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Some properties of sintered materials

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SOME PROPERTIES OF
SINTERED MATERIALS

by

S. H. WILLIAMS

TIME-TEMPERATURE-DEPENDENT

A Thesis
Submitted for the Degree of
Doctor of Philosophy
Loughborough University of Technology

Supervisor: Dr. R. Haynes

Department of Materials Technology.

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SUMMARY.

The mechanical properties of die compacted sintered nickel compacts prepared from closely sized sieve fraction of annealed Sheritt Gordon were determined.

The properties, especially fatigue properties, were found to be dominated by the presence of film like porosity in the grain boundaries. The occurrence of this porosity was at the original particle boundaries and its form was attributed to botryoidal shape of the original powder particles.

A mechanism for the propagation of the fatigue crack is suggested.

Several iron powders were carefully characterised. Thin steel strip was prepared from these samples using a slurry technique. Binder was used in the slurry to give coherent strip before rolling.

The properties of the green strip were found to be affected by the particle size and particle size distribution of the original powder. The presence of binder in the strip contributed towards the green strength especially at high roll loads.

Strength was observed to develop quickly during sintering, for sintering times of the order of sixty seconds duration although little or no densification occurred.

Specimens of near theoretical density were prepared by re-rolling and resintering. Despite the high density of these specimens the mechanical properties were poor.

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INTRODUCTION

1.00. INTRODUCTION.

Sintered materials are manufactured from metallic, ceramic or plastic powders. The powders are generally compacted to a coherent form and then sintered at an elevated temperature so that diffusion occurs between the powder particles, strengthening the particle/particle bonds. Densification may occur during sintering. Metal powders are usually sintered in an inert or reducing atmosphere to prevent oxidation.

Although this work is primarily concerned with the processing and behaviour of metal powders, many of the techniques and theories associated with the compaction and sintering of metals are equally applicable to both ceramics and plastics.

1.10. THE MANUFACTURE OF SINTERED METALS.

Most stable metals may be processed easily by the powder route. Metal powders may be prepared by a number of techniques involving either comminution, atomization or the direct reduction of metal salts in the solid form or in solution. Therefore particles of metal powders may have a variety of shapes depending upon the manufacturing technique employed. Consequently powders are classified as acicular, spongy, angular or spheroidal.

Although particle size may vary from powder to powder most commercial metal powders are finer than 80 mesh and the smallest particles are of the order of a micron in diameter. The largest fraction by weight is frequently between 150 mesh and 250 mesh.

Compaction of the metal powder is usually carried out in dies but recently other forms of compaction by extrusion or rolling have become of interest. When the metal powder is die compacted it is frequently blended with a lubricant, e.g. a metal stearate. The lubricant enables the powder particles to slide more easily over each other and so pack more closely together, also die wear is improved. Occasionally the lubricant is applied to the die wall only. Lubricants are generally not used when the metal powder is rolled.

Powder metallurgy dies may be single acting where only the top plunger moves relative to the die, or double acting, where both plungers move. Double acting dies give a more uniform stress distribution in the powder bed, moreover the use of two plungers enables the pressed compact to be ejected easily. In practice compaction pressures are usually in the range $230 - 475 \text{ N/mm}^2$ (15 - 30 t.s.i.) but pressures either greater or less than this may occasionally be used.

In certain applications the metal powder may be heated during compaction. This technique eliminates subsequent sintering and parts may be produced of very high density. However the tooling cost is considerably more expensive than the cost of cold pressing tools and the die life is reduced.

After compaction, the lubricant must be removed from the compact before sintering as the rapid heating rates involved in sintering cause rapid volatilization of the lubricant and the pressure of lubricant vapour may be sufficient to rupture the compact. In practice the compact is heated slowly to a

low temperature so that the lubricant volatilizes slowly and escapes from the compact. This procedure is frequently termed "de waxing". Thereafter the component is sintered in a reducing or inert atmosphere at a temperature above the minimum recrystallization temperature but beneath the melting point of the component.

Alloys may be processed by the powder route. The alloying elements may be introduced either by admixing powder of the alloying element with that of the base metal or by prealloying the material during powder manufacture.

Occasionally 1 - 2 per cent of a constituent of lower melting point than the basis powder is introduced, e.g. 1 - 2 per cent bronze in iron, and sintering is carried out above its melting point. This technique is known as liquid phase sintering and generally faster densification is achieved as well as superior mechanical properties by this method.

Sintered materials invariably contain some porosity which reduces the realisation of optimum mechanical properties. Consequently for certain applications high density parts are required. In order to achieve this, the sintered material may be recompact and resintered. The recompact of sintered parts in a die is known as "coining" and in practice pressures similar to or greater than the original compaction pressure must be used if significant densification is to be realised. "Coining" at lower pressures without resintering is frequently used to size sintered parts.

1.20. THE ADVANTAGES OF A POWDER ROUTE.

A powder route enables material to be processed with very few operations to the finished shape and size. Consequently die compaction can produce parts of complex design and close size tolerance without the expensive and lengthy machining operations that would be required to manufacture such parts conventionally. However because of expensive tool costs the powder route is more suitable for the manufacture of parts for which large numbers are required, e.g. gears, rocker arm brackets etc.

On the other hand tooling costs are reduced in powder rolling because the number of production stages compared to conventional strip production are markedly reduced. Fig.1.¹ compares a powder route and a conventional route for steel strip production. Conventional rolling processes dictate that progressively more passes are required to reduce strip thickness as the strip becomes thinner. Consequently the cost of very thin gauge strip is very high (Fig.2²). Most powder routes roll strip of finished gauge using two or three rolling stages, the first rolling operation being done very near to the finished strip thickness. Therefore the cost of thin strip produced by a powder route may be less than strip produced by a conventional route especially when highly alloyed strip is considered, e.g. stainless steel which is difficult to roll conventionally.

A compaction and sintering process enables highly alloyed or high melting materials to be manufactured more

easily because machining or grinding operations are minimised and because sintering is carried out beneath the melting point of the material. Thus a powder metallurgy route is very suitable for manufacturing metal carbides or tungsten. The compaction of highly alloyed materials is facilitated by using elemental powders which are considerably softer than the equivalent prealloyed powders. Material transport by diffusion during sintering develops the full mechanical properties of the alloy.

A powder route has enabled materials to be developed which cannot be manufactured conventionally. The control of porosity such that large amounts of interconnecting porosity are present in the finished material has allowed the development of self lubricating bearings. Porous nickel strip of high surface area which is used in electric cells is produced by a roll compaction process.³

Hydrometallurgical processes for the extraction of metal from ores are becoming of increasing importance, especially as leaner metal ores have to be worked. These processes directly manufacture metal powders, therefore a production route which uses metal powders as a raw material is economically more sound than a production route which would require the metal powder to be cast into a billet before processing.

Very many alloys have been developed because of the large number of applications for which metals are required. However the alloying elements which are added to the basis element are usually very few and properties are altered by varying the proportions of each element. If a powder production

route is used for the components then the manufacturer only has to stock a few elemental powders in order to be able to produce a wide range of alloys.

1.30. CURRENT APPLICATIONS OF SINTERED METALS.

Although it has been appreciated that metal powders can be used to manufacture parts and engineering materials for some time, the commercial growth of the industry began in the 1930's.

Most metal powder processes today die compact metal powder and die compacted metal parts have established themselves in the fields of bearings, bushes, gears, clutch and brake linings, hard metals, etc..

However despite the importance of the die compaction industry the powder metallurgy industry does not process a large tonnage of metal compared to the tonnage that is consumed by industries using more traditional techniques.

Consequently because metal powders are manufactured in small batches, the cost of metal powder compared to the cost of metal of similar composition supplied in the billet form is high, and it is only because of the production advantages of sintered materials that the technique is economic. However powder rolling is a process that would consume large amounts of metal powder and its adoption may substantially reduce powder costs.

The roll compaction of powders is not yet in general use although a number of investigators have advocated its adoption and pilot plants have been developed for many ferrous and non-ferrous alloys which have shown that the technique is practicable.

As early as 1857⁴ Bessemer showed that coherent strip could be made from particulate materials by passing brass turnings through a rolling mill. A patent was filed in 1902 by Siemens and Halske⁵ which claimed advantages for the roll compaction of high melting point materials compared with die compaction for the manufacture of wire bars.

More recently (1942) Mannesmann (Germany)⁶ developed a pilot plant for rolling steel strip. Sundwiger and Messingwerke A.G. in 1951 and later Chemetals manufactured copper and copper alloy strip from chemically reduced powders. However due to economic reasons, chiefly because of the high cost of metal powder, the work was terminated. Aluminium and stainless strip have been manufactured for atomic energy applications by H. Hausner and the Sylvania Electrical Products Ltd.

In the United Kingdom work has been carried out by Cambridge University⁷, the International Nickel Corporation⁸, the British Non Ferrous Metals Research Association⁹ and the British Iron and Steel Research Association² which is now part of the British Steel Corporation. It has been conclusively shown from these investigations that strip similar in properties to conventionally rolled strip may be manufactured. However the large difference in price between molten metal in the ladle and metal powder has generally precluded commercial development, despite possible production savings thereafter.

Therefore it appears that powder rolling is most likely to be used in the near future for high melting point

materials, materials that possess special properties, e.g. very porous nickel for electric cells and very thin gauge material, particularly if it is highly alloyed.

Commercially Sheritt Gordon Mines Ltd. manufactures nickel strip from powder which is used by the Royal Canadian Mint¹⁰ also Reynolds Metal Company¹¹ have processed aluminium and aluminium alloy powder to strip. Nickel strip is also made on a small scale by General Telephones and Electronics¹².

1.40. THE PROPERTIES OF SINTERED MATERIALS.

Sintered materials usually contain some residual porosity and so their properties are not equivalent to the properties of wrought materials of similar composition. However it has been shown¹³ that when the density of sintered materials approaches the theoretical density their properties are very similar to the properties of conventionally produced materials. This is confirmed by the results of investigators who have examined the properties of sintered strip where it is easier to attain the higher compaction forces that are required to give very high density material.

However it is usually not feasible to die compact metal powders to such high densities and in practice porosity contents of the order of 10 per cent are common. Consequently P/M parts have been, in the past, used in low stress conditions but recently there has been a trend to use them in more rigorous conditions in which cycling stresses may be present, e.g. gears, valve rocker brackets, sewing machine components etc.

The appreciation that a difference exists between the life of traditionally manufactured parts under a cycling load and life under a static load soon developed as the use of engineering machinery increased in the 19th century. Such a difference was explained by the metal "tiring" or becoming "fatigued". It was later realised that fatigue fractures were the most common type of metal failure and a knowledge of fatigue behaviour soon became an aspect of design.

Because sintered components have been used in low stress situations it has not been necessary to have detailed knowledge of their fatigue behaviour but as sintered parts are rapidly gaining acceptance and are being used in more highly stressed situations a greater knowledge of their fatigue behaviour is required.

Porosity is the distinguishing feature of most sintered parts. Many authors have shown that it has the major effect on the mechanical properties of sintered metals. It has also been found that other factors such as pore size and shape may have a significant effect upon mechanical properties although this effect is much smaller than that of porosity content.

A little information has been published concerning the influence of such factors as powder type, compaction pressure, sintering time and sintering temperature upon the fatigue properties of sintered metals. However the influence of these factors primarily affects density, pore type and grain size which in turn affects the fatigue properties.

1.50. FATIGUE TESTING AND TERMINOLOGY.

Fatigue testing may be carried out on finished articles or on small laboratory samples which have been carefully machined from the bulk material. Generally it is not possible to predict service life from laboratory tests, however in more fundamental studies of fatigue, the added complications of service testing must be avoided.

Laboratory fatigue tests may be conducted in rotating bending, push/pull, plane bending or torsional fatigue. In order to examine the properties of a material, identically machined test pieces are usually tested at different levels of stress (S) and the number of cycles to failure (N) determined. Frequently S is plotted as the ordinate and N as the abscissa, the graph is known as an S/N diagram.

Two forms of S/N curves are observed in practice (Fig.3). Curve A is typical of ferrous materials and the curve may be divided into two parts, the first in which the curve falls steeply at higher stresses and the second in which it becomes horizontal. The well defined stress at which the horizontal section occurs is known as the fatigue limit. Curve B is typical of most non ferrous materials; there is no well defined fatigue limit but the curve becomes less steep as the level of stress falls.

For samples that show a well defined fatigue limit, failure will not occur at any finite number of cycles below this limit. Samples that do not show a fatigue limit are

characterised by an endurance limit which is the stress required to give failure after a specified number of reversals, usually 10^7 or 10^8 reversals.

The ratio of fatigue or endurance limit to tensile strength is defined as the fatigue ratio and for most metals this ratio is reasonably constant and characteristic of the material.

Fatigue behaviour is markedly influenced by the surface condition and environment of the specimen. Notches substantially reduce the number of cycles to failure at a given stress level. In order to assess their effects notches of specific dimensions, i.e. with well known elastic stress concentrating ability, are machined into the samples and the fatigue properties are evaluated.

The effects of notches are characterised by the following factors:-

(1) The stress concentration factor, K_t , which is the ratio of the actual stress at the base of the notch, calculated from elastic theory, to the nominal applied stress.

(2) The fatigue stress reduction factor, K_f , which is the ratio of the fatigue strength of the unnotched to the notched fatigue samples.

(3) The notch sensitivity factor, q , which is defined from K_t and K_f .

$$q = \frac{(K_f - 1)}{(K_t - 1)}$$

Thus in principle when $q = 0$ surface notches have no effect and when $q = 1$, notches have their theoretical effect.

An alternative means of characterising the notch effect is to plot K_f/K_t versus K_t , together with curves representing $K_f = K_t$ and $K_f = 1$.

1.60. THE FATIGUE PROCESS.

During the fatigue process several stages are observed. Fatigue hardening and the development of extremely fine slip lines on the sample occur at first. Gradually the slip lines become deeper and longer and fatigue cracks nucleate. Intrusions and extrusions of the metal occur at this stage. The fatigue crack once initiated propagates parallel to the slip planes on which the critically resolved shear stress is highest. In the later stages the fatigue crack propagates at right angles to the principle stress. Eventually the crack attains such a size that it opens and closes during each reversal, allowing the crack tip to move across grains and grain boundaries. The cracks progress until the weakened material fractures by tensile tearing.

Two distinct areas can frequently be observed in a fatigue fracture, an area, often conchoidal in appearance where the fatigue crack has advanced in a stepwise manner and an area typical of a ductile fracture. Such fractures are commonly observed in wrought materials but generally not in the fatigue fracture of sintered materials.

LITERATURE SURVEY

2.00. LITERATURE SURVEY.

2.10. THE ROLL COMPACTION OF METAL POWDERS.

2.11. COMPACTION SYSTEMS.

a) Saturated Feed Systems.

Most of the proposed systems are of this type¹ (Fig.4)
The rolls are arranged horizontally and the stock passes through vertically.

Three zones may be identified with the process:-

- 1) The free zone where the powder is loosely packed and falls under its own weight.
- 2) The feed zone where the powder, although still non-coherent is being pulled into the compaction zone by roll action.
- 3) The compaction zone where the powder becomes coherent and is densified.

The angle that the feed zone subtends at the centre of the rolls is defined as θ_f , the feed angle, and the angle that the compaction zone subtends is defined as θ_c , the compaction angle.

If R is the roll radius, D_i the initial density, D_f the final density and h is the strip thickness, then assuming that the strip does not spread either longitudinally or transversally it may be shown that:-

$$D_f = D_i \left(1 - \frac{R\theta_c^2}{h} \right)$$

Thus the final density and hence mechanical strength of the green strip, since the two are inter-related, depends on the roll radius and angle of compaction. Because the angle of compaction factor is squared a small change in the angle of

compaction has a large effect on the final density. The angle of compaction depends upon roll radius, roll gap, roll surface finish, roll speed and powder type.

i) Powder Variables.

As in conventional compaction irregular, easily compressable, porous powders of a specific particle size distribution containing a large amount of fines produce stronger and denser green strip. However the flow properties of such powders is poor, consequently the rate at which material is conveyed to the roll gap is restricted. Therefore although such powders give satisfactory strip at low rolling speeds, strip quality is impaired at higher rolling speeds because insufficient material is fed to the compaction zone. 7. 8. 14.

It has been shown that the rate at which air can escape from the strip is important because air excluded from the compaction and feed zones travels upwards and impedes the flow of metal powder flowing into the compaction zone. The thickness of strip rolled from carbonyl nickel may be increased by 50 per cent if a hydrogen atmosphere instead of air is used around the rolling mill, due to the viscosity of hydrogen being much less than the viscosity of air.⁸.

Figure 5.¹⁴ shows the variation in flow rate with increasing roll speed for M.H.40, M.H.100 and M.H.300 iron powders. It is apparent that up to a critical roll speed the plot is linear but beyond this there is a departure from linearity. The critical speed at which this occurs is called the flow transition speed

and it may be seen that coarse powders which flow more easily have a higher flow transition speed than finer powders. Variation in roll gap has little effect upon this speed but at wider roll gaps the departure from linearity is greater because of the larger volume of powder being rolled and the greater amount of air that must be expelled from the powder.

Strip density is reduced as soon as the flow transition speed is exceeded. (Fig.6) Thus although M.H.300 produces a denser strip when rolled at slow speeds any commercially viable rolling speeds favours the better flowing M.H.100. It is also apparent that increasing the roll gap reduces the green strip density.

Particle shape affects the compaction angle. Irregular particles tend to interlock thereby increasing the compaction and feed angles. Thus at rolling speeds below the flow transition speeds irregular particles produce denser and stronger strip. Evans and Smith⁷ showed that irregular electrolytic copper powder can be rolled with a compaction angle of 6° which is reduced to $1\frac{1}{2}^{\circ}$ for spherical gas atomised powders. Also it has not been found possible to roll spherical stainless steel powder particles successfully using a saturated feed system.¹⁵ However some workers^{6,16} indicate that powder characteristics have little effect upon the compaction and feed angle which is approximately 7° for most materials. With increasing roll speed, roll load tends to decrease¹⁴ which may be due to a reduction in the feed angle and compaction angle.

Increasing roll speed when rolling M.H.100 iron powder causes a reduction in tensile strength¹⁴ for samples of similar

density.(Fig.7) This behaviour is also typical of J.J.M.300, M.H.30 and M.H.40 powders. However for electrolytic powder of 100 mesh, tensile strength and density show the same values irrespective of roll speed changes. Tundermann and Singer¹⁴ suggest that this behaviour may be due to the smoothness and acicularity of electrolytic powder so that particle movement and subsequent oxide film rupture may occur readily over a wider range of rolling speeds. However particle movement may not occur so readily, with irregular particles and so as rolling speed increases fewer oxide films are broken and strength diminishes.

ii) Roll Diameter.^{10.6.}

Increasing the roll diameter increases the radius of curvature at the roll gap, consequently increasing the compaction angle. Therefore either denser or thicker strip may be rolled.Fig.8.

iii) Roll Roughness.^{7.}

The compaction angle may be increased up to 100 per cent by using rough rolls but during rolling the rolls attain a smoother satin finish because of abrasion. Therefore in production it would be difficult to maintain a standard roughness and this parameter would be of little use as a means of improving strip properties.

iv) Powder Head.^{8.}

Increasing the powder head forces more powder into the roll gap. Figure 9 shows how density may be increased by extending the powder head when rolling nickel powder, but too large a powder head may cause the strip to split.

v) Temperature.^{17.}

Increasing rolling temperature by approximately 300°C has been shown to increase the thickness of copper strip by 25 per cent. This may be due to a reduction in the viscosity of air escaping from the compaction zone. However final mechanical properties may be affected by oxidation of the powder.

vi) Control of Thickness.^{1.}

Thickness control may be carried out by the fitting of shoes to the roll surface. (Fig.10) Selection of the correct type of shoe effectively alters the feed angle and so reduces the amount of powder flowing into the roll gap. Moreover the fitting of shoes may give more precise control of density and thickness than altering the roll gap.

vii) Density and Thickness Variation.^{1.}

Density and thickness variation may occur across the width of strip which has been rolled by a simple saturated feed system. At low rolling speeds density at the strip edge may be less than the density at the strip centre due to the tendency of the powder to flow sideways. At higher rolling speeds escape of air from the centre of the compaction zone to the centre of the feed zone causes a restriction of powder flow at the centre and a corresponding reduction in density. Figure 11 shows how density variations may be controlled by either the use of "shaded shoes" or a metering device.

viii) Work Hardening of Powder Particles.

Little work hardening has been found to occur.

Matsumura^{18.} estimated the increase in hardness to be less than

15 per cent.. Evans and Smith⁷ showed that a significant hardness increase only occurs with large rolling loads. Tundermann and Singer¹⁴, using iron powder, concluded that although work hardening of powder asperites occurs there is little work hardening within the powder particle itself. Thus they were able to reroll the green strip to a density of 7.1 g/c.c. without significant edge cracking.

b) Unsaturated Feed Systems.

An unsaturated feed system is a system which operates using a controlled flow of powder which is less than the maximum flow that occurs by powder flowing into the roll gap under free flow conditions. Such systems may be arranged horizontally or vertically and may incorporate devices to force or meter powder into the roll gap or to bind the powder particles together so that the first densification is of coherent strip. An example of the latter is the B.I.S.R.A. thin strip system.

i) The B.I.S.R.A. Thin Strip Process.² (Fig.12)

This process uses rollers in a vertical plane with the stock passing through horizontally. The process differs from other roll compaction systems in that the powder is mixed with a binder and coherent strip is produced before rolling. Consequently the quality variations associated with rolling speed and the flow properties of powders are largely avoided and extremely high line speeds may be attained. Moreover as strip may be cast very near the finished size the process is very suitable for rolling thin strip.

Seven operations are required:-

- 1) Mixing iron powder to form slurry.
- 2) Coating a continuous belt with slurry, subsequent drying and removal from the belt.
- 3) Roll compacting the bonded powder to strip.
- 4) Sintering in a reducing atmosphere.
- 5) Rerolling the sintered strip.
- 6) Resintering.
- 7) Finish planish or temper rolling.

The process can handle most kinds of iron powder and is very versatile. Because the aim is to manufacture thin strip finer powder grades are more suitable as such powders allow an optimum number of particles in the thickness direction of the strip. Moreover as the process manufactures strip of gauge sizes where there is a disproportionate increase in conventional strip cost, the technique may be economically competitive with conventional strip, particularly for more expensive materials such as stainless steel.

Generally, relatively porous fine pure soft powders have been found to give green strip of optimum properties because of their ease of compactability. Mild steel strip from powder shows similar properties to conventionally rolled, temper 3 tinplate. However specific iron powders, e.g. Makin 300 Pl. may give lower tensile strengths and poorer elongation values. (Fig.13) Stainless strip shows slightly higher tensile strength but poorer elongation values. (25 per cent compared to 40 per cent on a $\frac{3}{4}$ " gauge length). (Fig. 14).

