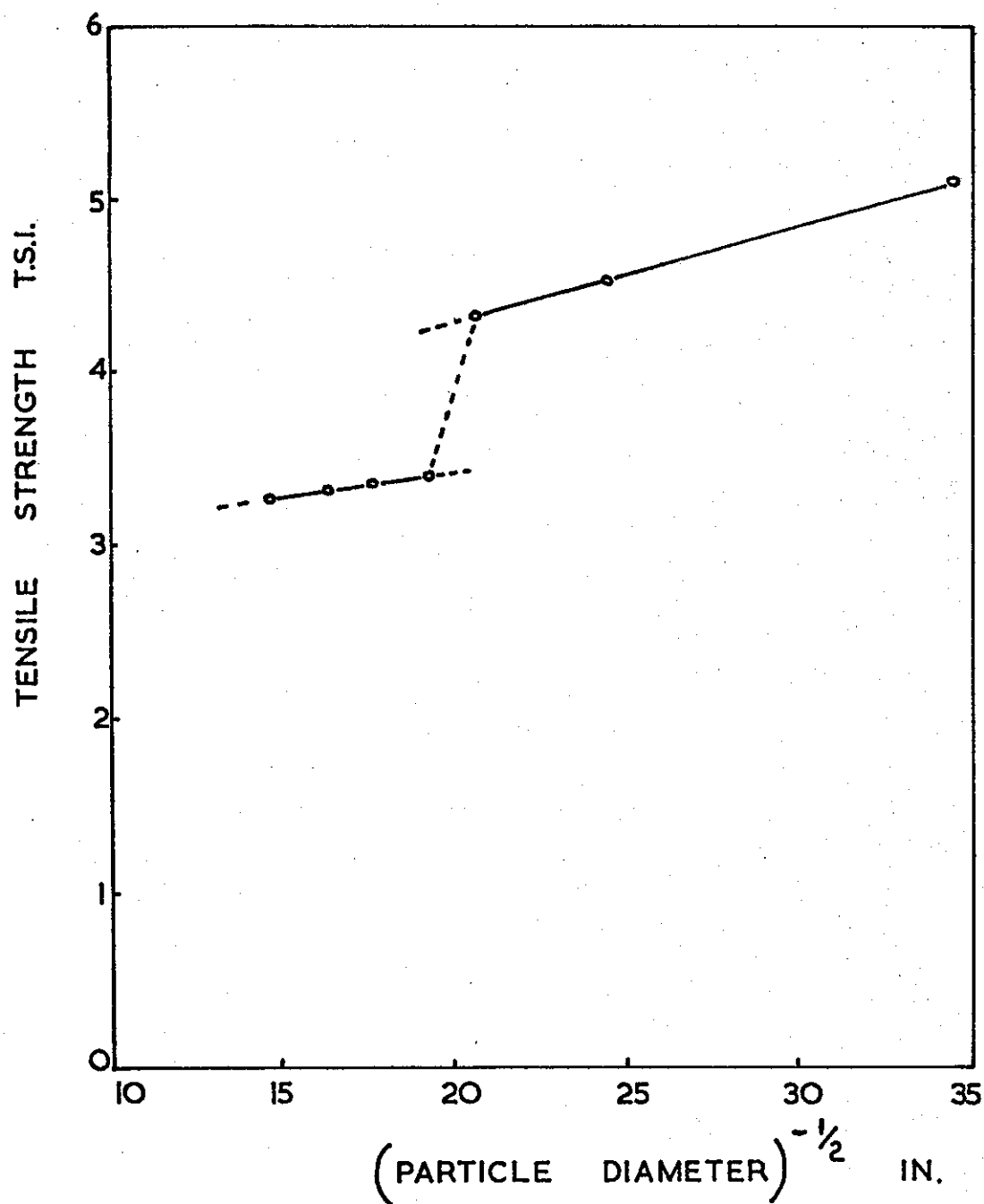


FIGURE 75

Relationship between tensile strength and
particle diameter of zinc powder compacted at
50 t.s.i.

APPENDIX IStress Calculations for Compound Die-Set Assembly.

The following calculations are based upon an envisaged maximum compacting pressure of 70 t.s.i.