2.12. SINTERING AND REROLLING.

In order to compete with conventional rolling systems a powder rolling plant must have a similar output. Consequently strip finishing speeds must be within the range of several hundred feet per minute to more than 1000 feet a minute for small gauge strip. Because the process must be continuous sintering times must be short if a sintering furnace of practical length is to be used. Little work has been done to assess the feasibility of looping the strip around rollers in the sintering furnace but B.I.S.R.A. has indicated that this practice mars strip finish and also complicates the initial "threading up" procedure.^{15.}

Therefore sintering times must be confined to a maximum of five minutes and because the time is so short, mechanical properties are not equal to those of conventionally rolled strip. Strip rolled by the B.I.S.R.A. slurry process shows a tensile strength of the order of 155N/mm^2 (10 t.s.i.) and an elongation of 1 per cent after the first sinter. J. Oakley of Manganese Bronze showed that sintering copper strip for two minutes in cracked ammonia developed a tensile strength of approximately one third of that of cast copper.

Because sintering times are so short a high sintering temperature which promotes fast sintering rates and rapid attainment of the temperature at which sintering commences, must be used. Consequently sintering temperatures of the order of 1200°C for steel and 1000°C for copper are required. The presence of sulphur severely impairs the mechanical properties of nickel.^{10.} A reduction in the sulphur content of nickel from

0.0038 per cent to 0.0002 per cent improves the tensile strength from approximately 250N/mm^2 (16 t.s.i.) to 365N/mm^2 (23.6 t.s.i.) and the elongation from 10 per cent to 45 per cent. In order to reduce the sulphur content of nickel strip to acceptable levels a sintering time of twenty minutes is needed.

Many workers have shown that hydrogen atmospheres promote faster sintering rates compared to inert or hydrogen/nitrogen atmospheres. Consequently pure atmospheres promote faster sintering rates of strip. Moreover in the event of hydrogen becoming trapped in any sealed porosity during the first sintering operation, no blistering will occur during subsequent rerolling and sintering because of the ease with which hydrogen may diffuse from the strip.

To manufacture strip of near theoretical density and hence of mechanical properties similar to conventional strip, a second rolling and sintering operation is required.

Rerolling mild steel and copper strip followed by resintering realised mechanical properties that were very similar to those of conventional strip.^{2,7} The severity of reduction of stainless steel strip originally rolled by the B.I.S.R.A. saturated feed system was dependent upon sintering temperature. Stainless steel strip sintered to 84 per cent theoretical density at 1200°C ¹ required 70 per cent further cold deformation followed by annealing at 1000°C to realise optimum properties but only 40 per cent reduction was required after sintering at 1400°C . Also higher elongation figures were obtained by using the higher sintering temperature. (Fig.15).

Little information is available concerning the effect of the final sintering temperature. Information for stainless steel¹ indicates that the final sintering temperature is not critical. Sintering at temperatures between 1150°C and 1350°C caused a hardness reduction of 20 V.P.N. from 180 V.P.N. in one to one and a half minutes. Longer sintering times of up to eight minutes at the higher temperatures cited may cause a hardness increase up to 165 V.P.N. due to the appearance of ferrite in the austenite matrix.

2.13. HOT COMPACTION PROCESSES.

Hot compaction processes enable strip to be rolled to almost 100 per cent theoretical density. Moreover the difficulties of attaining satisfactory mechanical properties after a short sinter are avoided.

The Reynolds Process¹¹ (Fig.16) manufactures aluminium and aluminium alloy strip from atomized powder. The powder is atomized from the melt and is transferred to a hopper above the rolls of a saturated feed system. The powder is preheated as it falls towards the rolls where it is fully densified to coherent strip.

The optimum powder particle size was found to be between -8 and + 60 mesh, the particle shapes being fat cigar shaped particles of apparent density of the order of 1 g/c.c. Particles either coarser or finer than this produced thinner strip. Large compaction angles of the order of 15° were found, possibly because of interparticle welding at the bottom of the hopper and the large

powder head. Strip of theoretical density could be manufactured over a range of rolling speeds from 15 metres per minute (50 f.p.m.) to 60 metres (200 f.p.m.).

The Reynolds Process has been used to manufacture most commercial series of aluminium alloys. The mechanical properties of such strip were as least as good as those of conventionally rolled strip. Provided that the amount of further cold deformation exceeded 5 per cent the properties of the compacted sheet were superior to those of conventional sheet. This may be due to a dispersion hardening effect caused by finely dispersed alumina particles.

Hot rolling green copper strip has been carried out by Shaw and Knoppe²⁰ in the United States. Copper powder was compacted at 1.2 metres per minute (4 feet/minute) to 1.52 mms. (0.060"), heated to 1040/50°C for two to two and a half minutes and then hot rolled to 1.38 mms. (0.035"). Further cold rolling to 1.18 mms. (0.030") and annealing in "exogas" for one hour at 550°C resulted in strip of tensile strength 234N/mm² (15.2 t.s.i.) and 46.8% elongation. This compares with 204N/mm² (13 t.s.i.) for conventionally rolled material. Further cold rolling and annealing to 0.14 mms. (0.000 54") increased the tensile strength to 242N/mm² (15.7 t.s.i.) and reduced the elongation to 23.3%. The reduction in elongation is typical of copper strip when it is rolled to such a small gauge.

Rolling the same copper powder by a number of cold rolling and sintering steps to 0.13 mms. (0.0005") gave a tensile strength of 260N/mm^2 (17 t.s.i.) and 26% elongation. The electrical conductivity of the hot rolled strip was 84.8% of the conductivity of oxygen free high conductivity copper. Subsequent hot rolling of the cold rolled and sintered strip increased the conductivity to 98% due to spherodisation of grain boundary, oxide films.

Such a procedure is unlikely to be adopted ^{with iron} because of the greater tendency of iron powder to oxidise and the difficulty of spherodising iron oxide.

2.14. ANISOTROPY OF STRIP FROM POWDER.

Little work has been done in this field, however it would appear that the properties of compacted strip in the transverse direction are better or equal to the properties in the longitudinal direction.^{7.11.}

2.20 THE FATIGUE OF SINTERED PARTS.

The fatigue testing and representation of the results of sintered materials are carried out in the same manner as conventional materials.

Although there are minor differences, sintered samples give a similar form of S/N curve when compared to conventional materials of the same chemical composition.²¹⁻²³. Consequently sintered iron still shows a fatigue limit and materials such as sintered copper do not. (Fig.17)²¹.

Sintered materials generally fail earlier than conventional materials of similar composition during fatigue testing. This feature is mainly due to the presence of porosity which reduces the cross-sectional area upon which the cycling stress acts. Frequently the observed reduction in specimen life is greater than may be calculated by only considering the reduction in cross-sectional area because of the stress raising effect of pores.

The fatigue limits of some materials however, e.g.iron,²² appears to be negligently affected by the pore stress raising effect, because when the results are corrected for the reduction in cross-sectional area they are very similar to wrought iron.

At stresses above the fatigue or endurance limits the pores of sintered materials exert a stress raising effect which increases as the amount of porosity present in the material increases. Consequently the slope of the S/N curve for sintered materials at these stress levels is less than the slope determined

for comparable wrought materials.²¹⁻²⁴ The stress raising effect of pores is small because for small amounts of porosity there is little difference between the S/N curves for conventional and sintered materials.^{22,24}

The number of cycles at which the S/N curve becomes or tends to become horizontal is greater for sintered materials.^{23,25} Consequently sintered plain carbon steels show a fatigue limit after $10^6 - 10^8$ reversals but for wrought plain carbon steels it is shown after $10^5 - 10^7$ reversals.

The fatigue ratio of sintered materials is generally between 0.3 and 0.5.²⁶ Kravic²⁷ and Radomysel'sky²⁸ have reported that the fatigue ratio for sintered steel increases as the tensile strength increases (Fig.18) but other investigators including Goetzel and Seelig²¹ report that the fatigue ratio decreases as the porosity increases.(Fig.18) The reduction in the fatigue ratio with increasing porosity is greater for iron than copper.

Interpretation of the tensile and fatigue data presented graphically by Dunmore and Smith²⁴ for hydrogen sintered copper compacts prepared from atomized copper powder indicated that the fatigue ratio increased from 0.42 to 0.45 finally falling to approximately 0.36 as the tensile strength increased. The reasons for the contradictory statements concerning the behaviour of the fatigue ratio is not clear but the scatter inherent in fatigue testing results may be a contributory factor.

A number of investigators^{23,25,29} have reported that the scatter in the fatigue results for sintered specimens is less than that

normally found in conventional materials. It is probable that fatigue nucleation will begin at the surface of sintered samples. Therefore the presence of porosity at the surface of the material will possibly act as a stress raiser for fatigue nucleation. Consequently the samples of a specific fatigue series should have very similar surface discontinuities from which fatigue cracking will begin.

It is likely that the overriding effect upon fatigue properties is total porosity, however pore size²¹ and to a much lesser extent pore shape exert some influence. Other structural factors, e.g. grain size²⁴ may also affect the fatigue properties. Therefore parts made from fine powders which yield fine well dispersed porosity tend to have better fatigue properties.²¹

2.21. COMPACTION PRESSURE.

Increasing compaction pressure leads to a reduction in total porosity and consequently an improvement in mechanical properties. Moreover Wheatley and Smith²² have reported that increasing the compaction pressure gives finer, more uniform porosity. This is also consistent with the work of Bockstiegel³⁰ who states that as the compaction pressure is increased the largest pores are reduced in size first.

The powder type also affects densification during compaction and so atomised copper powders compact to higher densities than electrolytic copper powders at the same compaction pressure.²⁴

2.22. SINTERING TEMPERATURE.

High sintering temperatures promote densification and pore spherodisation, however excessive sintering temperatures

may lead to grain growth and a corresponding deterioration in fatigue properties.²⁴

Dunmore and Smith²⁴ have shown that the tensile strength of copper compacts pressed at 465 N/mm^2 (30 t.s.i.) and sintered for four hours in nitrogen remains relatively unchanged until a sintering temperature of 300°C is exceeded. Between 300°C and 600°C there was a large improvement in the tensile strength from approximately 13.8 N/mm^2 (0.9 t.s.i.) to 224 N/mm^2 (14.5 t.s.i.). Sintering temperatures greater than 600°C led to a reduction in tensile strength because of grain growth. Despite these changes in tensile strength the endurance limit remained fairly constant for sintering temperatures from 450°C to slightly in excess of 1000°C . Therefore from 450°C to 600°C there was a marked reduction in the value of the fatigue ratio but a gradual increase with an increase in sintering temperature from 600°C to 1000°C . Hardness and elongation values followed similar trends to that of tensile strength. There was little change in the porosity for sintering temperatures of 450°C and above but at the higher temperatures the pores became more spherodised.

2.23. SINTERING TIME.

Generally there is little change in mechanical properties for sintering times from a few minutes to several hours. Copper compacts²⁴ showed a small increase in tensile strength from approximately 190 N/mm^2 (12.3 t.s.i.) to 220 N/mm^2 (14.3 t.s.i.) for an increased sintering time from a few minutes to several hours. The endurance limit remained reasonably constant and there was a slight decrease in hardness during this time, however elongation

values increased from approximately 11 per cent to 23 per cent. This was probably due to individual pores becoming more rounded.

2.24. OTHER FACTORS AFFECTING FATIGUE PROPERTIES.

Although compaction pressure, sintering time and sintering temperature are the most important variables which affect fatigue properties, mainly because of their effect upon total porosity, other factors have a small but significant effect.

a) Powder Type.

Specimens prepared from atomized copper powder show slightly higher endurance limits than samples from electrolytic copper powder of similar density.²⁴

Goetzel and Seelig²⁴ demonstrated that both iron and copper specimens prepared from -325 mesh powders gave better fatigue properties than samples prepared from +200 mesh powders. However Kothari²⁹ reported that a particle size of -250 +325 mesh iron powder gave optimum fatigue properties for iron/10% bronze specimens.

b) Sintering Atmosphere.

Specimens sintered in a hydrogen atmosphere densify more quickly than specimens sintered in other atmospheres.^{24,31.} Moreover hydrogen sintered copper compacts have better fatigue properties than nitrogen sintered compacts.²⁴

Wiest³² noted that the tensile and elongation values of sintered iron were enhanced by sintering in hydrogen especially for sample of high density although the effect on more porous samples was minimal. However the fatigue properties were

relatively unaffected. Some workers, e.g. F.N.Rhines³¹ have shown that sintering in a vacuum develops similar structure and properties to sintering in nitrogen.

The more rapid densification of samples sintered in hydrogen may be due to the reduction of metal oxide at the surfaces of metal particles thus enabling clean metal to metal contact to occur. Moreover, especially at low porosities, hydrogen may diffuse quickly from sealed porosity allowing further pore shrinkage to occur.

2.25. COINING.

Because of work hardening, coining without a further sintering or annealing stage increases the endurance or fatigue limit but lowers the fatigue ratio.³³ When coining is followed by a sintering or annealing step the mechanical properties are similar to the properties of samples of equivalent density processed by a single pressing and sintering operation.

2.26. NUCLEATION OF THE FATIGUE CRACK IN SINTERED PARTS.

It is well established that fatigue cracks nucleate at the surface of solid materials. Nucleation frequently occurs at small surface discontinuities which act as stress raisers. Wheatley and Smith²² found no evidence of internal crack nucleation from the surface of pores but because of the large area of internal porosity the possibility of internal crack nucleation cannot be ignored.

In order to determine the location of fatigue nucleation Wheatley and Smith tested three samples of sintered iron,

density 6.67 g/c.c. These samples would have been expected to fail after 100,000 cycles. However the testing of specimens was interrupted every 50,000 reversals and 0.05 mms.(0.002 inches) of surface was removed.

Fatigue lives of 120,000, 195,000 and 225,000 cycles were obtained. Although each sample survived for a greater time than 100,000 cycles the results are not conclusive because of the scatter inherent in fatigue results. However the results indicate that surface nucleation of fatigue is likely.

Peterson²⁵ evaluated the stress concentration factor for a spherical cavity to be of the order of 2. However for a hole in a flat strip he calculated from elastic theory that the stress concentration factor was 3. If it is assumed that a pore connected to the surface of a sample is more analogous to a hole in a flat bar than a completely enclosed spherical cavity, then fatigue crack nucleation would be more likely to start at the surface of a sample.

Pohl³⁶ related the internal notch effect of pores in sintered ferrous materials by the following equation:-

$$m = \frac{1 - \frac{E}{E_0}}{1 - \frac{D}{D_0}}$$

where E = Young's modulus of sintered material

E₀ = Young's modulus of solid iron

D = density of sintered material

D₀ = density of solid iron

This equation given an average value for the internal notch effect (m) of 2.5 which is similar to that derived by Peterson³⁵. Values for the fatigue strength reduction factor (K_t) were determined empirically by comparing the fatigue strength of sintered steels with that of conventional steels of the same structure. The internal notch effect (m) is equal to the theoretical stress concentration factor in spheroidal graphite cast iron.³⁹ However it has not been shown empirically to be equal to the stress concentration factor for pores in a sintered material. Therefore by assuming that the internal notch effect is equivalent to the theoretical concentration factor then values for the notch sensitivity factor q for ferritic, pearlitic and heat treated sintered steels may be calculated. These are 0.3, 0.45 and 0.8 respectively.

Once a fatigue crack has nucleated it is generally agreed that the stress concentration factor for a fatigue crack is greater than a pore, thus the cracks propagate until failure occurs.

2.27. THE NOTCH SENSITIVITY OF SINTERED MATERIALS.

The presence of external notches reduces the fatigue strength of sintered specimens.^{27,37,38.}

Pasquine³⁴ has reported fatigue data for steels containing 0.48% carbon and varying amounts of nickel. The notch sensitivity factor q was shown to be between 0.36 and 0.57 for a stress concentration factor of 2.2 and for specimens in the density range of 6.6 to 7.2 g/c.c. An isolated value of 0.76 was obtained for a quenched and tempered steel containing 7%

nickel of density 6.6 g/c.c. No relationship appeared to exist between porosity and notch sensitivity.

Similar data has been reported by Kravic,²⁷ Morita et al.³⁵ and Crocks et al.³⁶

2.28. THE EFFECT OF ALLOYING ELEMENTS AND HEAT TREATMENT.

Additions of copper or bronze (1 - 25%) are frequently made to iron powders. Subsequent sintering above the melting point of the second phase enables densification and a satisfactory pore structure to be obtained more rapidly.

Kothari²⁹ has shown that additions of up to 11% bronze to iron powder improved the fatigue limit from 152N/mm² (9.8 t.s.i.) to 200N/mm² (13 t.s.i.) but further additions of bronze up to 20% reduced the fatigue limit to 131N/mm² (8.5 t.s.i.).

Graphite additions of 1% reduce the fatigue limit for samples of 10% porosity but when large amounts of porosity are present (20 - 30%), similar graphite additions have little effect.²⁸

Additions of carbon from 0 to 0.8% increased the fatigue limit of a steel containing 2% nickel from 83N/mm² (5.4 t.s.i.) to 150 N/mm² (9.7 t.s.i.) Figure 19.³⁸

Alloying additions are also made to sintered iron to promote hardenability. Kravic and Pasquine³⁴ studied the heat treatment of a 0.48% carbon with a tensile strength of 1015N/mm² (65.7 t.s.i.) were reported for samples of 7.2 g/c.c. density.

The properties of quenched and tempered, sintered steels are similar to wrought steels of the same composition. However sintered steels may become brittle with high carbon contents. Therefore nickel must be added and the level of carbon controlled

in order to give acceptable properties.

It has been inferred²⁶ from the data of Raymond³³ that the hardenability of sintered steels is not as good as conventional steels. This may possibly be due to the finer grain size of sintered steels, the much greater free surface area and the poorer thermal conductivity.

2.29. A COMPARISON BETWEEN SINTERED AND OTHER MATERIALS.

Fatigue tests conducted under laboratory conditions show that the properties of sintered materials are not as good as those of wrought materials of similar chemical composition. In order to approach the properties of conventional materials the porosity of sintered materials must be less than 5%.¹³ Such high densities are rarely attained commercially. Consequently the properties of commercially sintered material appear to be not competitive with conventional wrought materials.

However in specific instances sintered materials may show superior properties to cast materials, e.g. copper.²⁴ Moreover the additions of small percentages of copper or bronze to iron powders, so that sintering occurs in the presence of a liquid phase significantly improves fatigue properties. Therefore the properties of sintered copper carbon steels are comparable with many nodular cast irons.²⁶ Figure 20 shows a comparison between the endurance limits of sintered materials with those of other materials.²⁶

The fatigue ratios of sintered steels are between 0.3 and 0.5, clustering about 0.4, whilst wrought steels are

of the order of 0.5. Sintered parts are less notch sensitive than wrought parts. Wrought steels show a value for the notch sensitivity factor q of 0.5 whereas sintered steels and nodular cast irons³⁹ show a value of 0.35. Although precise values for the notch sensitivity factor are not especially significant they are an indication that sintered materials are less notch sensitive than comparable wrought materials. This is possibly due to porosity at the surface of the sample which acts as a mild stress raiser. Consequently the stress concentration at an external notch is less than the stress concentration at a notch in a more uniform wrought material.

Raymond³³ has suggested that superior fatigue data is obtained on sintered unmachined specimens. While there is no doubt that fatigue results obtained on sintered parts give a much better indication of the service life of a component, the study of machined fatigue samples avoids the added complication of service testing. However it has been observed that sintered compacts show more porosity in the corners and peripheral areas. If this porosity acts as a mild stress raiser then the fatigue properties of unmachined samples may be slightly worse than machined samples of the same shape.

Although the fatigue properties of sintered specimens are generally not as good as conventional materials of the same chemical composition, alloying additions to the sintered material improve the fatigue properties. Therefore sintered components are increasingly replacing conventional components in situations where cycling stresses are present, e.g. rocker arm brackets.

SCOPE OF PRESENT WORK

3.00 SCOPE OF PRESENT WORK.

3.10. THE ROLL COMPACTION OF IRON POWDER.

The present work concerns the B.I.S.R.A. slurry process for the manufacture of thin strip. The aim of the work is to obtain a more thorough understanding of the factors which affect the rolling and sintering of iron powder by the B.I.S.R.A. slurry process. Thus such information may be useful in order to improve the quality of the strip or to reduce the rigorousness of the conditions which are currently required to manufacture sound strip. In order to fulfil this aim the work was divided into several stages.

3.10. Stage 1.

In the first stage a number of iron powders were carefully characterised and prepared. The particle parameters which were investigated are:-

- 1) Particle size.
- 2) Particle size distribution.
- 3) Surface area.
- 4) Particle porosity.
- 5) Particle deformability.
- 6) Particle shape.
- 7) Surface purity of the powder particles.

3.20. Stage 2.

In the second stage, the effect of the above parameters upon the properties of green strip prepared by the B.I.S.R.A.

slurry process was assessed. The following variables were thought to influence strip quality:-

- i) Slurry composition.
- ii) Roll speed.
- iii) Roll load.

i) Slurry Composition.

No attempt was made to investigate the role of slurry composition upon the quality of the green strip because the optimum slurry composition had already been established by B.I.S.R.A. Staff.⁴⁰ This slurry was adopted for the present work.

ii) Roll Speed.

A standard roll speed was used throughout the investigation because it had been reported that there was little change in quality for variations in roll speed from 0.3 metres to 150metres/minutes (1 to 500 f.p.m.) and because it was difficult to roll short samples over a wide range of rolling speed.

iii) Roll Load.

The effect of roll load on samples of "flexistrip", i.e. strip composed of iron powder and binder that had been cast on to a former, dried and removed, was determined over a wide range of roll loads.

3.30. Stage 3.

In the third stage sintering conditions and further processing of strip rolled in Stage 2 were investigated. The variables believed to affect properties were:-

- i) Sintering temperature.
- ii) Sintering time.

Variables affecting further processing would be:-

- i) Load during second rolling operation.
- ii) Sintering temperature of the second sinter.
- iii) Sintering time of the second sinter.

i) Sintering Temperature.

Although it is well known that changes in sintering temperature markedly affect densification, a standard sintering temperature was used throughout the work. The sintering temperature was the highest that is easily attained commercially because densification is faster the higher the temperature and the shorter is the time taken by samples to attain the temperature at which sintering commences.

ii) Sintering Time.

Although samples were sintered for a number of sintering times for the first sinter a standard time was chosen for the second sinter owing to the shortage of time and the difficulty of preparing a sufficient number of samples.

3.40 THE FATIGUE OF SINTERED NICKEL.

The effect of total porosity, pore size and pore shape upon the properties of sintered nickel prepared from Sheritt Gordon, Grade S nickel powder was studied.

The work included a brief examination of the compaction and sintering characteristics of nickel powder.

Specific fractions of the powder were prepared by three different techniques in an attempt to give three series of different total porosity of varying pore size.

THE ROLL COMPACTION OF IRON POWDERS

MATERIALS AND EXPERIMENTAL PROCEDURE

4.00. MATERIALS & EXPERIMENTAL PROCEDURE.

4.10. MATERIALS

The iron powders used were as follows:-

- 1) Makin powder, J.J.M. 300. (-300 mesh)
- 2) Sintrex electrolytic powder (-300 mesh)
- 3) Woodall Duckam powder (-100 mesh)
- 4) B.S.A. water atomized powder (-100 mesh)
- 5) B.S.A. water atomized powder (-300 mesh)

From the J.J.M. 300 powder two air elutriated fractions were prepared:-

- 6) A powder which was of a particle size distribution predominantly greater than 30 microns in size.
- 7) A powder which was of a particle size distribution predominantly less than 30 microns in size.
- 8) Woodall Duckam powder (-300 mesh)

4.11. PREPARATION OF THE ELUTRIATED FRACTION.

An elutriation column shown schematically in fig.21 was constructed. The iron powder was fed through a short length of 6.5 mm. ($\frac{1}{4}$ inch) glass tubing by an Archimedian screw into a vertical glass tube 1500 mms.(5 feet) long and 25 mms. (1 inch) in diameter. Compressed air was introduced tangentially into a conical flask at the bottom of the glass tube. Air pressure was controlled by bleeding air from the system through one arm of a 'T' piece on the inlet side of the apparatus. A short length of rubber tubing was fitted to this arm which was clamped by a Mohr clip. Variation in the setting of the Mohr clip altered the amount of air bled from the system. The coarse fraction was collected in the conical flask while the fine fraction was collected by allowing the air and iron powder coming from the top of the column to

expand into a large conical flask and thereafter into another flask in which two permanent magnets were situated.

Each coarse fraction and each fine fraction were elutriated twice. Powder was fed into the apparatus at an approximate rate of 15g per minute but because of the difficulty of operating the electric motor which was driving the screw at a constant speed, this rate was frequently either greater or less than this. Air pressure was adjusted until approximately half of the powder was swept up the column and the remainder dropped down the column.

Because of the difficulties of operating the elutriation apparatus under consistent conditions all of the powder that was used for strip preparation was prepared as a single batch beforehand. Each of the sub-batches that were collected from the column were blended and mixed in a laboratory ball mill using light ceramic balls for one hour.

A -300 mesh fraction of Woodall Duckam powder was obtained by sieving the -100 mesh fraction for one hour using an Endecott Sieve Shaker.

4.20. POWDER CHARACTERISATION.

4.21. SAMPLING.

A single batch of each powder was used throughout the investigation. Each powder was sampled by a "quartering and coning" technique until a sample size of approximately 4 Kgs. was attained. This was further broken down using a "Knight Splitter". This apparatus fed the powder down a rotating chute into a number of containers arranged in a circle around the chute. The desired sample was obtained by blending adjacent containers. Samples of approximately 100g. were obtained by the "Knight Splitter". Smaller samples if needed were obtained by further "quartering and coning".

4.22. PARTICLE APPEARANCE.

The powder particles were examined at low magnification with a binocular microscope and also at considerably higher magnifications using a Stereoscan Electron Microscope.

4.23. PARTICLE MICROSTRUCTURE.

A small amount of each powder was mounted in acrylic plastic and ground using silicon carbide papers, final polishing was carried out using diamond paste in the conventional manner. The specimens were etched in nital.

4.24. PARTICLE SIZE DETERMINATION.

(a) Sieve Analyses.

Sieve analyses for the powders concerned were obtained by sieving 100g of powder using an Endecott Sieve Shaker. The sieves that were used were 200 mesh, 240 mesh, 300 mesh and 350

mesh. The amount of powder remaining upon each sieve after sieving for one hour was carefully weighed.

(b) Sedimentation Techniques.

Particle size analyses were attempted using a Goering Kerr Photosedimentometer for the J.J.M. 300 powder. Various concentrations of glycerol and water were used to attain a satisfactory settling time and to give freedom from agglomeration. A concentration of 60% glycerol and 40% water was found to give an optimum settling time but some agglomeration and air bubble formation still occurred.

The Goering Kerr Photosedimentometer used a large settling container made of carefully ground glass. Because of the cost of such a container the use of ultrasonic frequency vibration to attain satisfactory dispersement of the iron powder was thought to be unwise,

Better results were obtained on J.J.M. 300 powder using a much smaller settling vessel which could be used with ultrasonic frequency vibration and an optical densitometer. A medium of liquid paraffin was found to be most suitable.

(c) Microsieving Techniques.

An ultrasonic microsieving apparatus was constructed for use with Mullard electroformed sieves which is schematically shown in fig. 22. The apparatus was immersed in alcohol so that the alcohol filled the top chamber to the level of the ultrasonic transducer. During sieving a frequency of approximately 20 KHz. was used and the liquid was stirred by a stirrer driven by an electric motor. Sieving was continued for periods of up to 8 hours. After sieving the powder was carefully dried and weighed.

To investigate the reproducibility of the technique Makin J.J.M. 300 powder was sieved for 3, 5 and 8 hour periods using a 31 micron sieve. The -300 mesh fraction of Woodall Duckam powder and Sintrex powder were sieved for 8 hours.

Sieving with a 20 micron sieve was not successful. Sieve blinding occurred despite considerable stirring and the ultrasonic vibration. Moreover the quantity of powder which passed through the 20 micron sieve was small and could not be accurately measured. However it was estimated that approximately 1% by weight of the Sintrex and Makin powders passed through the 20 micron sieve.

(d) Microscopy Measurements.

Quantitative microscopical data for the "as received" powders and the elutriated powders was obtained using a quantitative television microscope (Q.T.M.). The Q.T.M. was programmed to count particles in a number of fields and to group the number of particles counted into specific size ranges. Generally approximately 30 fields were examined and two samples of each powder were prepared. The particles were grouped into size ranges of 10 microns beginning from 50 microns and progressing to less than 10 microns.

Difficulty was experienced in dispersing the iron powder for microscopical examination:-

(i) A viscous solution of polystyrene in xylene and a small amount of iron powder was introduced on to a microscope slide. Using a needle the iron powder was stroked into the solution. Continued stroking broke up powder agglomerates. The solution and iron powder were then covered with a cover glass.

(ii) The powder was added to approximately 25 mls. of a 5% aqueous glycerine solution containing dispersants. The mixture was vibrated ultrasonically and stirred mechanically for approximately one minute. One drop of this solution was then transferred to a microscope slide and covered with a cover slip.

4.25. THE DETERMINATION OF SURFACE AREA.

The methods used to measure total surface area measure both the area of the external surface of the particles and the area of any porosity connected to the surface.

(a) Gas adsorption techniques.

These techniques are based upon B.E.T. theory.⁴¹ If a monomolecular layer of a gas can be adsorbed on to a powder then provided that the mass of the powder used is known and also the molecular size of the adsorbing gas, then the surface area of the particles can be calculated. As gas molecules are small they can penetrate the network of interconnected pores easily so that a value for total surface area is obtained.

Surface area measurements were made by nitrogen adsorption using an apparatus similar to that described by Emmett.⁴¹ However it was found that the sensitivity of the apparatus was insufficient to obtain accurate values for the range of iron powders considered because the samples had small surface areas. This necessitated using large vessels of non ideal shape to contain a large powder sample. Despite large samples being used (of the order of 50g) the amount of nitrogen adsorbed was still small so that errors occurring due to the 'dead space' in the apparatus were exaggerated.

(b) Fatty acid adsorption techniques.

The adsorption of fatty acid molecules on to a surface may be used as a basis for surface area measurements. Many workers including Harkins and Gans,⁴² and Orr and Dalaville⁴³ have used this method to determine total surface areas. The method used was based upon that described by Orr.

A solution containing a known weight of stearic acid dissolved in methyl alcohol was shaken with a known weight of iron powder for one hour. The amount of stearic acid adsorbed by the iron powder was calculated from the difference between the titres of an aliquot of the original solution and an aliquot of the solution after adsorption. Bromo thymol blue was used as an indicator and the methyl alcohol/stearic acid solution was titrated against standard caustic soda.

(c) Surface area measurement by other techniques.

The specific surface of a powder may also be determined by permeametry or by quantitative microscopical techniques. The values recorded by these methods are an indication of the area of the surface of the particles and do not include the surface area of interconnected pores. The specific surface of a powder is an indication of particle shape because irregular particles show a larger specific surface than rounded particles. If the results are compared to total surface area results then an estimation of the area of internal voids can be obtained. Also a value for mean particle size may be made if the number of particles which constitute the specific surface determination is known. This factor is easily obtained using microscopical techniques.

(i) Permeability methods.

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An apparatus similar to the Lea and Nurse permeability apparatus was constructed. This is shown schematically in fig.23. Iron powder was poured into a glass tube 150 mms.(6 inches) long and 25 mms. (1 inch) in diameter. The powder was supported by an aluminium mesh over which a filter paper was placed. Compressed air was passed through the powder bed and the pressure drop across the bed was determined by a manometer. The air subsequently passed through a venturi and the pressure drop across the venturi was determined by a manometer. This pressure drop was related to the rate of airflow by allowing the air to displace known amounts of water from inverted measuring cylinders.

The calculation of surface area from permeametry measurements is complex and a number of assumptions which may not be completely valid must be made. These assumptions may be in error for powders which have a large particle size range. Thus with the iron powders investigated absolute values of specific surface cannot be calculated with certainty. However as the particle size range for most of the powders is similar the results are considered to be comparable.

(ii) Metallographic methods.

Many techniques of quantitative metallography have been reported in the literature over the past forty years, e.g. Quantitative Stereology by C.C. Underwood, Addison Warley, 1970. Quantitative metallographic techniques rely upon counting the sizes of a large number of particles or colonies of phases. This may be done by chord size distributions or point counts. Integration of these values gives the projected

surface area of the particles or phase considered. If the particles or phases are randomly oriented then this value may be directly related to either total surface area or volume, if a sufficiently large number of counts is made. The Q.T.M. can give integrations of chord distributions quickly and so a large number of particles may be evaluated, thus reducing errors due to sampling error and segregation which may occur due to slide preparation.

4.26. PARTICLE POROSITY.

Particle porosity may consist of either closed or open porosity. As most of the powders considered have been prepared by hydrogen reduction the major portion of porosity in the powders is open.

Closed porosity of a powder may be easily found by apparent solid density measurements and by a comparison of these results to the theoretical density of iron.

Open porosity is more difficult to evaluate. Common techniques may be based upon B.E.T. desorption data or results obtained by mercury porosimeter methods. Unfortunately neither a B.E.T. apparatus of sufficient sensitivity nor a mercury porosimeter were available.

The apparent solid density and hence close porosity of the iron powders considered was found by using a specific gravity bottle technique with xylene. Both the xylene and iron powder were degassed before use. Ultrasonic and vacuum techniques were used to disperse any vapour bubbles before the

specific gravity bottle and contents were weighed.

A technique occasionally used in the coal industry for determining the open porosity of coal is to impregnate the coal with water. By following the weight changes with respect to time of the coal/water mixture, an inflexion point may be noted on the curve which corresponds to the time when water has evaporated from the surface of the iron powder but not from the open pores. Thus by comparing the weight of dry coal with this weight of coal plus water an estimate of the amount of open porosity present can be made. This technique was employed with a number of iron powders using chloroform as an impregnating liquid. Weight changes with time were followed by the use of an accurate single pan balance. Unfortunately no reproduceable or meaningful results were obtained.

It should be possible to obtain an idea of the open pore surface area by comparing surface area measurements obtained by either microscopy or the Lea and Nurse method with surface area measurements obtained by adsorption techniques. However only relative results have been obtained by the Lea and Nurse method and the results obtained by quantitative metallography are slightly in error due to unavoidable particle agglomeration upon the microscope slide.

⁴⁵
Yarnton has commented upon a method of determining the microporosity of tungsten powders by the use of various liquids in standard density determinations. By selecting a range of liquids of varying molecular size, a micropore size distribution may be obtained. This method was attempted, but no meaningful results were obtained because the variations obtained by using different liquids were of the same order of magnitude as the experimental error. Some liquids reacted with the iron

powder concerned.

Although meaningful results for open porosity could not be obtained it is believed that this is not too serious an omission as large amounts of open porosity are indicated by measurements of total surface area and metallographic examination.

4.27 PARTICLE DEFORMABILITY.

The ability of a particle to deform under pressure is a function of both the amount of particle porosity present and the ability of the metal lattice to deform plastically. This latter factor depends upon the internal strain in the lattice and also upon the chemical composition of the metal. Particle deformability has been assessed by microhardness techniques and static compaction data. However it is appreciated that other particle parameters e.g. particle size affect compaction data and that microporosity may influence microhardness results.

(i) Compaction data.

The powders studied were compacted in a double acting die with six drops of carbon tetrachloride as lubricant to ease ejection. No die wall lubricant was used. The pressed compacts measured 31.5 mms. ($1\frac{1}{4}$ inches) x 6.4 mms. ($\frac{1}{4}$ inch) x 6.4 mms.

Density measurements were made upon the pressed samples by the mercury displacement method and calculated as is shown in appendix

(ii) Microhardness data.

Microhardness results were obtained using the Vickers' Microhardness Tester. Indentations were made upon etched samples to reduce any error caused by preparation of the sample and to avoid areas of porosity.

4.28. PARTICLE SHAPE.

Powder particles may be rounded, acicular, regular or irregular and many investigators have shown that the shape of a particle may influence compactability.

Shape factors may be calculated in a number of ways combining results obtained by permeametry, microscopical data and total surface area measurements. However it is believed that such data is little better than a qualitative appraisal of shape combined with values of specific surface for this investigation. Therefore a qualitative appraisal of shape was adopted.

4.29. SURFACE PURITY.

Despite the possible importance of surface purity no satisfactory method was developed to measure this parameter.

In an attempt to determine surface purity, several of the powders were examined by reflectance spectrophotometry using wavelengths of light from the far ultraviolet to the near infrared. Unfortunately no distinct peaks were present in a plot of the intensity of the reflected wavelength against frequency of the incident illumination. Thus the method was found to be unsuitable for detecting or measuring surface purity.

4.30. STRIP PREPARATION.

All samples of strip were prepared using the slurry process developed by the British Iron & Steel Research Association (B.I.S.R.A.). Small quantities of slurry were prepared from iron powder mixed with 4% aqueous methyl cellulose, additional water and glycerol. "Supronic", a commercial wetting agent was used to suppress air bubble formation and "Geigy", a commercial inhibitor was used to limit particle rusting before drying. The iron powder was added very gradually to the mixture of methyl cellulose, water and glycerol continuously stirring with a stirrer driven by an electric motor. Slow stirring for approximately three hours was continued to ensure homogenisation before the mixture was used.

Two methods of producing 'flexistrip' were used. Strip from Makin powder, the derived Makin powders and Woodall Duckam powder were prepared by spreading the slurry on to stainless steel troughs 38 mms. ($1\frac{1}{2}$ inches) by either 0.625 mms. or 1.25 mms. (25 'thou' or 50 'thou') deep. The wet slurry was dried for five minutes at 150° C in an oven before being removed from the trough. Strip removal was facilitated by the application of 1% alcoholic oleic acid to the trough before spreading the strip.

Strip from the B.S.A. -100 mesh and B.S.A. -300 mesh powders was made using the pilot plant built by B.I.S.R.A. (fig.24) The thickness of the 'flexistrip' was controlled by altering the height of the gate. Again strip removal was facilitated by coating the continuous stainless steel band with a 1% alcoholic oleic acid solution.

Because of the difficulty of measuring stress during rolling, measurements of roll force were used. Consequently all 'flexistrip' made on the trough was cut into standard strip 28.6 mms. ($1\frac{1}{8}$ inches) wide while all the strip made on the pilot plant was prepared in 102 mms. (4 inches) widths. Preparing samples to standard widths obviated the need for edge trimming.

Rolling was carried out using a two high mill with 300 mm. ($12\frac{1}{2}$ inch) ^{diameter} rolls at a roll speed of 3.65 metres (12 feet) per minute. Roll load was measured by two load cells connected to the top roll.

The samples of strip were fed by hand into the roll gap. Roll loads from 40 kN. (4 tons) to 220 kN. (22 tons) per 25 mm. (1 inch) width were used. The upper limit of this range was determined by the onset of edge cracking. The B.S.A. powders were rolled at a single roll load of 160 kN. (16 tons) per 25 mm. (1 inch) width because of the difficulty of obtaining coherent strip at low roll loads and the restricted range of roll loads, especially when rolling 100 mm. (4 inches) strip.

The green strip was sintered in a stream of purified, dried hydrogen ^{at $1,180^{\circ}\text{C}$} . The gas dried by passing through a column of molecular sieve. Oxygen was then removed by a "De Oxo" catalytic purifier. The resultant water vapour was removed by another column of molecular sieve. A series of sintering times from 10 seconds to 300 seconds were used. Samples approximately 100 mms. (4 inches) long by either 10 mms. (0.4 inches) or 25 mms. (1 inch) were placed on a skeletal stainless steel boat and pushed into the hot zone of the furnace where it was held for the

prescribed time. Measurement of the sintering time was started after the sample had been pushed into the hot zone. A time of five seconds was allowed for the introduction to and extraction from the hot zone of the furnace. Cooling was achieved by pulling the sample into a water cooled part of the refractory furnace tube. The sample was allowed to cool for one minute before the furnace was purged with an inert atmosphere and the sample extracted.

Rerolling of the sintered strip was carried out using the same two high rolling mill. Standard width strips of 28.6 mms. ($1\frac{1}{8}$ inches) were used upon which a 25 mm. gauge length was scribed to establish elongation during rolling. A roll load of 150 kN. (15 tons) per 25 mm. (1 inch) width was used for the second compaction of each sample. This load was the highest roll load that could be used without edge cracking of the strip occurring.

Resintering was carried out in the same apparatus as the first sinter. A standard sintering time of thirty seconds was used for each sample.

4. 40. DENSITY DETERMINATION.

Although it was appreciated that the presence of methyl cellulose would reduce the accuracy of the density measurements, no attempt was made to remove it with the exception of samples prepared for the xylene impregnation method. The estimated weight contributed by the methyl cellulose was calculated to be much less than 1%. The error arising from this is considered to be less than the error arising in the density measurements.

4.41. DETERMINATION OF GREEN STRIP DENSITY.

A number of methods for the determination of density were evaluated:-

(i) Mensuration.

Samples of strip approximately 20 mms. x 20 mms. (0.78 inches x 0.78 inches) were prepared by carefully scoring the strip with a sharp scalpel, breaking and abrading the edges with silicon carbide paper. The sample was clamped between two glass slides for the latter operation in order to ensure a straight edge and to prevent damage to areas in the vicinity of the edges.

Thickness determinations were made by measuring the sample using a sensitive comparator and slip gauges. Unfortunately the spring loaded ball of the comparator indented the strip so that low readings were obtained. On the other hand sandwiching the strip between two slip gauges and measuring gave high readings because the slip gauges positioned themselves on 'high spots' of the material. The length of the edges of the strip was determined by projecting the sample image on to the ground glass screen of a Vickers' Projection Microscope. The dimensions of the image

were measured by calipers. The magnification of the microscope was determined by measuring the projected image of a slip gauge.

Finally by determining the weight of the sample to two decimal places the density could be calculated.

(ii) Immersion in mercury.

Samples similar to the samples prepared for mensuration were used for this technique. Each sample was weighed in air and the force required to immerse it in mercury was measured. The density was calculated as shown in appendix 1.

The method proved insufficiently accurate due to the experimental error inherent with the mercury balance technique and the small upthrust given by the small samples of strip.

(iii) Impregnation with xylene.

If the internal voids of a sample are filled with a known volume of xylene and the density of the sample is evaluated by immersion in xylene, the bulk density of the sample can be calculated.

Samples for this technique were prepared by heating the material to 400°C in hydrogen to eliminate methyl cellulose from the material.

Subsequently each sample was weighed and placed in a vacuum dessicator. Air was evacuated from the dessicator and xylene introduced until the samples were immersed. Subsequent admission of air into the dessicator forced the xylene into the pores of the material.

The samples were removed from the dessicator and their weight when immersed in xylene determined. Finally the xylene

impregnated samples were weighed in air. Excess drops of xylene were removed by a filter paper and weighing was carried out as quickly as possible to minimise errors caused by the evaporation of xylene. The results were calculated as shown in appendix 2.

This technique also proved to be insufficiently accurate because of the small amount of xylene absorbed by the strip and to the rapid volatilization of xylene during weighing.

(iv) Immersion in other fluids.

The densities of conventional materials are frequently determined by weighing samples in air and subsequently immersing the sample in water or other convenient liquid and reweighing. This technique must be altered when applied to porous materials because the internal voids of the material are quickly filled with the liquid media. Many previous investigators have coated the sample with an impervious coating to prevent this.

Generally the most suitable coating agents are organic based and tend to dissolve in organic liquids. Consequently a number of organic coating materials which would be inert during subsequent immersion in water were examined. Silicone fluids, grease and a 5% solution of polystyrene in xylene were tried as coating materials and the latter agent was found to be the most suitable.

Samples were prepared in the same way as the mensuration samples. They were weighed and dipped into the polystyrene solution and allowed to dry. After drying the samples were redipped and put into a warm place to promote the evaporation of xylene. Surplus drops of xylene were removed by a filter paper. The weight of the polystyrene coated samples when immersed in water was subsequently determined.

Although it was found that there was still a tendency for either the coating or the samples to absorb water, if the weighings were made within 30 seconds consistent results were obtained.

This technique was found to be the most satisfactory of those investigated and was used to determine the density of all the samples prepared. Corrections for the weight and the volume of the polystyrene were made in calculating the density values.

4.42. SINTERED DENSITY.

The density of material from the first and second sinters was determined using the same procedure as for green strip.

4.50. RESISTIVITY DETERMINATION.

Standard samples of green strip 10 mms. wide (0.4 inches approximately) were prepared by clamping the strip in a jig and milling on a "Tensilcut" machine. The dimensions of the sample were measured and the resistance of the sample was found using a "Kelvin Bridge".

Apparatus shown schematically in fig.25 was used to determine resistivity. The apparatus was made of "Tufnol" and showed an infinite resistance when a determination was made in the absence of a sample. Current was passed through the strip by screwing the two roughened brass faces of the clamps firmly on to the strip. Potential measurements were made by screwing the two pointed screws into the strip so that indentations were made on the strip.