According to Timoshenko¹ for the shrink fit of two cylinders, the residual internal pressure P after assembly is given by

$$P = \frac{E \cdot \delta}{b} \frac{(b^2 - a^2)(c^2 - b^2)}{2 b^2 (c^2 - a^2)} \quad \text{-----} \quad (1)$$

where E = Young's Modulus (the average value for the two materials taken to be very similar)

a = inner radius of smaller cylinder

b = inner radius of larger cylinder

c = outer radius of larger cylinder

δ = "interference", i.e. difference in radius between outer radius of smaller cylinder and inner radius of larger cylinder.

Substituting values of a = 0.6915 in.

b = 1.750 in.

and c = 4.0 in.

E = 28×10^6 p.s.i.

an interference δ of 0.004 in.

gives P = 22,507 p.s.i.

Considering each cylinder separately, subjected to this pressure :

For the outer cylinder

$$\sigma_r = \frac{b^2 \cdot P_i}{(c^2 - b^2)} \cdot \left(1 - \frac{c^2}{r^2} \right) \text{-----} (2)$$

$$\text{and } \sigma_t = \frac{b^2 \cdot P_i}{c^2 - b^2} \cdot \left(1 + \frac{c^2}{r^2} \right) \text{-----} (3)$$

where σ_r = radial stress

σ_t = tangential stress

r = radius at any point within the cylinder

P_i = internal pressure (22,507 p.s.i.)

thus from equations (2) and (3)

when $r = b$ $\sigma_{rb} = -22,507$ p.s.i.

$\sigma_{tb} = 33,162$ p.s.i.

when $r = c$ $\sigma_{rc} = 0$ p.s.i.

$\sigma_{tc} = 10,655$ p.s.i.

For the inner cylinder

$$\sigma_r = - \frac{b^2 \cdot P_o}{(b^2 - a^2)} \cdot \left(1 - \frac{a^2}{r^2} \right) \text{-----} (4)$$

$$\text{and } \sigma_t = - \frac{b^2 \cdot P_o}{(b^2 - a^2)} \cdot \left(1 + \frac{a^2}{r^2} \right) \text{-----} (5)$$

where P_o = external pressure (22,507 p.s.i)

Thus from equations 4 and 5

when $r = b$ $\sigma_{rb} = -22,507$ p.s.i.

$\sigma_{tb} = 30,836$ p.s.i.

when $r = a$ $\sigma_{ra} = 0$ p.s.i.

$$\sigma_{ta} = 53,343 \text{ p.s.i.}$$

If the assembly is now considered as a monobloc with an internal bore pressure of 70 t.s.i.

$$\sigma_{ta} = 74.3 \text{ t.s.i.}$$

$$\sigma_{tc} = 4.31 \text{ t.s.i.}$$

N.B.

G110 which is a double vacuum melted quality maraging steel of Firth-Brown Limited, was aged for 3 hours at 480°C from the original as-supplied (solution treated) condition. The 0.2% Proof Stress value was 115 t.s.i. sufficiently in excess of the maximum developed stress within the bore of 74.3 t.s.i. during compaction.

Reference

- 1 S. Timoshenko Strength of Materials Part 2 1956
Van Nostrand London

APPENDIX 2Calculation of Surface Area of Powders

Using Kozeny's¹ equation as modified by Carman² :

$$S_w^2 = \frac{1}{K\eta\rho^2u} \cdot \frac{e^3}{(1-e)^2} \cdot \frac{\Delta p}{L} \text{ ----- (1)}$$

Where S_w = specific surface area per unit weight: $\text{cm}^2.\text{g}^{-1}$

K = aspect factor assumed = 5

η = viscosity of air : P (0.000182 at 22°C)

ρ = density of powder : g. cm^{-3}

u = volumetric flow rate/cross sectional area of cell

e = (volume of cell) - (volume of powder)

= volume of 'open' air voidage in cell

Δp = pressure drop across cell

L = length of powder column cm

From these data:

$$e = 1 - \frac{4W}{\rho\pi D^2L} \text{ ----- (2)}$$

Where W = weight of powder g

D = dia. of cell c m.

$$\text{and } u = \frac{dV}{dt} \cdot \frac{4}{\pi D^2} \text{ ----- (3)}$$

where $\frac{dV}{dt}$ is the volumetric flow rate.