The resistance of sintered specimens was determined on the same jig. It was found that the samples of green strip prepared above after sintering could be used for such determinations. Sintering was carried out in the manner previously described.

4.60. TENSILE TESTING.

4.61. GREEN STRIP.

It was thought that a standard test piece shape would not be suitable for tensile testing because of the tendency of the strip to fracture outside the gauge length. Consequently samples of the type shown in fig.26 were prepared. The width of the minimum cross-section was approximately 20 mms. (0.78 inches) and the radius of the gauge length was approximately 120 mms. (4.7 inches).

Samples were prepared by cutting blanks slightly oversize with a scalpel. The blank was then placed between two stainless steel templates of the required size. The templates were tightened and the surplus material was removed by silicon carbide papers, finishing with grade 600.

Since a tensile testing machine with a sufficiently low range was not available a testing arrangement using an "Instron" mechanical testing machine fitted with a compression load cell was devised. Testing was carried out at a cross head speed of approximately 1 mm. per minute.

Because of the "lever effect" of the beam (fig.27), recordings made by the "Instron" were not direct readings of force. Consequently the apparatus was calibrated by weights throughout the entire range of force used during testing.

Difficulty was experienced in gripping the sample satisfactorily but the procedure finally adopted was to attach the green tensile sample to flat steel plates with self adhesive tape. Using this arrangement slip of the sample within the self

adhesive tape seldom occurred but because of extension of the tape during testing, elongation as shown by the "Instron" recorder was not representative of the specimen.

4.62. SINTERED STRIP.

Samples were prepared by milling strip clamped between stainless steel templates on a "Tensilcut" machine. A parallel gauge length of 25 mms. with radii of curvature of 50 mms. at the extremities was used. After cutting on the "Tensilcut" machine the edges of the sample were carefully abraded with silicon carbide paper grades 400 and 600.

Subsequently the samples were tested using a "Hounsfield Tensometer".

THE ROLL COMPACTION OF IRON POWDERS

RESULTS

5.00. RESULTS.

5.10. POWDER APPEARANCE.

All powders had a dull grey appearance with the exception of the B.S.A. water atomized powders which showed a reddish/brown colour due to appreciable rusting during manufacture.

At low magnification the J.J.M. 300 and Woodall Duckham Powders were similar in appearance although some of the particles of the Woodall Duckham powder were much larger than the J.J.M. 300 particles. Both powders were composed of rough irregular particles. The Sintrex powder particles were acicular and appeared to be smoother than either the J.J.M. 300 or Woodall Duckham powders.

The water atomized powders were spheroidal and showed a greater portion of fine particles compared to the other powders.

"Stereoscan" photographs (fig.28) showed clearly the rough nature of the surface of the Makin and Woodall Duckham powders. Moreover "craters" at the surface of some of the powder particles indicated the presence of internal porosity. The acicular nature of the Sintrex powder particles was readily apparent (fig.29). However the surface was not as smooth as was indicated by observation at lower magnification. Instead the surface appeared to show many surface folds.

5.20. PARTICLE MICROSTRUCTURE.

The microstructures of the powder are shown in fig.30. The internal porosity of the Makin and Woodall Duckham powder particles was confirmed. Moreover the microstructure indicated that the porosity was mainly interconnecting. The Sintrex powder

particles on the other hand showed very little internal porosity. The structures of all the powders investigated showed very fine inclusions with the exception of Makin particles where slate grey coloured inclusions were evident. These inclusions were believed to be iron oxide. Subsequent microhardness results strengthened this belief. The structure of the water atomized powders was martensitic.

5.30. PARTICLE MICROHARDNESS.

The microhardness of the Makin and Woodall powder particles was very similar (133 Hv) but the Sintrex powder was significantly softer (91.5 Hv). Microhardness results on the slate grey phase observed in the J.J.M. 300 powder particles were 336 Hv which was consistent with the belief that the constituent was iron oxide. The water atomized powders were much harder (B.S.A. -100 mesh 517 Hv., B.S.A. -300 mesh 525 Hv.) due to the formation of martensite during quenching.

5.40. PARTICLE SIZE ANALYSIS.

Results of sieve analyses including microsieve analyses are shown in table 1. Sedimentation data for J.J.M. 300 is given in table 2 and the Q.T.M. results in table 3. Attention should be drawn to the fact that the sieve analysis results and sedimentation results are presented as a percentage weight distribution whereas Q.T.M. data is expressed as a percentage distribution by number.

B.S.A. -300 mesh and J.J.M. 300 powders are seen to be substantially below 350 mesh, however Sintrex and Woodall Duckan powders have a significant fraction between 300 and 350 mesh.

(B.S. sieves). On the other hand Woodall Duckam powder shows a smaller fraction by weight between 31 microns and 350 mesh (of the order of 45 microns).

The effectiveness of air elutriation is shown by the fines of J.J.M. 300 powder having a fraction greater than 30 microns of approximately six per cent by number whereas "as received" J.J.M. 300 shows between approximately twentytwo to twentyfive per cent to be of particle size greater than 30 micron. The particle size distributions of the J.J.M. 300 and Woodall Duckam -300 mesh powders were very similar. However the -300 mesh fraction of B.S.A. powder showed a fraction seventy-five per cent by number finer than 20 microns. Because of the acicular nature of the Sintrex Electrolytic powder a particle size distribution by sieve analysis does not necessarily give an indication of real particle size. However the indicated particle size distribution is similar to that of the "as received"

-300 mesh Woodall Duckam and J.J.M. 300 powders.

Results obtained by the Q.T.M. also include projected surface area data. These results are discussed later. However a measure of mean particle size was calculated from this data. In order to simplify the calculation the assumption was made that all the powder particles were spherical. This assumption gives results that are a close approximation to mean particle size for the chemically reduced powders, which are approximately spherical but is in error for the acicular electrolytic powder. The results are shown in table 4. Comparison of these results with the particle size distribution by Q.T.M. indicated that the

mean particle size by surface area was less than expected.

This was due to the greater contribution made towards surface area by the smaller particles.

5.50. SURFACE AREA RESULTS.

Total surface area results by stearic acid adsorption are given in table 5. It is apparent that the Sintrex Electrolytic powder was of appreciably lower surface area than the chemically reduced powders, probably due to the absence of internal open porosity connected to the surface of the samples. The influence of particle size on surface area is demonstrated by the water atomized powders. Despite very little internal porosity both the -100 mesh fraction and the -300 mesh fraction showed high surface area results ($0.25 \text{ m}^2/\text{g}$) and these results were similar to the results given by the very porous Woodall Duckam powder. B.E.T. surface area data obtained on different batches of the powders examined are given in table 6. The results were of the same order of magnitude as total surface area results obtained by stearic acid adsorption, however J.J.M. 300 showed a higher surface area ($0.31 \text{ m}^2/\text{g}$ compared with $0.13 \text{ m}^2/\text{g}$) as also did the Sintrex Electrolytic powder ($0.14 \text{ m}^2/\text{g}$ B.E.T. compared with $0.074 \text{ m}^2/\text{g}$ stearic acid adsorption). This difference may be attributable to variations between batches of the powders but is more probably due to the powders concerned having extremely fine internal porosity so that the large stearic acid molecules cannot penetrate all parts of the internal porous network but the smaller gas molecule can. It is also possible that a double molecular layer of gas was adsorbed by the powder

particles during the B.E.T. determination because these results are approximately twice the stearic acid adsorption value and a value of $0.3 \text{ m}^2/\text{g}$ corresponds to a very small mean particle size (approximately $2\frac{1}{2}$ microns).

Specific surface results obtained by the Lea & Nurse method are shown in table 7. Because of the assumptions which need to be made to calculate a value for the specific surface of the powders the results obtained were not absolute values. Nevertheless the results should be comparable to each other. Therefore the results have also been expressed upon a relative basis, defining the fine fraction of Makin powder as having the specific surface of 1.00.

Table 8 shows surface area results obtained by the Q.T.M. Again these results have been expressed upon a relative basis. Comparison between these results and those obtained by the Lea & Nurse method showed reasonable agreement for most powders. However the surface area of the ^{-3rd mesh} Woodall Duckam powder as measured by the Q.T.M. was significantly higher than that measured by the Lea & Nurse method. This feature was ascribed to the surface roughness of Woodall Duckam powder preventing close packing of particles in the permeametry bed. Thus the resistance offered to air flow would be less than expected.

5.60. APPARENT SOLID DENSITY RESULTS.

The apparent solid density results are reported in table 9. All of the results were very close to the theoretical density of pure iron. Consequently because of experimental error the estimates of the amount of closed porosity present in the powders

must be treated with reservation. However the results obtained for the fine fraction of J.J.M. 300 and the Sintrex Electrolytic powder showed densities sufficiently removed from the density of pure iron to be significant. The low density of Sintrex Electrolytic powder was thought to be due to either extremely fine interconnected porosity, such that the xylene molecules were unable to penetrate the inner regions of the powder particles, or to closed porosity. The former supposition was supported by the anomaly between the B.E.T. surface area measurements and the stearic acid measurements ($0.14 \text{ m}^2/\text{g}$ and $0.07 \text{ m}^2/\text{g}$).

5.70. COMPACTION RESULTS.

The compaction results are given in table 10. The Woodall Duckam -300 mesh powder and the two "as received" powders densified to approximately 6.50 g/cc . However the coarse elutriated fraction and fine elutriated fraction of J.J.M. 300 powder only densified to 6.35 g/cc . and 6.04 g/cc . respectively. Both of the B.S.A. water atomized powders could not be compacted successfully. The Sintrex powder compacted to a similar density at both 386 kN/mm^2 . (25 t.s.i.) and 463 kN/mm^2 . (30 t.s.i.) compaction pressures but the other powders showed a significant density increase when the compaction pressure was increased over the same range.

5.80 CHEMICAL ANALYSIS.

Although the chemical analyses of the powders investigated were not determined the chemical analysis for the water atomized B.S.A. -100 mesh powder is given in table 11⁴⁰, this analysis was determined by the supplier. Typical analyses for the other powders examined are also shown in table 12⁴⁰. These results were not

determined upon the batches of powders examined but upon different batches manufactured to the same specification. Because the analysis of the B.S.A. water atomized powder was obtained for the -100 mesh fraction, it is not necessarily the true analysis for the -300 mesh fraction sieved from the same batch. The oxygen figures for the powders were obtained by a vacuum fusion technique.

The carbon content of the B.S.A. water atomized powder was 0.15% compared to 0.08% in the J.J.M. 300 powder, 0.015% in the Woodall Duckam powder and 0.008% in the Sintrex Electrolytic powder. The sulphur and phosphorous contents were too low to have an appreciable influence on mechanical properties and also were the silicon contents (range 0.01 to 0.07%). The manganese content of the J.J.M. 300, Woodall Duckam and B.S.A. powders were similar (0.25 - 0.35%) however only 0.04% was present in the Sintrex powder. The oxygen content of the Makin powder (0.85%) and B.S.A. powder (0.33%) was significantly higher than the Sintrex powder (0.15%) and the Woodall Duckam powder (0.18%).

The high oxygen content of the Makin powder is consistent with the observation of slate grey inclusions, believed to be iron oxide, in the micrographs. Moreover such a high oxygen content may account for the low apparent density of the fine fraction of J.J.M. 300 powder.

5.90. STEREOSCAM PHOTOGRAPHS OF FLEXISTRIP.

S.E.M. photomicrographs of "flexistrip" obtained by B.I.S.R.A. of the B.S.A. -100 mesh powder compacted at approximately 100kN/25 m.m. (10 tons/inch) width showed that the methyl cellulose surrounded the powder particles as a porous, fibrous network.

Contact of the methyl cellulose with the powder particles was not continuous but consisted of numerous strands or filaments.

(of the order of 20) which secured each particle to the methyl cellulose network.

5.100. GREEN STRIP DENSITY.

The increase in density with increase in roll load is shown in fig. 31. It was apparent that high densities (7 - 7.35 g/c.c.) were attainable at roll loads of 120 kN/25 mm. (12 tons/1" width). The density/roll load relationship for the fine fraction of Makin powder was linear over the whole range of roll load investigated but a significant departure from linearity occurred for the other powders. Optimum densification was shown by the Sintrex powder.

5.110 GREEN STRENGTH.

Fig. 32 shows the relationships between green strength and roll load. At intermediate roll loads of 100kN - 140kN/ 25 mm. width (10/14 tons/1") there was a large variation in the range of green strengths (of the order of 50 - 60 N/mm².) for the coarse and fine fractions of J.J.M. 300 respectively. However at higher roll loads, of the order of 180kN/25 mm. (18 tons per 1") width, the green strengths of strip from the fine fraction of J.J.M. 300, "as received" J.J.M. 300 and Woodall Duckam -300 mesh powder were very similar (of the order of 160N/mm².) but the green strength of strip rolled from the coarse fraction of J.J.M. 300 remained at approximately 103N/mm². for roll loads above approximately 100kN/25 mm. (10 tons per 1") width. Values for green strength for the Sintrex powder could not be obtained above 140kN/25 mm. (14 tons/1") width because of substantial

edge cracking. Green strengths of the B.S.A. -100 mesh and -300 mesh fractions were 25.2N/mm^2 and 19.9N/mm^2 respectively.

5.120. RESISTIVITY OF GREEN STRIP.

The variation of resistivity with increase in roll load for strip rolled from the powders examined is shown in fig.33. Strip rolled from the fine fraction of J.J.M. 300 and "as received" J.J.M. 300 showed little variation of resistivity for values of roll load up to $180\text{kN}/25\text{ mm}$. (18 tons per 1") width at which there was a slight increase in resistivity. The increase in resistivity at higher roll loads was very pronounced with strips manufactured from the coarse fraction of J.J.M. 300 and from Woodall Duckam powder. In the latter case there was a sharp increase in resistivity values for roll loads exceeding $110\text{kN}/25\text{ mm}$. (11 tons per 1") width. Although strip from Sintrex powder was examined over a much smaller range of roll load the indication was that not only did Sintrex powder show much higher resistivities but that there was also a very large increase in resistivity with increase in roll load.

Generally green compacts compacted by conventional powder metallurgy techniques may be expected to show a decrease in resistivity with increase in compaction force. The only strip which followed this trend was strip from Woodall Duckam powder for which there was a distinct decrease in the value of resistivity for the load range $40\text{kN}/25\text{ mm}$. to $100\text{kN}/25\text{ mm}$ s. (4 tons/1" to 10 tons/1") width.

5.130. SINTERED DENSITY.

Some results of sintered density are given in table 13. It was apparent that little or no densification occurred during sintering. This was anticipated because sintering times were very short.

5.140. SINTERED STRENGTHS.

All specimens of strip showed a rapid increase in strength during the first 60 seconds of sintering, as shown in table 14. Sintering for up to a further 240 seconds longer generally showed little further improvement in tensile strength. Most samples of strip attained strengths between 160 to 210N/mm². after sintering for 60 seconds. Notable exceptions were strip rolled from Sintrex powder and the -100 mesh fraction of B.S.A. powder. Strip rolled from Sintrex powder showed a continual increase in strength with sintering times of up to 300 seconds for samples rolled at 95kN/25 mm. and 120kN/25 mm. (9.5 tons/1" and 12 tons/1") width. Samples rolled at 45kN/25 mm. (4.5 tons/1") width attained a maximum tensile strength after two minutes sintering. Moreover only strip rolled at 120kN/25 mm. (12 tons/1") width developed a tensile strength greater than 200 N/mm². Strip rolled from the -100 mesh fraction of B.S.A. powder showed a maximum strength of 150N/mm². after 300 seconds sintering, moreover the rate of increase in tensile strength with sintering time was significantly lower than that of the -300 mesh fraction.

Some samples of strip showed a decrease in tensile strength after sintering for 120 seconds, e.g. all of the specimens prepared from ^{-300 mesh} Woodall Duckam powder and specimens prepared from.

"as received" Makin powder rolled at 160kN/25 mm. (16 tons/1") width.

Specimens rolled at the higher roll loads usually possessed higher tensile strengths than samples rolled at lower roll loads. Exceptions were strip rolled from Woodall Duckam powder at 130kN/25 mm. (13 tons/1") width which was significantly less strong than similar strip rolled at 90kN/25 mm. (9 tons/1") width and strip from "as received" J.J.M. 300 rolled at 160 kN/25 mm. (16 tons/1") width, which showed a slightly higher tensile strength than strip rolled at lower roll loads after 60 seconds but a lower tensile strength after 120 and 300 seconds sintering. However it was apparent, particularly with strip rolled from "as received" Makin powder and Makin derived powders that the difference in strength between samples rolled at low roll loads 50kN/25 mm. (5 tons/1") width and samples rolled at higher roll loads (of the order of 200kN/25 mm. - 20 tons/1" width) was only of the order of 30N/mm².

Strip rolled from the fine fraction of J.J.M. 300 possessed tensile strengths which were very similar to the strengths of strip rolled from the "as received" J.J.M. 300 powder. However strip rolled from the coarse fraction possessed tensile strengths which were significantly lower after sintering times of 30 seconds and 60 seconds.

The elongation values were very low (less than twelve per cent) and significant elongation was only attained by samples sintered for long sintering times. Generally the elongation after

thirty seconds sintering was minimal. After sixty seconds the elongation was between two and six per cent and after three hundred seconds between six and twelve per cent. However all samples prepared from ^{-300 mesh} Woodall Duckam powder, with the exception of the series rolled at 90kN/25 mm. (9 tons/1") width showed very poor elongation figures (less than three per cent). Some samples, particularly specimens prepared from the coarse fraction of Makin powder developed an "orange peel" effect around the fracture area after sintering for three hundred seconds.

5.150.. RESISTIVITIES OF SINTERED STRIP.

The results of the resistivities of sintered strip are given in Figs.34 & 35. All of the specimens examined showed a very large reduction in resistivity during the first thirty seconds sintering. Only samples of strip prepared from B.S.A. powders were sintered for sintering times shorter than thirty seconds and it was apparent that even after ten seconds sintering, the resistivity was of the same order as resistivities developed after sintering for three hundred seconds. Strip from the -300 mesh fraction of B.S.A. powder had lower resistivity after sintering for ten seconds than strip from the coarser fraction. However after sintering for sixty seconds the resistivities of strip rolled from the two powder types had become very similar.

Results obtained for the resistivity of sintered strip showed appreciable scatter which made interpretation more difficult. This was particularly applicable to the resistivities of sintered strip rolled from ^{-300 mesh} Woodall Duckam powder, Sintrex powder and the

Makin derived powders where results were only obtained in duplicate. However distinct trends were observed in the variation of resistivity with sintering time for strip prepared from these powders. Most specimens developed a resistivity of between 150×10^{-7} ohm-cm. and 160×10^{-7} ohm-cm. after sintering for thirty seconds and the resistivity remained within this range even after sintering times of three hundred seconds. Strip made from Sintrex Electrolytic powder developed the lowest resistivities and most of these results lay between 120×10^{-7} ohm-cm. and 130×10^{-7} ohm-cm. Specimens rolled at low roll loads (of the order of 50kN/25 m.m. - 5 tons/1" width) had higher resistivities than specimens rolled at other roll loads ($160 - 180 \times 10^{-7}$ ohm-cm.) however roll loads in excess of 90kN/25 m.m. (9 tons/1") width were sufficient to develop resistivities in the $150 - 160 \times 10^{-7}$ ohm-cm. range. It is noteworthy that with the major exception of specimens prepared from Sintrex powder that there was very little variation in resistivity for sintering times of between thirty seconds and sixty seconds.

5.160. STEREOSCAN PHOTOGRAPHS OF SINTERED FRACTURES.

The Stereoscan photographs of the sintered fractures are illustrated in fig. 36. 37. & 38.

It was apparent that even after a short sintering time of ten seconds the B.S.A. powders showed areas possessing characteristic fibrous dimpling of a ductile fracture. However

the particles gradually lost their identities and a more coherent fracture appearance was obtained showing many more areas where fracture had occurred in a ductile manner.

Similar tends were followed by strip prepared from the derived Makin powders. However even after a sintering time of three hundred seconds the particulate nature of the strip can easily be recognised.

5.170. PROPERTIES OF THE FINISHED STRIP.

The results of the mechanical properties of the finished strip are shown in table 15.

The results were very scattered and incomplete but it is apparent that they were not equivalent to similar results for thin gauge mild steel plate manufactured conventionally.

THE ROLL COMPACTION OF IRON POWDERS

DISCUSSION

6.00 DISCUSSION CONCERNING THE ROLL COMPACTION OF IRON POWDERS.

Theories concerning the nature of green strengths of pressed metal powders attribute the strength to particle interlocking and cold welding. Increasing the compaction pressure causes the number and size of the cold welds to increase as repacking, plastic flow and densification of the metal powders occurs. The relationship of compaction pressure to density is not linear, mainly due to the work hardening of powder asperities.

During the roll compaction of iron powder by the slurry process there is an increase in the resistivity of green strip which occurs at a value of roll load which is specific for each powder. At approximately this roll load the green density also tends to level off or fall, at a value of 7.25 to 7.4 g/c.c. However the density of strip rolled from the fine fraction of J.J.M. 300 increased over the whole of the roll load range examined. It is noteworthy that the density of strip from the fine fraction of J.J.M. 300 is less than the other powders and that a decrease only occurred for strip prepared from the other powders at 7.25 to 7.5 g/c.c. Strip rolled from the J.J.M. 300 fine fraction did not attain this value. Therefore it is possible that a similar reduction in density might occur for strip rolled from J.J.M. 300 fines at roll loads greater than those examined. Because of substantial edge cracking, strip rolled from Sintrex powder could not be prepared for roll loads greater than 140kN per 25 m.m. width (14 tons per inch width).

Thus it would appear that when "flexistrip" is rolled

with progressive increments of roll load there is a stage at which the number of particle/particle welds increases followed by another stage where the welds are broken. Therefore a reduction in the density of particle packing would be expected, which is observed from strip prepared from "as received" Makin powder and the coarse fraction of Makin powder. The main reason that the density attains a constant value for strip prepared from ^{- 300 mesh} Woodall Duckham and Sintrex powders is probably due to the work hardening of powder particle asperities but it may be possible that particle welds formed in the early stages of compaction are broken and new ones, contaminated by methyl cellulose are formed. Thus the resistivity increases but the density remains constant. This mechanism probably occurs in strip rolled from J.J.M. 300 and the coarse fraction of J.J.M. 300 but the rate of formation of new welds is less than the rate at which old welds break, therefore the density decreases.

Strip rolled by the B.I.S.R.A. slurry process contains methyl cellulose, this is evident in Fig. 39 which shows a stereoscan photograph of a green strip fracture of strip rolled from -100 mesh B.S.A. powder rolled at 100kN/25 m.m. width (10 tons/inch width). It is apparent that there is very little porosity present because the methyl cellulose occupies the interstitial spaces between powder particles. Thus if the strip is rolled at higher roll loads than 100kN/22 m.m. width (10 tons/inch width), the extra pressure would squeeze the methyl cellulose between the powder particles possibly forcing them apart. Therefore

this behaviour of methyl cellulose accounts for the resistivity variations and to a certain extent for the density variations.

The methyl cellulose may be able to be accommodated in the internal porous networks of the chemically reduced powders. This is not possible with the relatively solid Sintrex electrolytic powder. Consequently at roll loads above 140kN/25 m.m. width (14 tons/inch width), the methyl cellulose can only force the powder particles apart, causing considerable edge cracking and splitting.

Conventional die compaction shows that the Woodall Duckam, Sintrex and "as received" Makin powders were densified to approximately 6.5 g/c.c. by a compaction load of 463N/m.m.² (30 t.s.i.) but that the fine fraction of J.J.M. 300 only densified to 6.04 g/c.c. and the coarse fraction to 6.35 g/c.c. Moreover the maximum density increase from 387N/m.m.² (25 t.s.i.) to 463N/m.m.² (30 t.s.i.) was of the order of 0.27 g/c.c.

Because of the smaller area upon which the rolls act during roll compaction much higher densities are attained by this method than conventional die compaction. Therefore a roll load of only 40kN/25 m.m. width (4 tons/inch width) was sufficient to densify Woodall Duckam powder to approximately 7.00 g/c.c. However the relative densification behaviour of the powders by die compaction was very similar to roll compaction, e.g. Sintrex electrolytic and Woodall Duckam powders showed optimum densification in both die and roll compaction. On the other hand the coarse fraction of J.J.M. 300 showed better densification by roll compaction than die

compaction.

It is generally agreed that particle size distribution affects the density of die compacted metal powders. It has been suggested that the ideal particle size is such that the voids between large powder particles are filled by finer particles and that even finer particles pack into the interstitial spaces of these. Therefore it is probable that the less efficient packing of the coarse fraction of J.J.M.300 is less of a disadvantage during roll compaction than die compaction because there are more internal voids present to accommodate the methyl cellulose.

The B.S.A. water atomized powders could not be die compacted successfully. Moreover the density of strip rolled from these powders was considerably less than the density of strip prepared from the other powders (6.8 g/c.c. compared to 7.2 g/c.c. approximately). Because of the hardness of the particles (550 Hv) little plastic deformation at the particle weld areas could occur, therefore the bonds formed were weak.

Despite the variation that occurs in density as the roll load increases there is no corresponding reduction in green strength. The green strengths of the powders investigated showed a rapid initial increase in strength, up to roll loads of the order of 100kN/22 m.m. width (10 tons/inch width). This was followed by a stage where there was very little increase, after which

there was a further rapid rise in green strength. However the coarse fraction of J.J.M. 300 showed little increase in green strength with loads above 100kN/25 m.m. width.

Strip rolled from Sintrex electrolytic powder showed higher green densities and resistivities but lower green strengths than strip rolled from either the fine fraction of J.J.M. 300 or "as received" J.J.M. 300. Moreover strip rolled from the fine fraction of J.J.M.300 gave optimum green strength results, but the lowest green density results. The green strength of the fine fraction of Makin powder and J.J.M. 300 is approximately 50% greater than the strength of the coarse fraction of J.J.M.300. Despite the large difference in green strength the density of strip rolled from the J.J.M.300 coarse fraction is greater than the fine fraction. Because the powders were derived from the same batch of "as received" powder it is very probable that only the difference in particle size distribution and specific surface is responsible for such behaviour.

In order to account for the anomalies between green strength and green density, the methyl cellulose must contribute towards the green strength of the strip, and to reduce the amount of stress acting upon each particle/particle contact area during tensile testing. The specific surface of the fine fraction of J.J.M.300 is approximately twice that of the specific surface of the larger particles. Therefore the adhesive effect of the methyl cellulose is developed over a much greater area resulting in higher green strengths. Also because the surface area of the Sintrex powder

is low compared with the other powders, the adhesive effect is much less and so strip prepared from Sintrex powder shows poor green strength results.

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It has been suggested that there needs to be a minimum number of particles across the strip thickness in order to realise optimum strength. This number is thought to be 10 particles. The maximum particle size of the coarse fraction of J.J.M. 300 is approximately 50 microns. Consequently in some areas less than ten particles may be present across the strip thickness. It may be possible that when there are only a few particle/particle contact areas across the strip thickness, then work hardening of powder asperities rapidly occurs, so that particle/particle welds do not increase in area as rapidly during compaction as in strip containing more particles across the strip thickness where the load is distributed over more particle/particle contact areas so that the plastic deformation per contact area is less.

The surface of the green strip contains a large number of notches and each notch is situated at particle boundaries. Strip rolled from fine powder has many more particle boundaries than strip prepared from coarse powder, therefore during tensile testing the stress raising effect per particle boundary for coarse powder is much greater and tensile failure may occur earlier.

Strip prepared from the B.S.A. atomized powders was of much lower green strength than the other powders examined (of the order of 20N/mm^2 - 1.3 t.s.i.). This was thought to be entirely attributable to powder particle hardness. Because of

the slightly higher carbon content (0.15% carbon) and the very low thermal capacity of individual powder particles, a martensitic structure formed on quenching so that the particles had hardness of the order of 550 Hv.

In contrast to the relationship of green strength and particle size distribution shown by the fine fraction of J.J.M.300 and the coarse fraction of J.J.M.300, the green strength of strip prepared from the -100 mesh fraction of B.S.A. powder was greater than the green strength of the -300 mesh fraction. This might be due to the presence of small particles in the -100 mesh powder that have welded to the coarser particles during atomizing. Consequently it would be more difficult for the particles to slide over each other and small welded particles would aid the adhesive effect of the methyl cellulose. Also, despite attempts to manufacture strip of uniform thickness from the atomized powders, strip rolled from the -100 mesh fraction was significantly thicker than strip from the -300 mesh fraction. (0.29 mms. compared to 0.25 mms.) Consequently effects due to the pressure distribution upon particle/particle welds may be minimised for the 100 mesh powder. Moreover the B.S.A. -100 mesh powder possessed an exceptionally large fraction, between 0 and 10 microns compared to the chemically reduced and electrolytic iron powders. Therefore a number of particles greater than the minimum ideal number would probably be present in the thickness direction of the strip.

Evidence of a large fine fraction in the -100 mesh and -300 mesh fractions of the water atomized powder is shown in the

Q.T.M. data and also total surface area results by stearic acid adsorption. The latter results are of special interest because they indicated that the -100 mesh fraction was of greater surface area than the -300 mesh fraction. Moreover the measured total surface area of both powders was greater than the other powders examined. Because of the lack of internal porosity in the atomized powders the results are entirely attributable to particle size. Q.T.M. data indicates that the bulk of the particles are between 0 and 10 microns. This figure is likely to be lower than the true fraction between 0 and 10 microns because of the restrictions of optical microscopy for detecting particles of between 0 and 1 micron and because of unavoidable particle agglomeration which would lead to the fine particles being counted in a larger size group.

The green strength, resistivity and density results for strip from the electrolytic and chemically reduced powders indicate that significant contribution towards green strength is made by the methyl cellulose and that an increase in surface area may substantially raise the green strength. However the green strength results for strip prepared from the atomized powders are approximately one fifth to one tenth of the values determined for the other powders, despite the surface area of the atomized powder being approximately twice that of the chemically reduced powders. Therefore it is possible that particle interlocking is more important than has been discussed previously. Alternatively, because of the large surface area of the atomized powders, a greater proportion of binder than was added to the

slurry might be required to give a coherent film around each of the particles.

The density results across the width of the 100 m.m. (4"), prepared from the B.S.A. powders showed a variation of 0.14 g/c.c. total scatter. The strip rolled from the -100 mesh powder showed a higher density in the centre, whereas strip prepared from the -300 mesh powder showed a higher density at the edges. It would be expected that if the rolling mill rolls were not cambered then the density in the centre of the strip would be less than the density at the strip edge because of roll bending. Alternatively if the camber of the rolls overcorrected for roll bending then the density at the strip centre would be greater than at the edges. Consequently because the density variations of the two powders indicate opposite trends they cannot be attributed to roll bending. However because of the limited number of samples examined the results are not conclusive and are more likely to be due to experimental error.

Most modern theories of sintering have established that during sintering vacancy diffusion from particle contacts occurs which leads to an increase in the volume of metal between particles and an increase in the radius of curvature of the material at the particle/particle welds. Consequently the strength of the material increases because of the increase in bond area and a reduction in the stress raising effect of the "notches" at the particle/particle welds. Densification occurs because of vacancy diffusion to free space at the peripheral areas of the sample.

It is generally thought that vacancies may diffuse through the metal powder particles, i.e. by volume diffusion or across the surface of powder particles, i.e. by surface diffusion. Measured diffusion coefficients indicate that diffusion by a surface mechanism is faster than a volume mechanism but as the amount of surface available for diffusion is restricted, then more material is likely to be transported by a volume mechanism.

Therefore during sintering there should be a decrease in the resistivity of the material, an increase in density and an increase in strength.

All of the specimens examined showed a rapid increase in strength and a decrease in resistivity during the first 30 seconds sintering. Generally there was little difference between the resistivity of strip sintered for 30 seconds and the resistivity after sintering for 300 seconds, indicating that little densification was occurring, however all samples showed a significant gain in strength on increasing the sintering time from thirty to sixty seconds. Therefore it would appear that after the first few seconds sintering little or no material transport to particle/particle contacts occurs, but that there is diffusion occurring within the bonds to reduce their notch sensitivity.

The lowest resistivities were developed in strip prepared from Sintrex electrolytic powder, possibly because of its greater chemical purity. Sintrex powder differed from the other powders because with the exception of strip rolled at

45kN/2.5 m.m. width (4.5 tons/inch width), all sintered specimens decreased in resistivity for sintering times of between 60 seconds and 120 seconds and there was an increase in strength for sintering times of up to 300 seconds. Thus it would appear that strip prepared from Sintrex powder shows greater sintering activity than the other powders. This is difficult to explain because of the low surface area of the powder which would indicate that less material would be transported by the faster surface diffusion mechanism compared to the other powders.

Except for specimens sintered for ten seconds the resistivities of strip prepared from B.S.A. -100 mesh powder and B.S.A. -300 mesh powder were very similar. Despite this the strength of strip prepared from the -300 mesh is much greater than the strength of strip from the -100 mesh fraction for sintering times greater than thirty seconds. This behaviour is almost certainly due to the larger grain size of the -100 mesh fraction because of the coarser initial particle size from which the strip was prepared. Because the grain boundaries of the strip are situated at the particle/particle boundaries and because most of the porosity is present at the grain boundaries, then a greater stress would be concentrated at the grain boundaries of the coarser powder during tensile testing.

Generally roll load appeared to have little effect upon resistivity for loads greater than 80/90kN/25 m.m. width (8/9 tons/inch width). This behaviour is compatible with the

green density results. However roll load had a much greater effect on sintered strength. Samples prepared from "as received" Makin powder, and both the coarse and fine fractions of Makin powder showed an increase in sintered strength with increasing roll load. The results obtained for the sintered strengths of Sintrex strip show considerable scatter but indicate the general trend that the sintered strength increases as the roll load increases.

This behaviour is difficult to interpret. The results indicate that high roll loads affect the sintering activity. Therefore the increase in the concentration of vacancies caused by the work hardening of powder particles must influence the sintering activity even though the effects of work hardening should be removed very early in heat treatment.

Strip prepared from ^{-300 mesh} Woodall Duckam powder and strip from J.J.M. 300 rolled at 160kN per 25 m.m. width showed a decrease in strength after sintering for 60 seconds. Grain growth was observed to occur in some samples which was evident by the occurrence of the "orange peel" effect on the surfaces of tensile specimens. Therefore it is likely that grain growth accounts for the reduction in strength of these samples.

Little change in density occurred during sintering as may be expected by such short sintering times. Therefore it is most unlikely to attain strip of theoretical density by a single rolling and sintering operation and that rerolling and resintering must be employed to achieve this.

The tensile results of the finished strip are unfortunately very scattered and all show very poor elongation values. Moreover the tensile strengths are substantially less than the tensile strength of temper 3 tinplate. These results are considerably at variance with those obtained by B.I.S.R.A. despite the properties of the green and first sintered strip being very similar to those obtained by B.I.S.R.A. The most likely explanation is that a time of several months elapsed between the sintering of the green strip and the subsequent rerolling and resintering. During this time growth of oxide films may have occurred within the strip which would severely curtail the attainment of optimum properties.

Despite the poor properties of the finished strip the indication is that the load of the first compaction stage is relatively unimportant as further densification occurs easily during the second roll compaction.

Although the results are very few the indication is that strip prepared from the finer fractions give better strength and elongation values.

This current work has been sufficient to give an insight into the phenomena occurring during roll compaction and sintering, however much work requires to be done to obtain a fuller picture of the mechanism operating.

The study of the compaction and sintering of strip is especially convenient because experimental techniques such as

the determination of resistivity and X-ray diffraction may be used to study the phenomena occurring during the first stages of sintering. Specific attention should be drawn to the form of the porosity developed in the strip during the first stages of compaction and sintering and to the determination of small amounts of residual porosity in the finished strip which may contribute towards premature failure.

THE ROLL COMPACTION OF IRON POWDERS

GENERAL CONCLUSIONS

CONCLUSIONS.

Chemically reduced powders of fine particle size and large surface area may be rolled to strip possessing higher green strength but lower density than powders of coarse particle size.

The presence of the binder causes a reduction in the green strength and green density of strip rolled from powder at high roll loads and may contribute towards edge cracking and splitting.

Strength develops within a few seconds during sintering but no densification occurs even after sintering times of five minutes. However material transport within the "neck" areas occurs, which significantly reduces the internal notch effect, so that the tensile strength improves with longer sintering times.

Strip prepared by the B.I.S.R.A. slurry method may be susceptible to deterioration when stored in the intermediate stages. However on a continuous production line this should not be a disadvantage.

THE FATIGUE PROPERTIES OF SINTERED NICKEL

MATERIALS AND APPARATUS

7.00. MATERIALS AND APPARATUS.

Sherritt Gordon nickel powder Grade S was used throughout the investigation. The average chemical composition of the powder was Co 0.068%, Cu 0.007%, Fe 0.011%, S, 0.020%, C 0.008% balance nickel.

The powder was chosen because of the near spherical shape of the particles. Thus it was hoped that if closely sized fractions of the powder were pressed and sintered a uniform pore size would be developed and by varying the particle size of the powder fraction the pore size could be varied.

Closely sized fractions were obtained by sieving the "as received" materials for one hour using an "Endecott" sieve shaker. The sieve sequence used was 100 mesh, 150 mesh, 200 mesh, 240 mesh, 300 mesh and 350 mesh B.S. sieves.

Initial compaction experiments with the -100 + 150 mesh fraction were attempted using a small double acting die so that a finished compact size of 25 mms. x 6.25 mms. x 6.25 mms. (1" x 0.25" x 0.25"). Sodium stearate, stearic acid and carbon tetrachloride were used as admixed lubricants. In some instances silicone oil was used as a die wall lubricant. However samples from which the fatigue and tensile specimens were obtained were pressed using a double acting die to give samples 100 mms. x 10 mms. x 10 mms. (4" x 0.4" x 0.4") wide. A taper of 0.25 mms. per 25 mms. (0.1" per 1") was incorporated in the ejection part of the die to avoid laminations.

The sieve cuts obtained from the "as received" powder were annealed in a vacuum furnace at 650°C under an atmosphere

of 97% nitrogen, 3% hydrogen. In order to ensure that the air had been completely excluded from the powder mass the vacuum furnace was pumped to a vacuum of 10^{-4} Torr before introduction of the inert atmosphere and subsequent heating. The furnace is shown schematically in fig. 40.

The same furnace was used for preliminary sintering. However for such sintering the oil diffusion pump was used so that a vacuum of the order of 10^{-6} Torr was maintained for the duration of the annealing operation. The temperature gradient of the furnace was determined over a 150 mm. length and was found to be within plus or minus 2°C .

Some of the samples were compacted to high densities using an isostatic compaction technique. The press used oil as the load transferring medium and the sample was sealed from the oil by placing it into a tightly fitting polypropylene tube plugged with rubber bungs.

Final sintering of the pressed nickel compacts was achieved using a platinum resistance furnace. The temperature gradient of the furnace was determined to be within 4°C at $1,300^{\circ}\text{C}$. A sintering atmosphere of 75% hydrogen, 25% nitrogen was used for all of the specimens from which fatigue samples were to be prepared. However atmospheres less rich in hydrogen were used for some initial sintering experiments. The sintering atmosphere was dried and purified before use by passing the mixture through "molecular sieve" to remove water vapour and subsequently through a "De Oxo" Catalytic Purifier followed by another "molecular sieve" column to remove oxygen. It was estimated that the cleaned gas had a dewpoint better than -40°C before introduction into the

sintering furnace.

Tensile tests were made on a "Hounsfield Tensometer" for which No. 13 were machined and fatigue tests were conducted at 50 Hz. using a Bristol Siddeley type 1B Wohler fatigue testing machine. Unnotched fatigue samples were machined to the specification shown in fig.41 using the following procedure:-

- 1) The samples were turned to approximately 5.25 mms.
- 2) A 3.5 mm. radius was turned to 0.12 mms. oversize.
- 3) Two cuts were made 0.025 mms. deep by turning.
- 4) One cut was made 0.0125 mms. deep by turning to give the finished size.
- 5) The sample was polished longitudinally to remove any transverse machine markings. Final polishing was carried out using 600 grade silicon carbide paper and "Brasso" polishing.

Pore size distributions were measured on transverse sections of the pressed compacts. The samples were metallographically prepared in the usual manner by grinding on progressively finer grades of silicon carbide paper followed by polishing with 6 micron and finally 1 micron diamond paste. Ion etching was used to develop the microstructure and the mean grain size quoted is the number average chord length.

Grain size determinations of the specimens were made using a linear intercept method.

Observations of the fatigue fractures were made using optical and scanning electron microscopy.

THE FATIGUE PROPERTIES OF SINTERED NICKEL

EXPERIMENTAL TECHNIQUES

8.00. EXPERIMENTAL TECHNIQUES.

8.10. POWDER CHARACTERISTICS.

The tap density of the powders was measured by adding 100 g. of powder to a measuring cylinder and measuring the volume after tapping. A Hall flowmeter was used to evaluate the flow rate of the "as received" powder and powder fractions used in the investigation. Metallographic samples were prepared in the usual way.

8.20. INITIAL TRIALS.

8.21. COMPACTION.

The initial compaction trials were conducted using the 25 mms. x 6 mms. x 6 mms. die. Sodium stearate was initially used as a die wall lubricant. Application was effected by preparing the soap as an emulsion in acetone and coating the die wall. After the evaporation of the acetone the die was bolted together and filled with powder. Compaction pressures of 309N/mm^2 . (20 t.s.i.), 386N/mm^2 . (25 t.s.i.) and 463N/mm^2 . (30 t.s.i.). were used with both the "as received" powder fraction and the -150 + 200 mesh powder fraction.

Further trials were carried out using an "admixed" lubricant. Such a lubricant is usually introduced as a solid and a homogeneous mixture is obtained by ball milling for several hours. However it was thought that such a treatment might result in the distortion of the metal powder particles. Consequently stearic acid was chosen as the lubricant which was dissolved in petroleum ether. The solution was then introduced to the nickel powder. The mixture was vigorously stirred until the

majority of the petroleum ether had evaporated. The residual solvent was allowed to volatilize by maintaining the nickel powder at approximately 60°C until the mixture appeared to be free running. Lubricant additions of $\frac{1}{4}\%$, $\frac{1}{2}\%$ and 1% were made and compaction trials were conducted at the compaction pressures mentioned previously.

The adoption of these techniques with the larger die giving compacts of 100 mms. x 10 mms. x 10 mms. (4" x 0.4" x 0.4") was not successful. Compacts were either not coherent or severely laminated. Later trials were conducted using carbon tetrachloride mixed with the powder and a die wall lubricant of silicone oil. Although this technique produced better compacts some laminations still occurred however these were avoided by using annealed powder. Compaction loads of up to 542N/mm². (35 t.s.i.) were evaluated. This later technique was adopted for the preparation of tensile and fatigue specimens.

8.22. "DE WAXING".

Samples prepared using either sodium stearate or stearic acid as a lubricant were slowly heated to 400°C in a stream of dried, purified hydrogen and maintained at that temperature for thirty minutes to allow the lubricant to volatilize.

8.30 SINTERING.

Samples were sintered in vacuum and in hydrogen at 950°C and 1000°C for varying times. Further trials were conducted at temperatures of 1200°C and 1300°C in a sintering atmosphere of 97% nitrogen/3% hydrogen, 50% nitrogen/50% hydrogen or 75% hydrogen/25% nitrogen. A heating rate of 5°C. per minute was maintained.

for all of the trials and cooling from the more elevated temperatures of 1200°C and 1300°C was achieved in eighty five minutes.

A isolated sample from the -240 + 300 mesh powder which had been pressed at 350 N/mm^2 . (35 t.s.i.) to 6.8 g/c.c. was oxidised for one hour at 550°C , before sintering in hydrogen at 1300°C for sixty minutes.

8.31 FURTHER TRIALS.

A number of samples of specimens were prepared by pressing at 540 N/mm^2 . (35 t.s.i.) and sintering in vacuum at 1000°C . These specimens were repressed at varying loads and resintered at 1300°C .

In attempt to press samples of high density an annealed sample of the -200 + 240 mesh powder was pressed at 465 N/mm^2 . (30 t.s.i.) in the smaller die (25 mms. x 6 mms. x 6 mms. - $1" \times \frac{1}{4}" \times \frac{1}{4}"$). The sample was sintered in hydrogen for fifteen minutes at 750°C . Multiple repressing and annealing of the samples at the above pressure and temperature was continued.

8.40. THE PRESSING OF TENSILE AND FATIGUE SAMPLES.

All of the bars from which the fatigue samples were prepared were pressed using the larger 100 mms. x 10 mms. x 10 mms. die ($4" \times 04" \times 04"$). 1.5 mls. of carbon tetrachloride was used as an "admixed" lubricant and silicone oil as a die wall lubricant.

Three basic series were produced:-

1) Series 1.

This series was of 10 - 13 per cent total porosity.

Annealed fractions of the -150 + 200 mesh, -200 + 240 mesh and -240 + 300 mesh fractions were pressed at 340N/mm^2 . (22 t.s.i.) sintered for one hour at 1000°C . in a vacuum of pressure 10^{-6} Torr and repressed at 310N/mm^2 . (20 t.s.i.). Final sintering was conducted for three hours at 1300°C . in an atmosphere of 75% hydrogen 25% nitrogen.

ii) Series 2.