$$\text{and } \Delta \rho = H \rho_w \cdot g$$

$$\rho_w = \text{density of water (0.9975 g. cm}^{-3} \text{ at } 22^\circ\text{C)}$$

$$H = \text{water head in burette} = \text{cm.}$$

$$\text{and } g = \text{gravity constant}$$

From (1) (3) and (4)

$$\frac{dV}{dt} = \frac{H \rho_w g \pi D^2}{4K\eta \rho^2 L S_w^2} \cdot \frac{e^3}{(1-e)^2} \quad \text{-----} \quad (5)$$

Integrating between V_1 and V_2 , representing a range of volume of air sucked through the column :

$$\int_{V_1}^{V_2} \frac{dV}{H} = \int_{V_1}^{V_2} \frac{dV}{V} \cdot \frac{V}{H} = \int_{t_1}^{t_2} \frac{\rho_w \cdot g \cdot \pi D^2 e^3}{4K\eta \rho^2 L S_w^2 (1-e)^2} dt \quad \text{----} \quad (6)$$

$$\text{i.e. } \frac{V}{H} \cdot \log_{10} \frac{V_2}{V_1} \cdot 2.303 = \frac{(t_2 - t_1) \rho_w g e^3 \pi D^2}{4K\eta \rho^2 L S_w^2 (1-e)^2} \quad \text{---} \quad (7)$$

$\frac{V}{H}$ represented the volume of water per unit head of water in the burette; for the burette and beaker used the reciprocal value $\frac{H}{V}$ was measured and a mean value of 0.673 used.

Rearranging

$$S_w^2 = \frac{(t_2 - t_1)}{(\log_{10} V_2 - \log_{10} V_1)} \cdot \frac{g \rho_w e^3 \pi D^2}{4K\eta \rho^2 L (1-e)^2} \cdot \frac{H}{V} \quad \text{-----} \quad (8)$$

and substituting for e in equation 2 gives

$$S_w^2 = \frac{1}{K\eta \rho^2 L} \left[\frac{1 - \frac{4W}{\rho \pi D^2 L}}{\frac{4W}{\rho \pi D^2 L}} \right]^3 \cdot \frac{t_2 - t_1}{\log_{10} V_2 - \log_{10} V_1} \cdot \frac{\rho w g \pi D^2}{4.2.303} \cdot \frac{H}{V} \quad (9)$$

From equation (9) an Olivetti desk computer programme was derived and recorded on a master card. The sequence of operations there-after ran as follows:

- 1 Press General Reset Switch
- 2 Check 3 centre buttons are all out → Record RR,
Print PR, key B relay.
- 3 Insert master card applicable to programme,
- 4 Switch decimal place to six
- 5 Press routine selection key W
- 6 Enter 44.936338 C/M↑
- 7 Enter 1.272727 C M↑
- 8 Enter density of powder D/M↑
- 9 Enter diameter of cell D M↑
- 10 Enter viscosity of air F M↑
- 11 Enter length of powder column
- 12 Press start button
- 13 Enter weight of powder
- 14 Press start button
- 15 Enter slope from graph ordinate first
- 16 Press start button
- 17 Enter abscissa
- 18 Press start button
- 19 Print out gives surface area of powder

References

- 1 J Kozeny Über Grunwasserbewegung Wasserkraft und
 Wasserwirtschaft 1927 22 .67
- 2 P C Carman J. Soc. Chem. Ind. 1938 57 225.

APPENDIX 3Calculation of green density by the MercuryBalance method

The procedure adopted was as follows

- (a) The dish containing cleaned triple distilled mercury was placed in an outer dish and the whole placed on the top pan balance and weighed W_1
- (b) The sinker was lowered into the mercury until contact of the probe was achieved and the weight recorded W_2 ($> W_1$).
- (c) The sinker was removed, the specimen carefully placed on the mercury surface and the weight recorded W_3 .
Thus the specimen weight is $W_3 - W_1$.
- (d) The sinker was carefully lowered to submerge the compact until, again contact of the probe was achieved and the weight recorded : W_4 . Thus the specimen weight plus the "down thrust" to immerse the specimen is $W_4 - W_2$.

$$\text{The density of the compact} = \frac{W_3 - W_1}{W_4 - W_2} \times 13.546$$