This series was prepared using the three powder fractions mentioned previously and was of 3 - 4 per cent total porosity.

The annealed powders were initially pressed at 290N/mm^2 . (18.7 t.s.i.) and sintered for fifteen minutes at 850°C . They were subsequently repressed at 310N/mm^2 . (20 t.s.i.) followed by isostatically repressing at 770N/mm^2 . (50 t.s.i.). Final sintering was again conducted at 1300°C . for three hours.

iii) Series 3.

This series was prepared from annealed, "as received" powder as an attempt to secure a density as close as possible to the theoretical density. However the final porosity content was of the order of 4 - 5 per cent.

Samples were initially pressed at 290N/mm^2 . (18.7 t.s.i.) and vacuum sintered for fifteen minutes at 870°C , isostatically pressed at 1080N/mm^2 . (70 t.s.i.) and finally sintered for three hours at 1300°C .

8.50. DENSITY DETERMINATION.

The density of all the rectangular pressed compacts was found by mensuration but the mercury balance technique was used for the machined tensile and fatigue samples.

THE FATIGUE PROPERTIES OF SINTERED NICKEL

RESULTS

9.00 RESULTS.

9.10 POWDER CHARACTERISTICS.

The values for apparent density, tap density, and flow rates for the "as received" powder and the sieve fractions investigated are given in table 16.

The "as received" powder showed a slightly higher apparent density and tap density indicating the importance of particle size distribution upon these properties. However the powder characteristics of the sieve fraction were very similar.

Figure / illustrates the microstructure of the "as received" powder. It is evident that the porosity is present as a number of fine concentric rings. 42

9.20. INITIAL COMPACTION TRIALS.

All the samples pressed using the smaller die were laminated. Compacting from 310N/mm^2 . (20 t.s.i.) to 450N/mm^2 . (30 t.s.i.) gave densities of 6.0 g/c.c. to 6.3 g/c.c. However such low densities were probably mainly due to the presence of laminations.

Samples pressed using a smaller die and an "admixed" lubricant were considerably more successful.

Densities of 6.6 g/c.c. to 6.7 g/c.c. were measured on specimens containing one quarter per cent and one half per cent lubricant. Several laminations were present in the samples containing one per cent lubricant.

The technique of mixing a small amount of stearic acid with the powder was not successful with the 100 mm. x 10 m. (4" x 0.4") die as compacts so pressed were laminated. The use of an annealed powder helped to reduce the occurrence of laminations but not to prevent them entirely. However substituting carbon tetrachloride as an "admixed" lubricant gave lamination free compacts of approximately 6.8 g/c.c. for all the powder fractions investigated.

9.30 INITIAL SINTERING TRIALS.

Sintering at 1000°C either in vacuum or hydrogen gave no densification. However increasing the sintering temperature to 1200°/1300°C increased the density of samples from the -200 + 240 mesh fraction pressed to 6.8 g/c.c. to 7.3/7.6 g/c.c. Similar densification occurred both in

97 per cent nitrogen, 3 per cent hydrogen and 75 per cent hydrogen, 25 per cent nitrogen mixtures.

The density of the isolated sample sintered in hydrogen for one hour after oxidising in air at 550°C increased from 6.7 g/c.c. to 7.8 g/c.c.

9.40. TRIALS INVOLVING FURTHER TREATMENT.

Repressing samples, originally pressed to 6.8/6.9 g/c.c. and sintered in vacuum for 60 minutes at 1000°C , at loads of 155N/mm^2 . (10 t.s.i.) to 465N/mm^2 . increased the densities to 7.5/7.8 g/c.c.

Mutiple repressing followed by annealing gave final densities of 8.3/8.4 g/c.c. The increase in density with successive compactions is shown in table 17.

9.50. MICROSTRUCTURES OF SINTERED SPECIMENS.

Although densities of 7.6/7.8 g/c.c. could be attained by a single pressing and sintering operation, the shape of the pores of the specimens was more irregular and film like compared with specimens that had been sintered at a lower temperature and pressed to a density of 7.6/7.8 g/c.c. followed by a three hour heat treatment at 1300°C .

The porosity of the samples used in the three fatigue series was in two forms. Normal rounded porosity was evident in all of the samples associated with film like porosity. The film like porosity was especially prevalent in the samples of lower density. (Fig. 43) The film like porosity was more prominent in samples electrolytically etched in 5% sulphuric acid than

specimens prepared by ion etching (Figs.43.44). Consequently pore size distributions were measured on ion etched samples.

Fig. 45 shows a fractograph of the "as received" powder which was compacted and sintered to four per cent residual porosity.

It is evident that the film like porosity extends throughout the entire structure and is at the boundaries of the original powder particles of the specimen. Moreover it is apparent that much of the rounded porosity is linked with the film like porosity.

Pore size distribution, mean pore size and porosity contents for specimens from the three fatigue series are shown in table 18.

The microstructures of two of the specimens (figs.44.46.) showed equiaxed, twinned grains but the grain sizes of the specimens from each of the fatigue series did not vary greatly. The average particle sizes of the powder fractions investigated were 89 microns (for the -150 + 200 mesh fraction), 68 microns (for the -200 + 240 mesh fraction) and 57 microns (for the -240 + 300 mesh fraction). Therefore the occurrence of one or two twins per particle would lead approximately to the values of grain size determined.

9.60. TENSILE PROPERTIES.

Table 19 shows the tensile properties of representative samples from each of the series of specimens. All of the tensile fractures were intergranular and fracture occurred with a reduction of area value which was large for sintered materials but low compared with wrought nickel. The elongation of each of the samples

was very similar and independent of the original particle size or processing sequence. It is evident that the original powder particle size for each series has only a small effect upon the final tensile strength but the data shows that samples prepared from the coarser fractions are weaker than samples prepared from the medium and finer fractions.

9.70. FATIGUE PROPERTIES.

The fatigue results for the three series are given in table 20. The endurance limit together with the endurance ratios are listed in table 19. Unfortunately considerable scatter was evident for specimens fatigued at higher stresses but the endurance limits for each of the fatigue series was clearly defined. The endurance limits of the three groups of samples of approximately four per cent porosity were almost identical and had the same value as the four per cent porosity samples prepared from the "as received" powder. Increasing the total porosity content to approximately twelve per cent substantially reduced the endurance limit. Moreover the endurance ratio decreased from 0.45 to approximately 0.36. The higher porosity group also showed some evidence of the effect of particle size upon properties because samples prepared from the -150 + 200 mesh fraction showed a lower endurance limit compared to samples prepared from the fine and medium fractions.

Fractographs obtained using a scanning electron microscope showed an increasing coarseness of fracture for both

the four per cent porosity series and the twelve per cent porosity series as the particle size increased. The fracture appearance of the specimen groups containing twelve per cent porosity was of three distinct types. One type showed well rounded features with small undulations which appeared to be derived from the original particle shape (Fig.47), another appeared slightly flattened as might have occurred if two particles had been pressed against each other but not sintered together and the final facet showed the rough striated appearance of the fatigue fracture. In some of the latter areas fatigue striations were evident, while in others some dimpling was present, however some further areas existed where the type of fracture could not be readily determined.

Fractographs of the fatigue series containing lower porosity (figs.48. 49.) differed from the high porosity series in that the fracture surface appeared more crystalline and that rounded porosity was less evident. Examination of the fracture surface at higher magnification showed smooth facet like surfaces which were interconnected by rougher regions frequently showing striations.

THE FATIGUE PROPERTIES OF SINTERED NICKEL

DISCUSSION

10.00. DISCUSSION.

The initial compaction trials showed that a lubricant of carbon tetrachloride was superior to conventional lubricants, e.g. sodium stearate and stearic acid, because the incidence of laminations in compacts were reduced by this lubricant. Because of the nature of conventional lubricants it is doubtful whether carbon tetrachloride would facilitate the sliding of powder particles over each other. It is possible that as carbon tetrachloride is an organic liquid of high vapour pressure, that some air would be excluded from the uncompacted powder by carbon tetrachloride vapour. During compaction the ^{increase in} pressure would cause the carbon tetrachloride to liquify thereby reducing the internal pressure within the compact enabling the powder particles to move closer together.

The sintering trials on specific fractions of Sheritt Gordon nickel powder clearly show its poor sintering activity because temperatures in excess of $1,200^{\circ}\text{C}$ were required for significant densification.

The fact that a more homogeneous structure was obtained by a technique of pressing and sintering at $1,000^{\circ}\text{C}$ followed by coining and resintering at $1,300^{\circ}\text{C}$ is probably due to the more porous areas in the compact, present after the first compaction stage being reduced during the coining operation.

Unfortunately the original aim of producing three distinct groups of pore size in a number of series of differing porosity was

not realised because of the distinctive form of porosity produced. The shape of the pores were of two types, rounded and film like. The rounded porosity was thought to originate from the voids formed at particle corners. However some of the porosity (of the order of 1 - 2%) originated from the powder particles themselves. The microstructure of the powder particles shows the presence of a number of concentric rings of fine porosity. This is consistent with the processing technique used in the powder manufacture. This porosity would spherodise during sintering to give porosity within the grains. The film like porosity probably originated from the original particle boundaries.

The Sheritt Gordon powder particles were botryoidal in shape. Therefore during compaction it was unlikely that the full facets of the particles came into contact. Instead welds would be formed at particle asperities. Subsequent sintering would occur at these asperities but as little or no densification occurred during sintering it is unlikely that the particles would move together to give a particle/particle bond over the entire periphery of the particles. Consequently the gap between any two particles would later be observed as film like porosity.

Some disagreement exists between measurements of porosity as determined by the Q.T.M. technique and density. Such a discrepancy may be attributed to the failure of the Q.T.M. to distinguish between film like porosity and grain boundaries. Indeed the distinction was frequently difficult to make when

examining the particles through an optical microscope. Consequently for a given porosity content the Q.T.M. only measured the rounded porosity stemming from the powder particle corners of the original particles and to a lesser extent from the intra particle porosity.

The film like porosity appears to have dominated the fatigue behaviour. This is illustrated by the intergranular fashion in which the fatigue cracks propagated. Therefore the fatigue results of each density group were very similar.

It was not possible to confirm that fatigue cracking was initiated at the surface of the fatigue specimens. This type of nucleation is thought to be the most probable because the samples were tested in rotating bending in which the surface of the sample is most highly stressed. Moreover it is most likely that fatigue crack nucleation would occur at the points where the film like porosity intersects the surface because such surface discontinuities would be very effective stress raisers.

Once the fatigue crack has nucleated it would propagate in a step by step manner until it encountered a film like pore. Thus the fatigue crack would be stopped and the fatigue process would have to be renucleated at the pore tip.

Thus once fatigue cracking has initiated, propagation is discontinuous because the fatigue crack would be frequently stopped and fresh nucleation would need to occur at the pore edge. Finally the weakened material would fracture by a tensile mechanism.

Many investigators have reported that sintered materials

show less scattered results than wrought materials. However the fatigue results obtained at higher stress levels show considerable scatter which is consistent with the work of Radomysel'sky²⁸. This is thought to be due to the important role that film like porosity plays in fatigue crack propagation and the fact that the connectivity of the film like porosity may vary from sample to sample.

The endurance limit of the fatigue series containing 12 per cent porosity pressed from the coarse -150 + 200 mesh powder was significantly lower than the endurance limits of samples of the same porosity content but pressed from the medium and finer fractions. The particle boundaries of the coarser powders occupy less area than the particle boundaries of the finer powders. Therefore the stress concentration on the particle boundaries of samples prepared from the coarser powder is greater and the specimens fail earlier.

The fractographs of the fatigued specimens showed three distinctive areas in the fatigue fracture. One of these areas appeared slightly flattened as might have occurred if two particles had been pressed together but not sintered. These areas may have originated from the coining operation. Thus when the compacts were repressed the powder particles were moved closer together but insufficient force was available to cold weld the particles so that the initiation of true particle/particle bonds by sintering would be more difficult.

THE FATIGUE PROPERTIES OF SINTERED NICKEL

GENERAL CONCLUSIONS

CONCLUSIONS.

The series containing four per cent porosity showed higher endurance limits and lower endurance ratios than the series containing twelve per cent porosity. The endurance limits of each group of the four per cent series were very similar. However the group of specimens of twelve per cent porosity prepared from the coarser powder showed a slightly lower endurance limit than specimens of the same porosity content prepared from the finer powder.

The fatigue properties were governed by the form of porosity which was of two distinct types, rounded and film like. The rounded porosity arose mainly from the corners of the original particles and to a lesser extent from porosity within the original powder particles. The film like porosity arose from particle boundaries. The fatigue fracture was granular and propagated along the film like porosity.

The failure to attain varying pore size was entirely due to the incidence of the film like porosity.

SOME PROPERTIES OF SINTERED MATERIALS

CONCLUSIONS

11.00. CONCLUSIONS.

The mechanical properties of sintered materials are strongly influenced by the presence of porosity. Total porosity has the major effect upon properties but pore shape may contribute a very important part especially if it is film like.

Particle size distribution has a definite effect upon compaction properties. Powders containing a large fraction of fine particles generally compact to give stronger but necessarily denser specimens.

In order to obtain densification during sintering, sintering temperatures must be high. No significant densification occurred when sintering iron or copper, sintering at temperatures 400°C from their melting points.

APPENDICES

APPENDIX 1.

THE DETERMINATION OF DENSITY BY THE
MERCURY BALANCE TECHNIQUE.

If W_a = the weight of the specimen in air

W_b = the weight to immerse the mercury balance
without a sample.

W_c = the weight to immerse the mercury balance
plus sample.

D = the density of mercury at room temperature.

Then the weight required to immerse the sample
= $W_c - W_b$

The upthrust of the specimen in mercury = $W_a + (W_c - W_b)$

Therefore the volume of the specimen = $\frac{W_a + (W_c - W_b)}{D}$

and the density of the specimen = $\frac{D \cdot W_a}{W_a + (W_c - W_b)}$

APPENDIX 2.

THE DETERMINATION OF THE DENSITY OF SINTERED
SAMPLES BY THE XYLENE IMPREGNATION METHOD.

If W_a = weight of sample in air

W_x = weight of xylene impregnated sample.

W_y = weight of sample in xylene.

D = density of xylene.

Then:-

Weight of xylene absorbed by the sample = $W_x - W_a$

The volume of xylene absorbed = $\frac{W_x - W_a}{D}$

This is equivalent to the volume of the interconnected porous network of the specimen.

The upthrust when immersed in xylene = $W_a - W_y$

Therefore the apparent volume of the sample = $\frac{(W_a - W_y)}{D}$

The total volume of the specimen = $\frac{(W_a - W_y)}{D} + \frac{(W_x - W_a)}{D}$

the density of the sample = $\frac{W_a}{\left(\frac{W_x - W_y}{D} \right)}$

$$= \frac{D W_a}{W_x - W_y}$$

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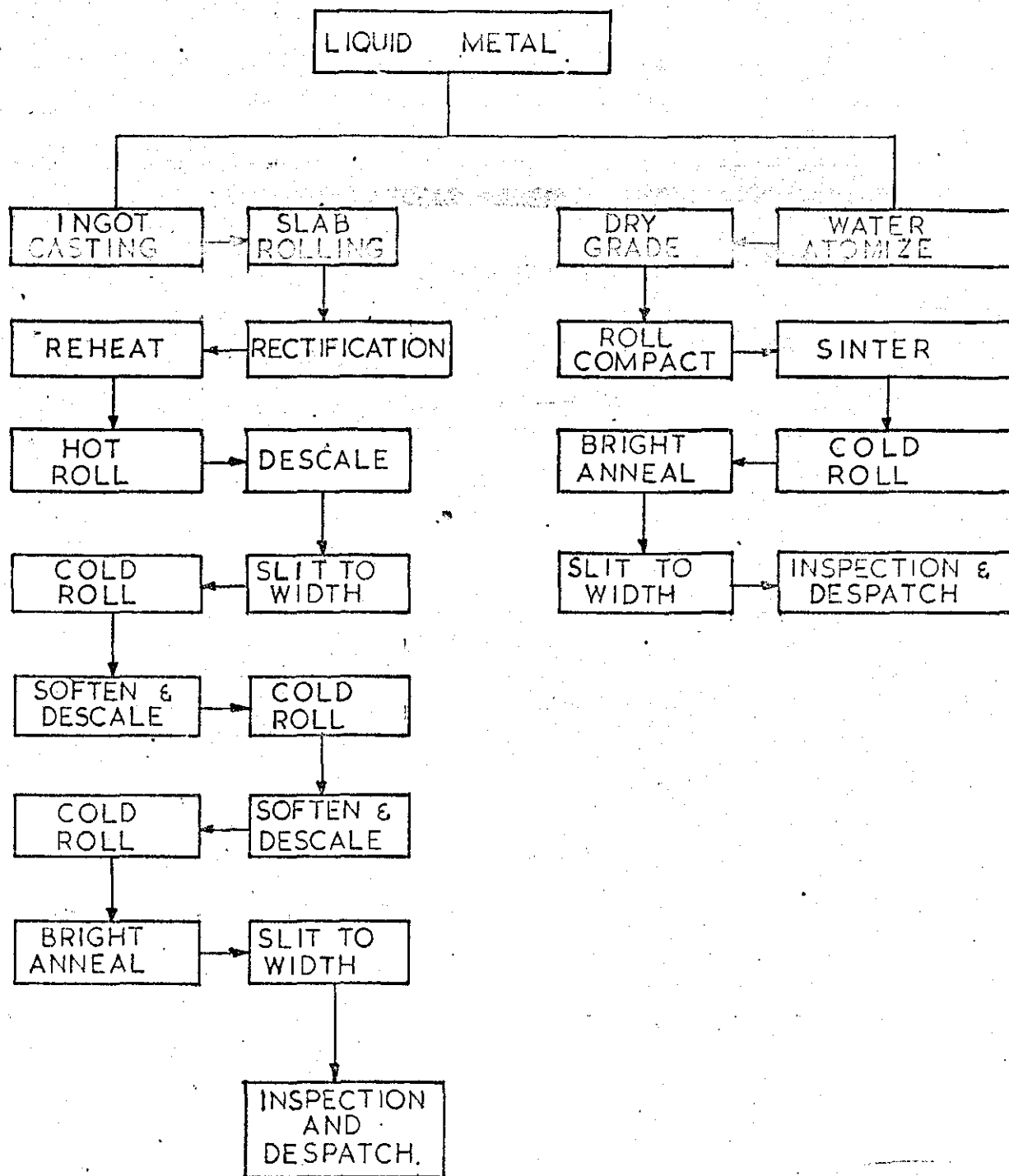
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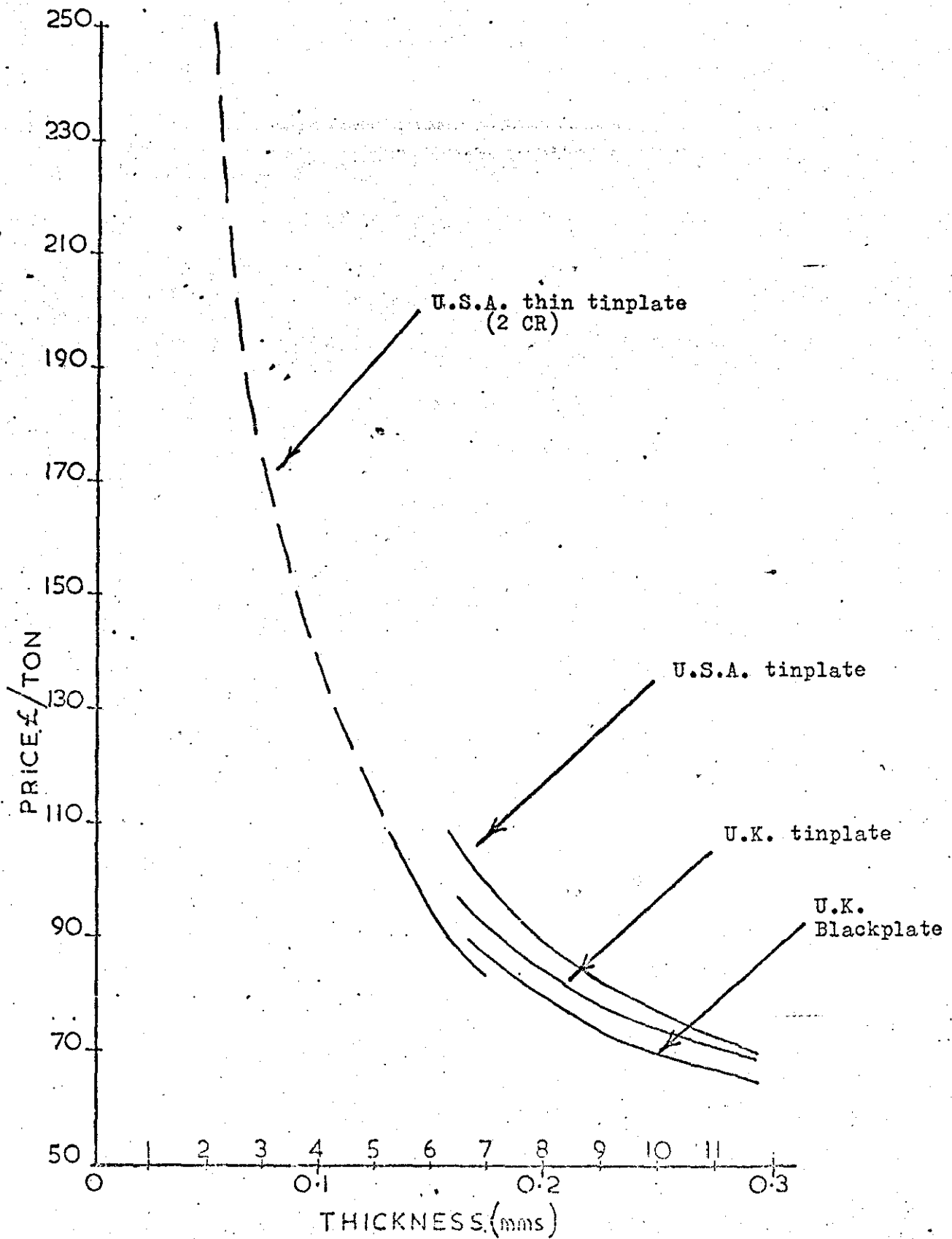
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FIG. N° 1.



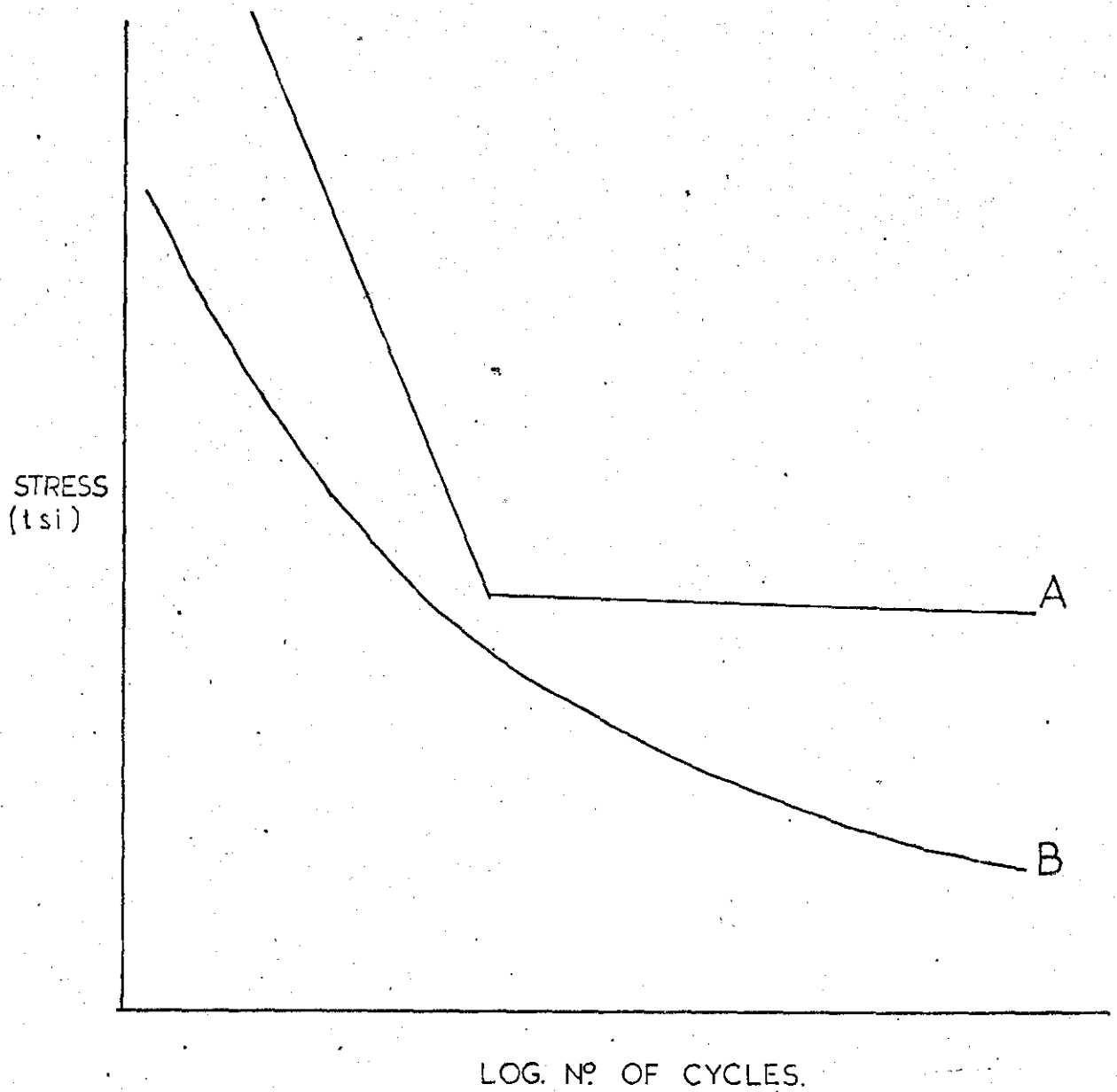
COMPARISON OF CONVENTIONAL AND
POWDER PRODUCTION ROUTES FOR STRIP.

FIG. N° 2



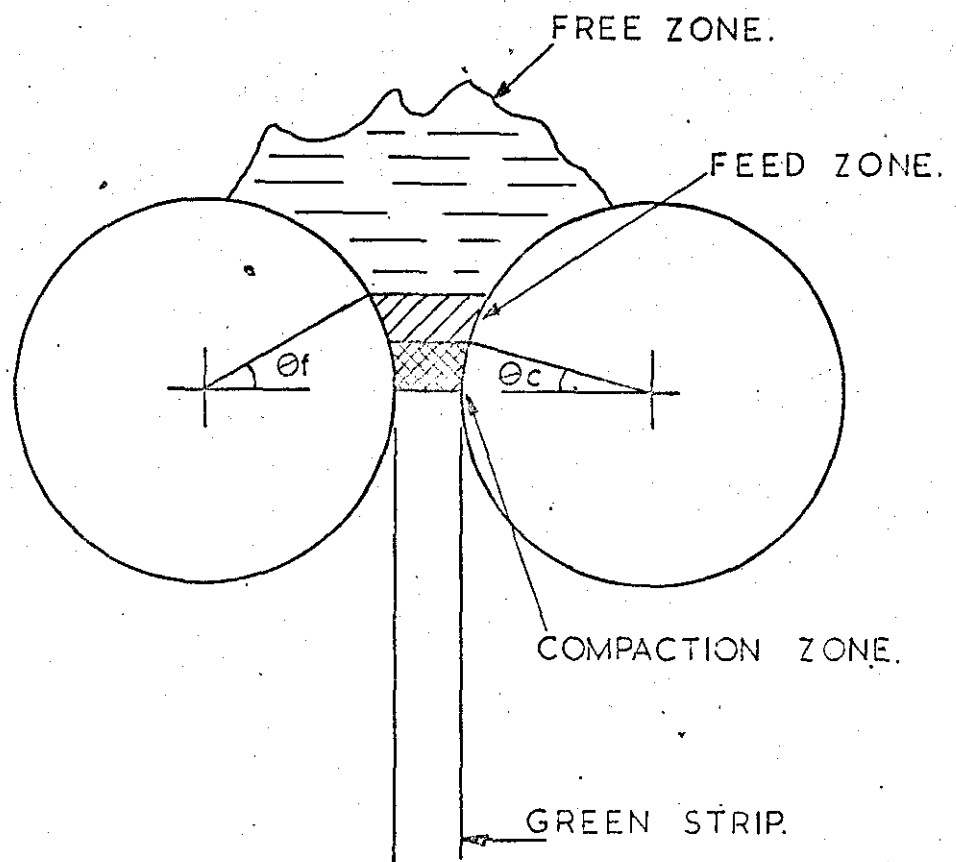
SELLING PRICES OF THIN
STEEL STRIP (1968.)

FIG. N° 3.



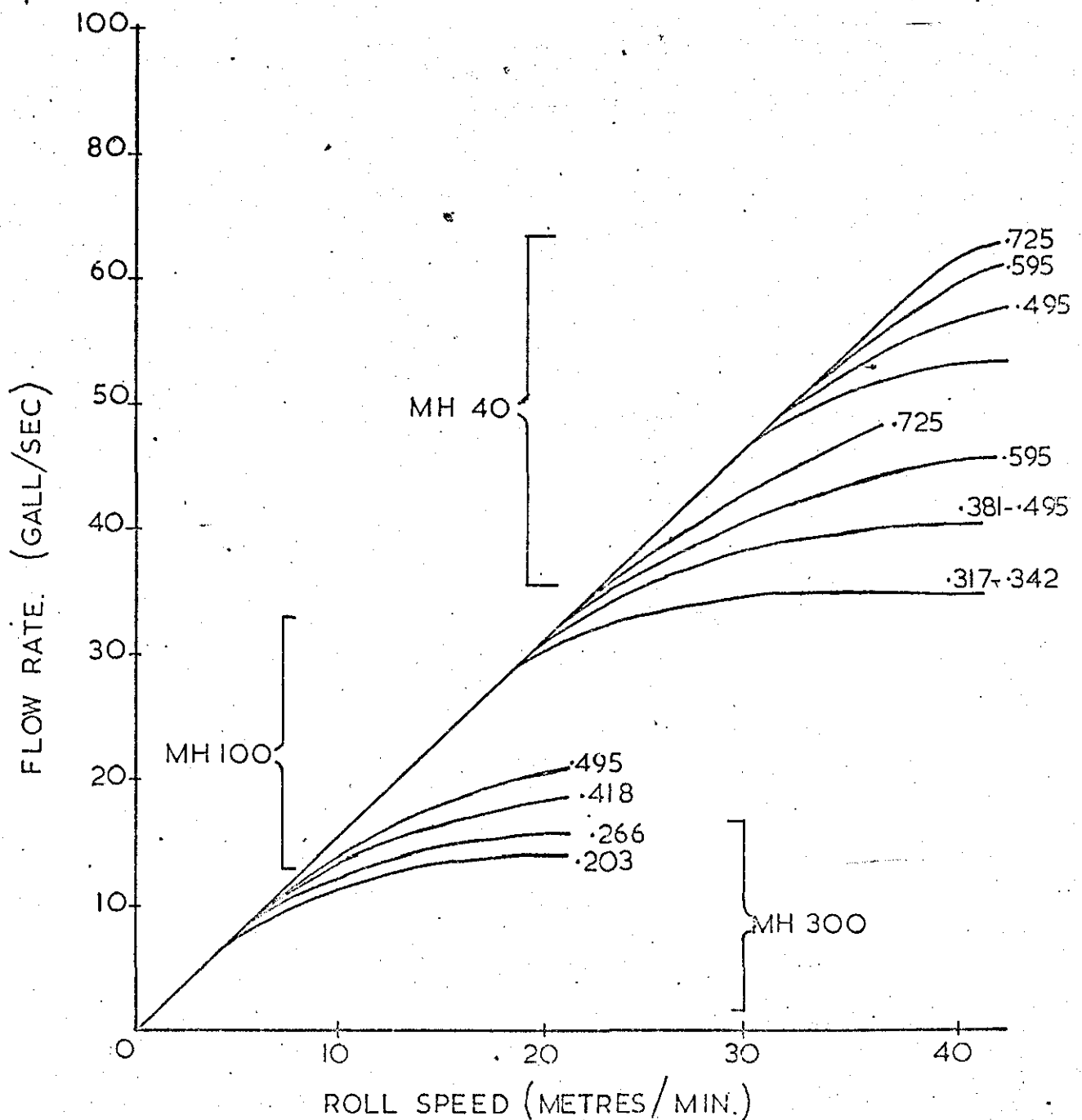
S/N CURVES FOR STRAIN
AGEING AND NON STRAIN
AGEING MATERIALS.

FIG. N° 4.



ZONES OF A SATURATED FEED SYSTEM.

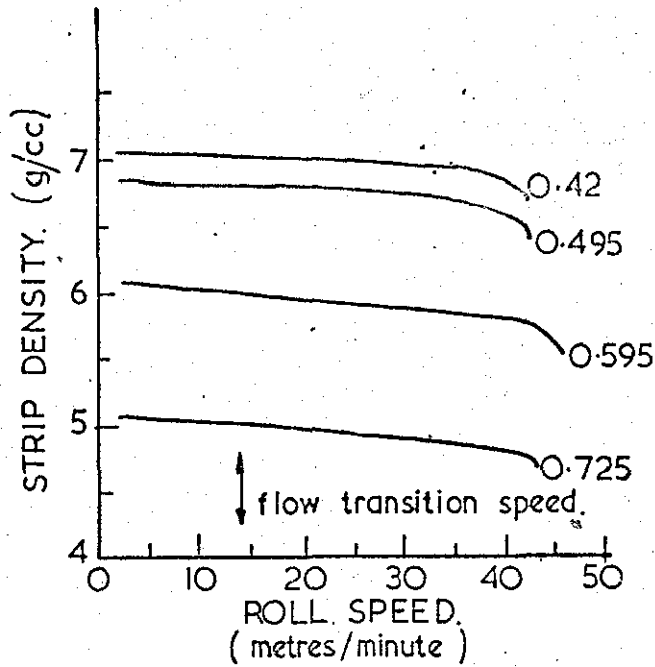
FIG. Nº 5



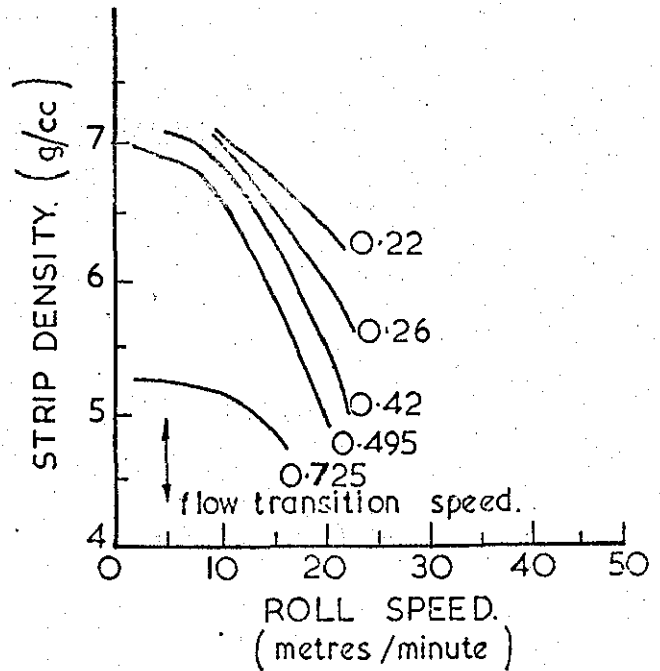
VARIATION IN THE POWDER FLOW RATE WITH ROLL
SPEED DURING ROLLING AND AT THE VARIOUS INDICATED
ROLL GAPS (mms) FOR NH 40 MH 100 & MH 300 POWDERS.

FIG. N° 6

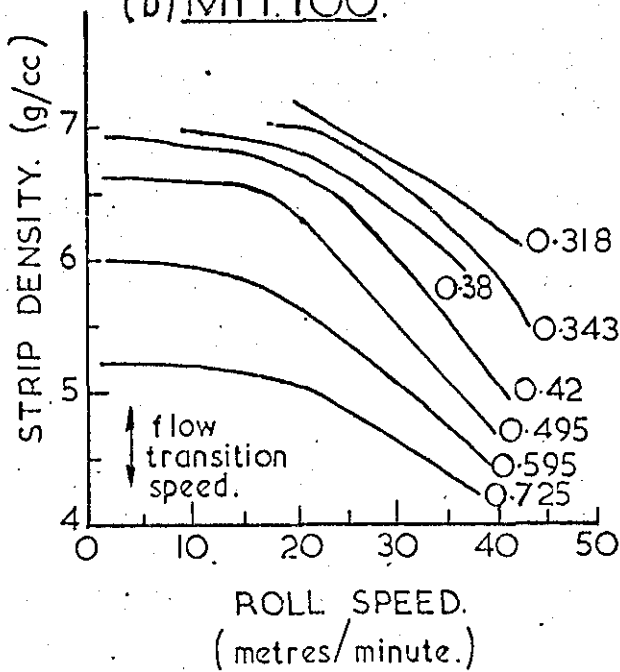
(a) MH. 40.



(c) MH. 300.

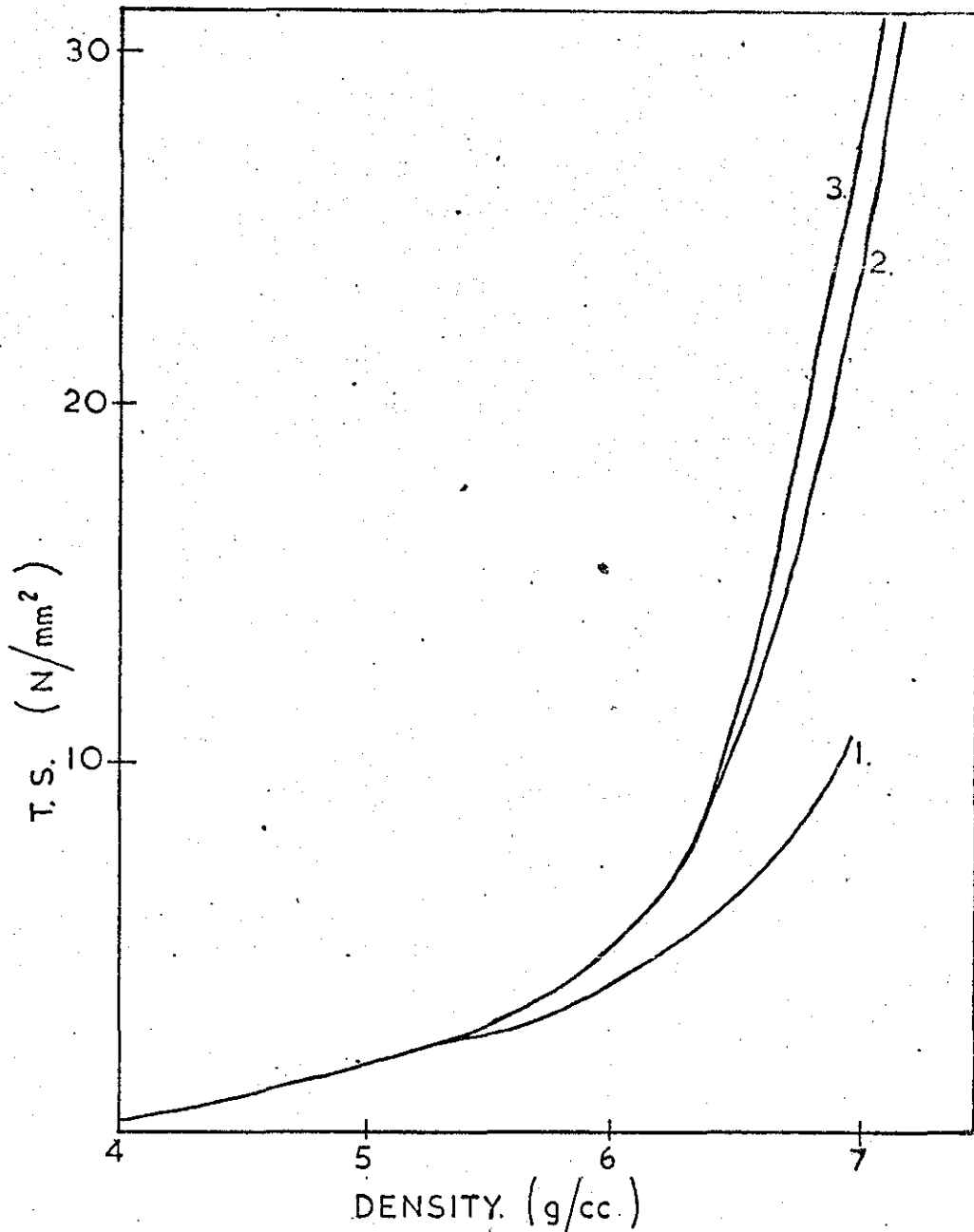


(b) MH. 100.



VARIATION IN STRIP DENSITY WITH ROLL
SPEED FOR MH 40 100 AND 300 AT VARIOUS
ROLL GAPS INDICATED IN MMS.

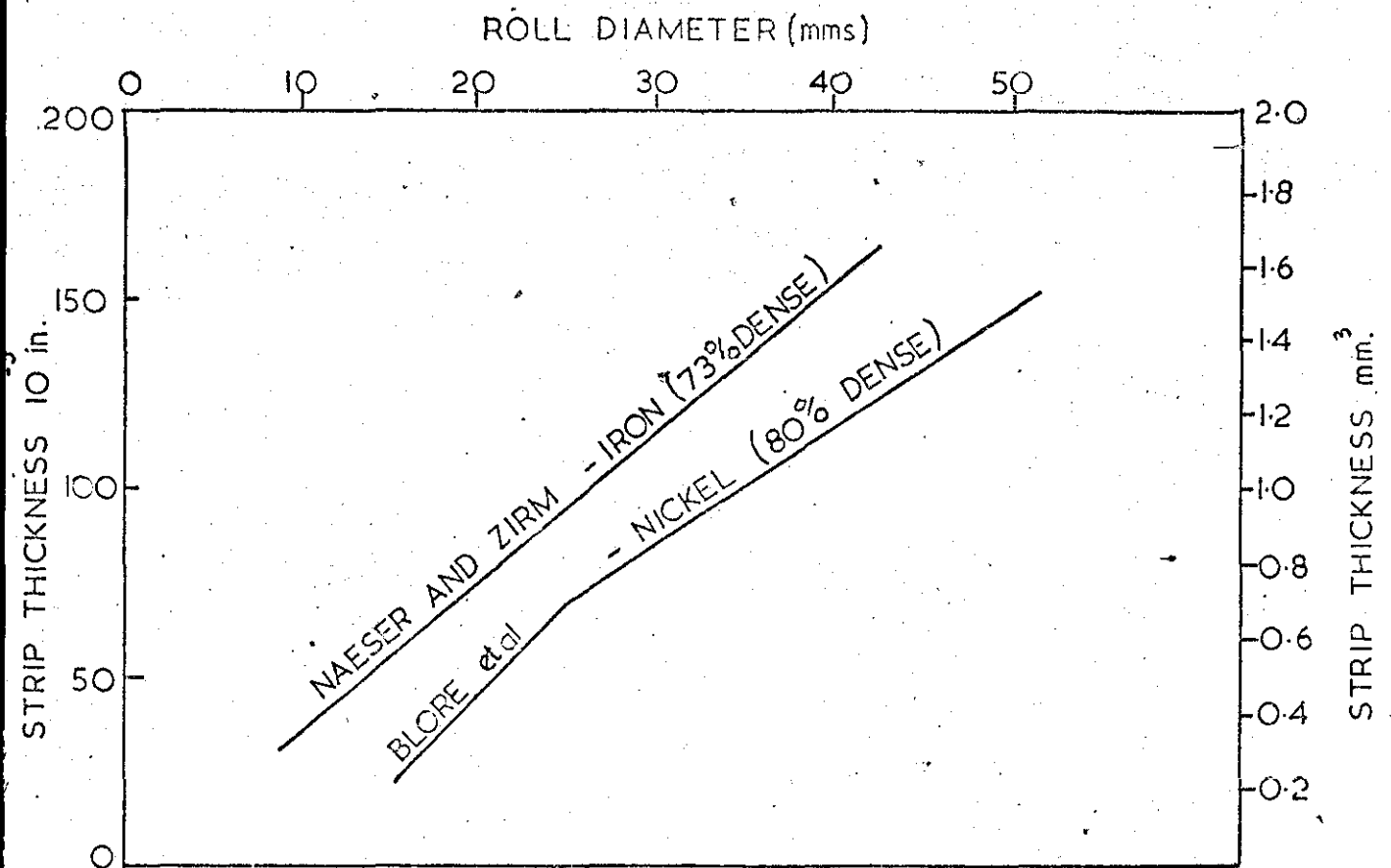
FIG. N° 7



CURVE 1. 30-42 METRES/min ROLL SPEED.
CURVE 2. 15-30 METRES/min ROLL SPEED.
CURVE 3. 0-15 METRES/min ROLL SPEED.

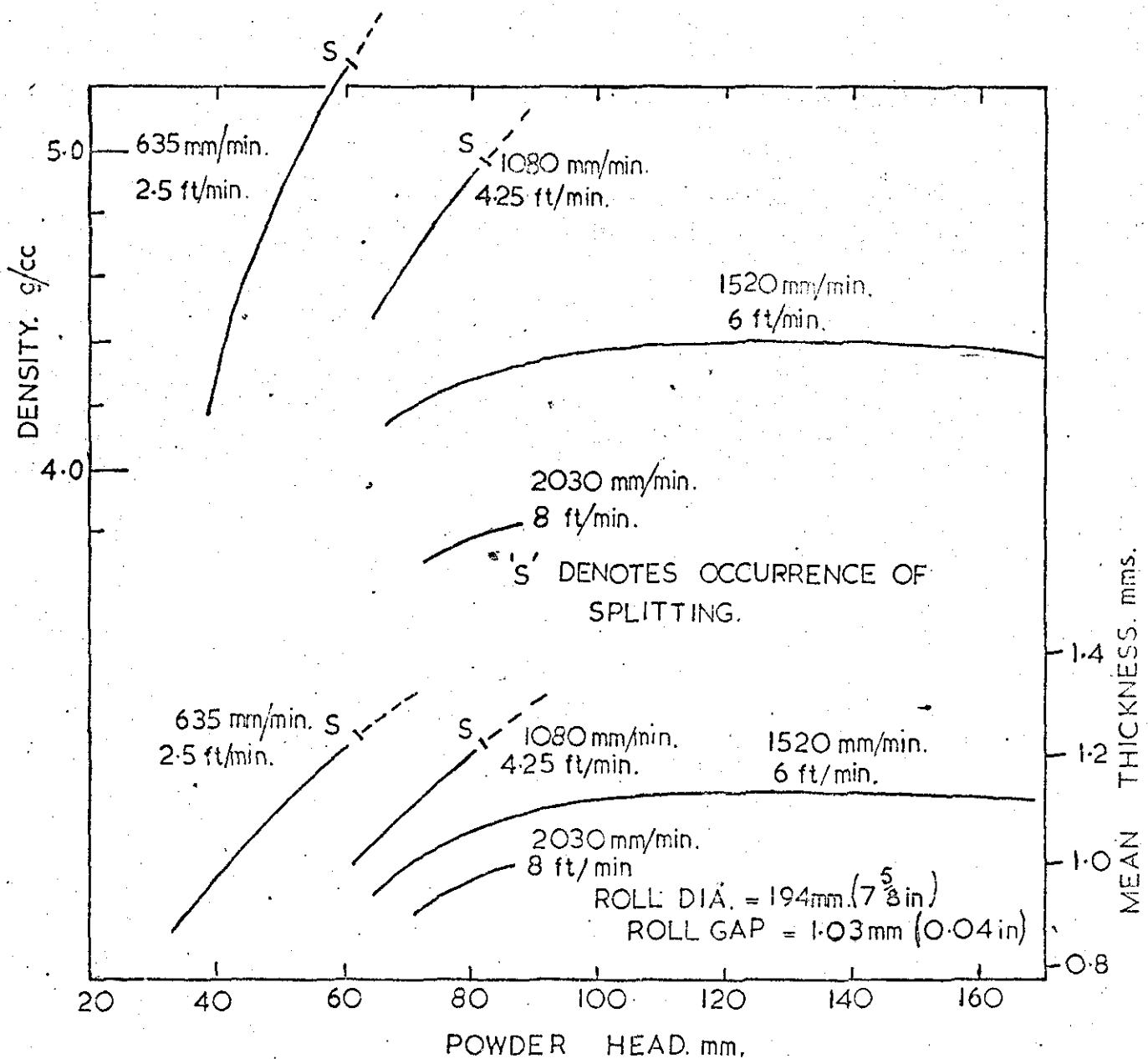
STRIP TENSILE STRENGTH AS
A FUNCTION OF DENSITY.

FIG. N° 8.



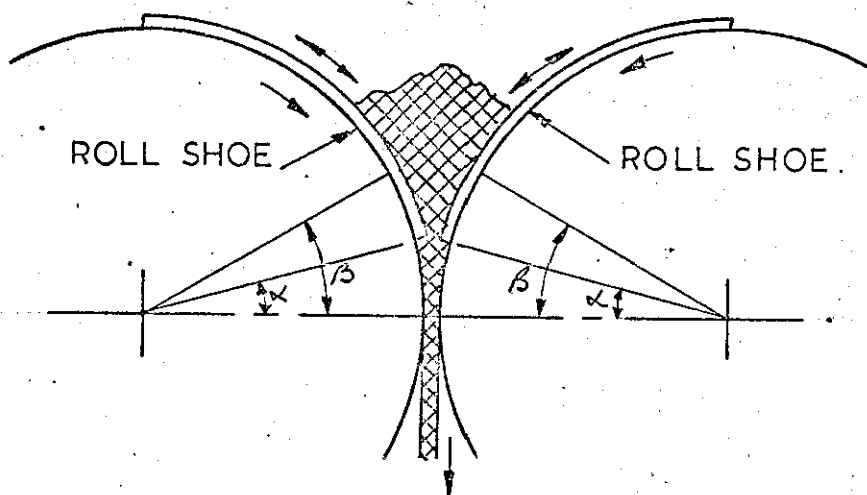
EFFECT OF ROLL DIAMETER ON STRIP
THICKNESS ACCORDING TO NAESER
ZIRM AND BLORE ET AL.

FIG. N° 9.



EFFECT OF POWDER HEAD ON DENSITY
AND THICKNESS OF NICKEL STRIP
ACCORDING TO WORN AND PERKS.

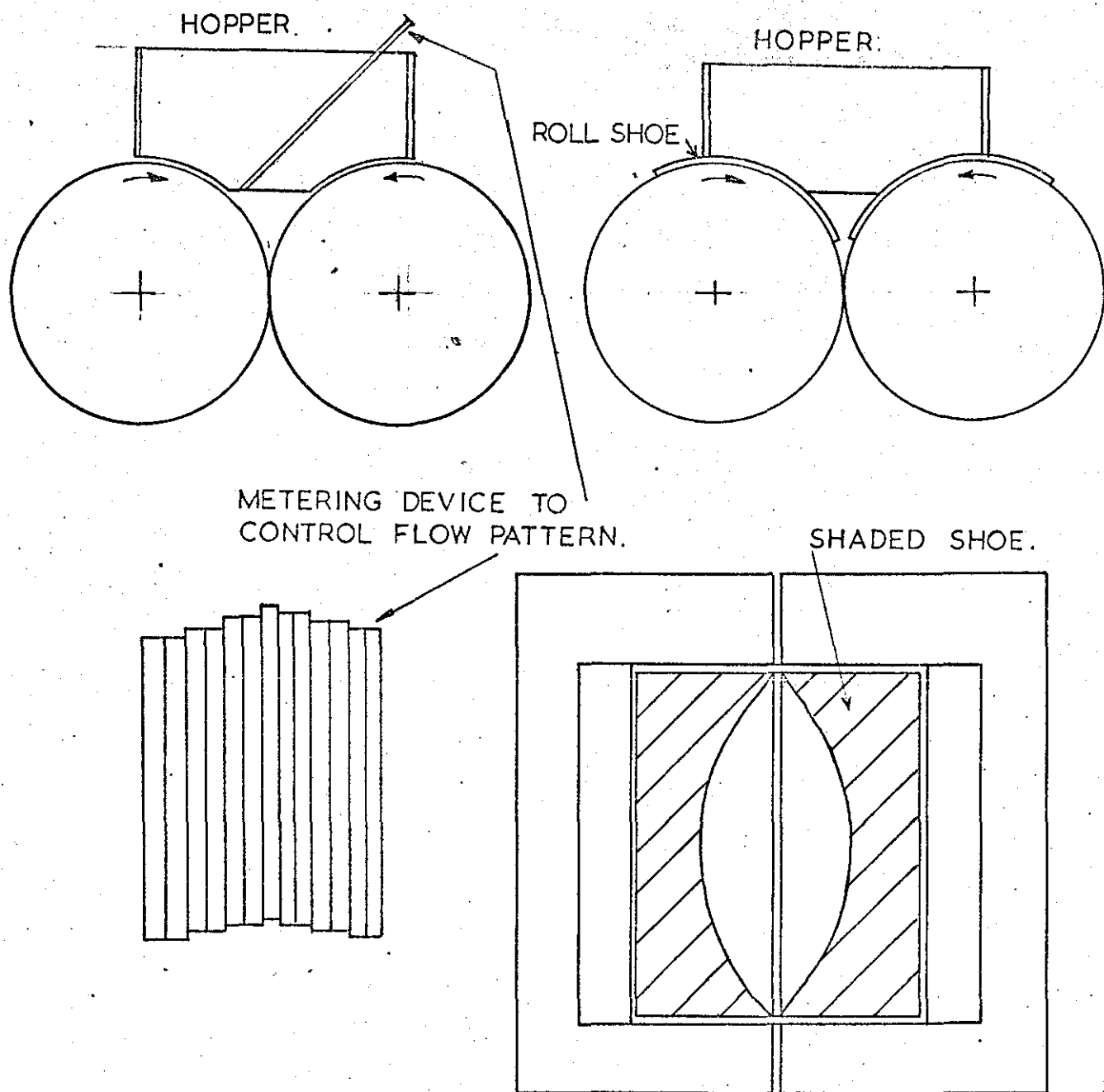
FIG. N° 10.



USE OF ROLL SHOE TO ALTER ANGLE OF BITE FROM β TO α .

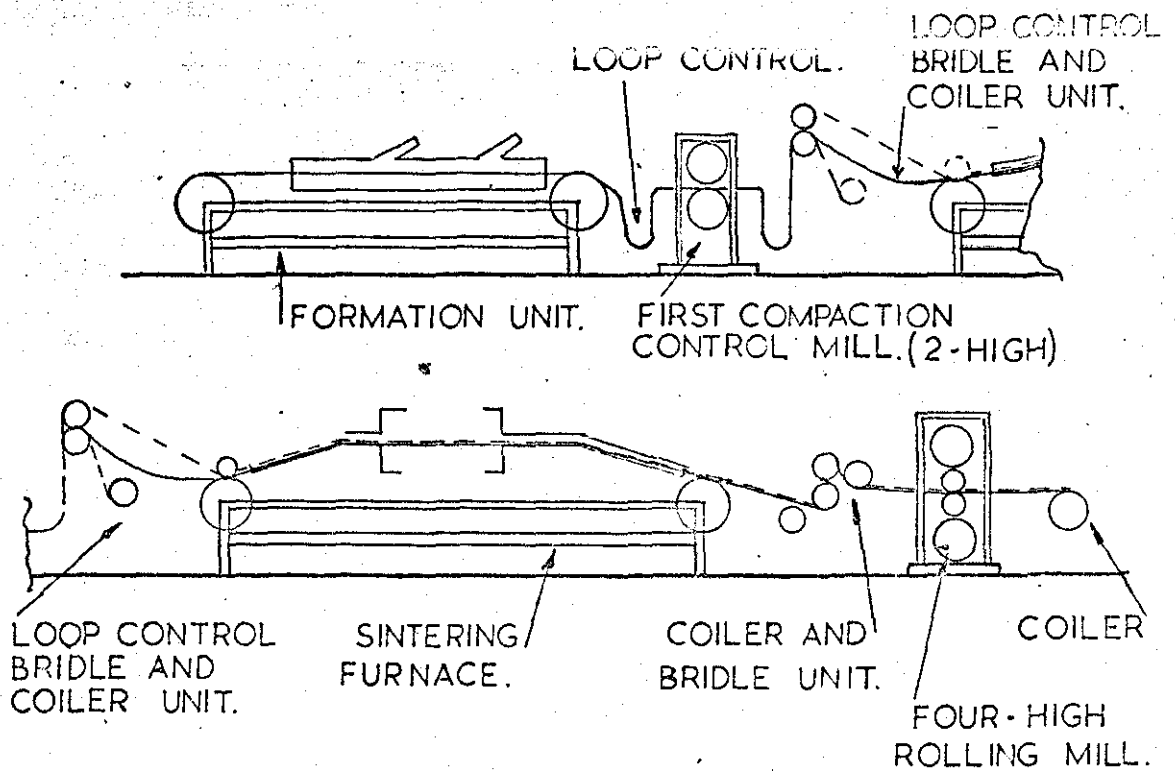
CONTROL OF POWDER FEED
ACCORDING TO FRANSSEN.

FIG N° II.



CONTROL OF POWDER FEED AND
SPREAD ACCORDING TO WORN.

FIG N° 12.



EXPERIMENTAL PLANT FOR THE
PRODUCTION OF THIN STRIP FROM POWDER.

—— FIRST PASSAGE THROUGH PLANT.
- - - SECOND PASSAGE THROUGH PLANT.

FIG. N° 13.

TENSILE PROPERTIES OF STRIP MADE FROM IRON POWDER BY THE SLURRY PROCESS.

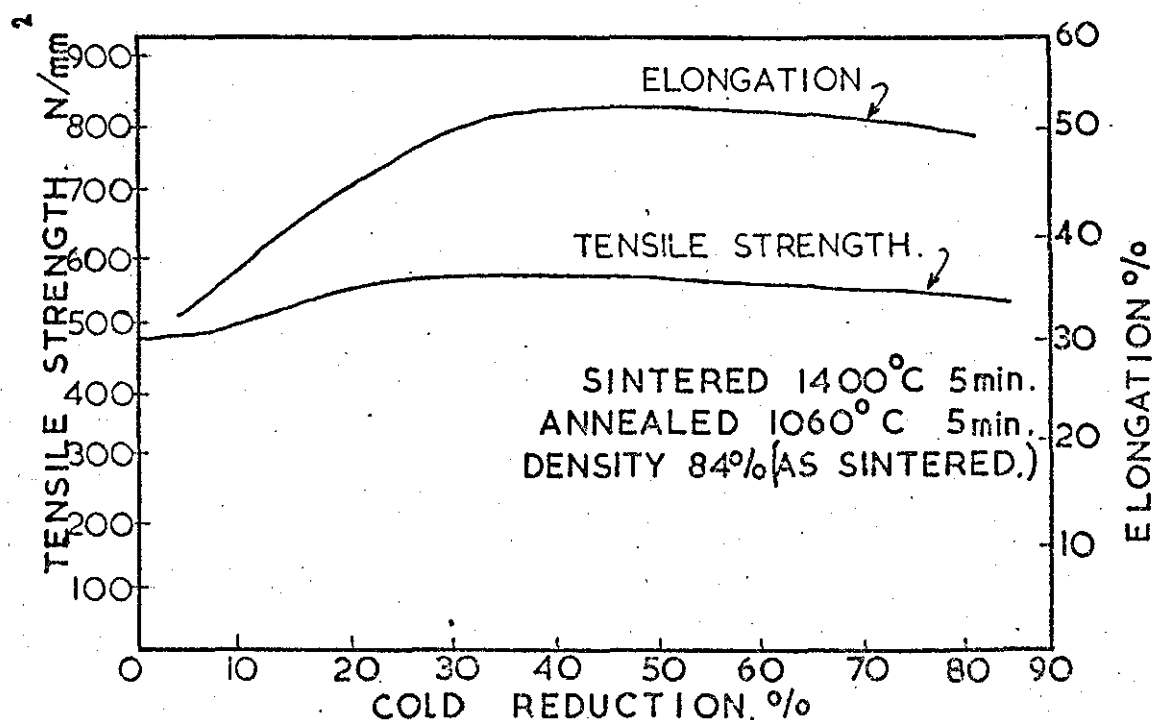
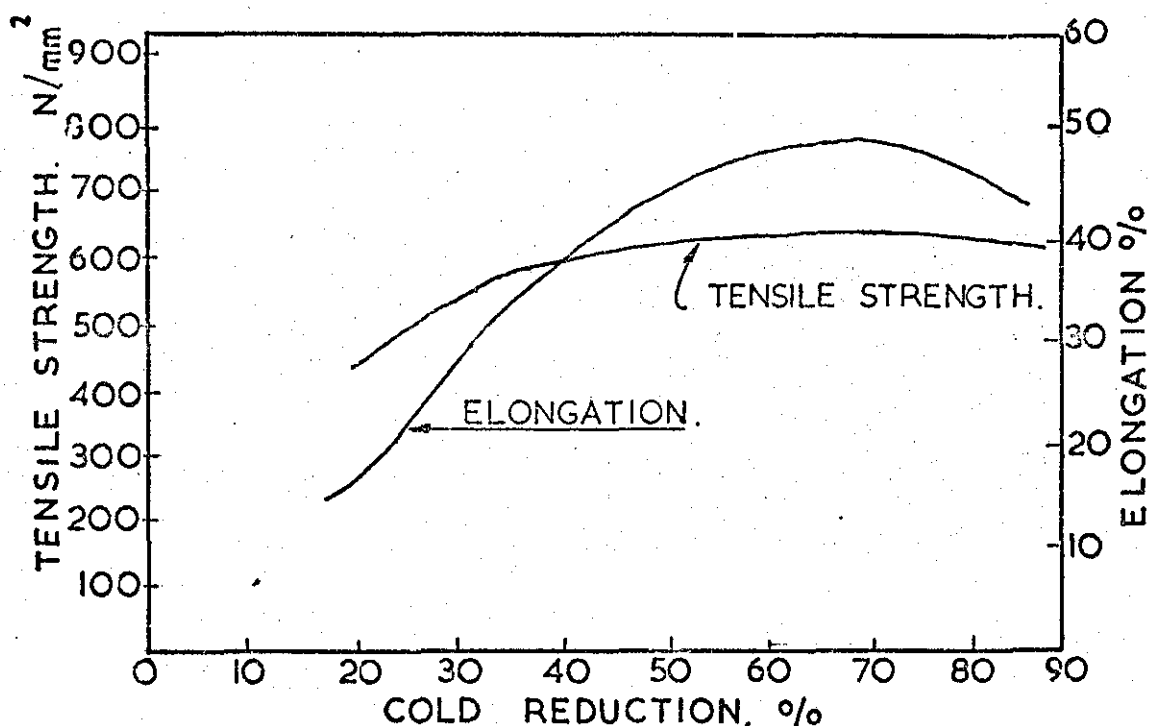
Material	TS N/mm ²	Elongation % (19 m.m. gauge- length.)
Makin 300 PI	294	15
Sintrex Electrolytic - 300 mesh (annealed)	324	22
Woodall-Duckam Peace River Batch 103 (sieved to -300 mesh)	371	24
Conventionally produced general- purpose-quality tinplate (Temper 3)	356/386	20/30

FIG. N° 14.

TENSILE PROPERTIES OF STRIP MADE FROM STAINLESS-STEEL BY THE SLURRY PROCESS.

Material	TS N/mm ²	Elongation % (19 m.m. gauge-length.)
304 L stainless-steel powder, - 300 mesh	600	25
Conventionally produced 304 stainless-steel strip	541	40

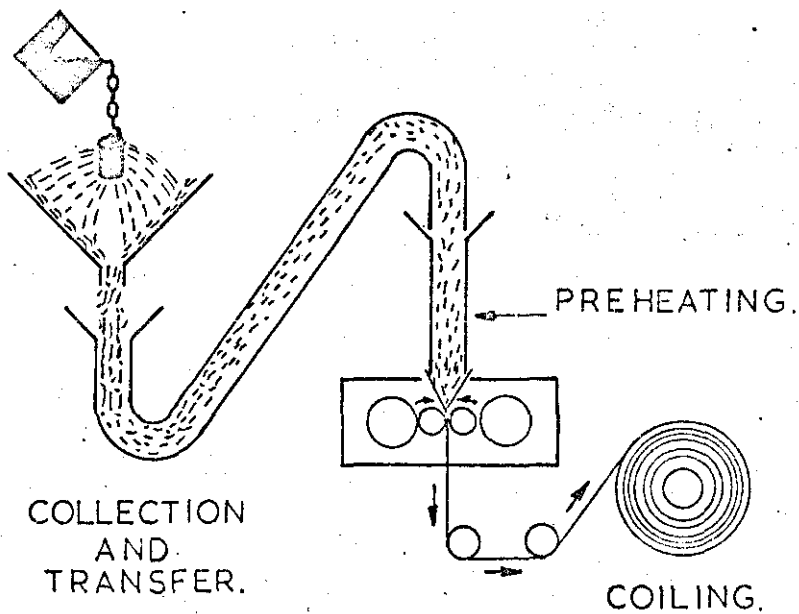
FIG. N° 15



THE EFFECT OF COLD REDUCTION ON
THE TENSILE PROPERTIES OF ANNEALED STRIP

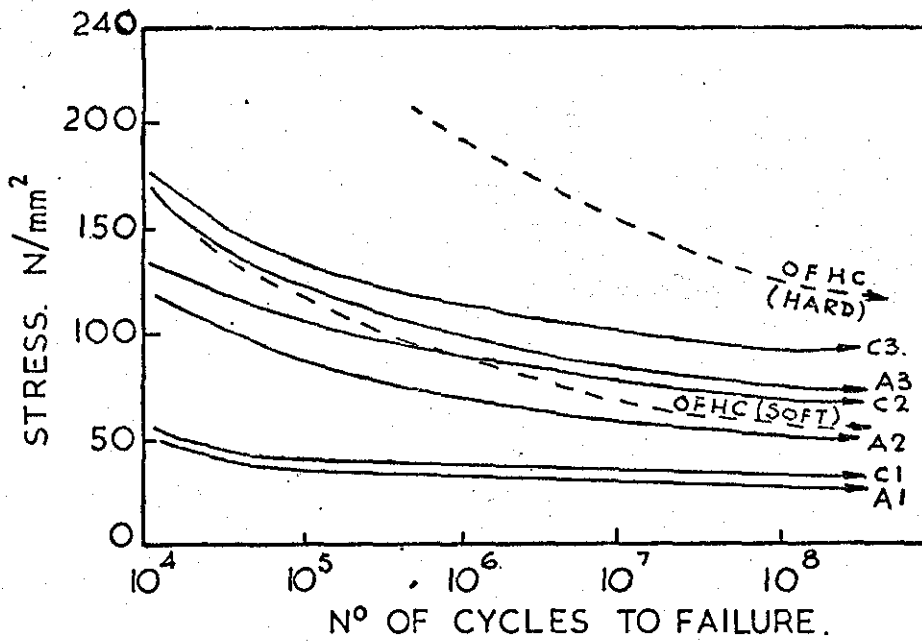
FIG. N° 16.

CASTING.



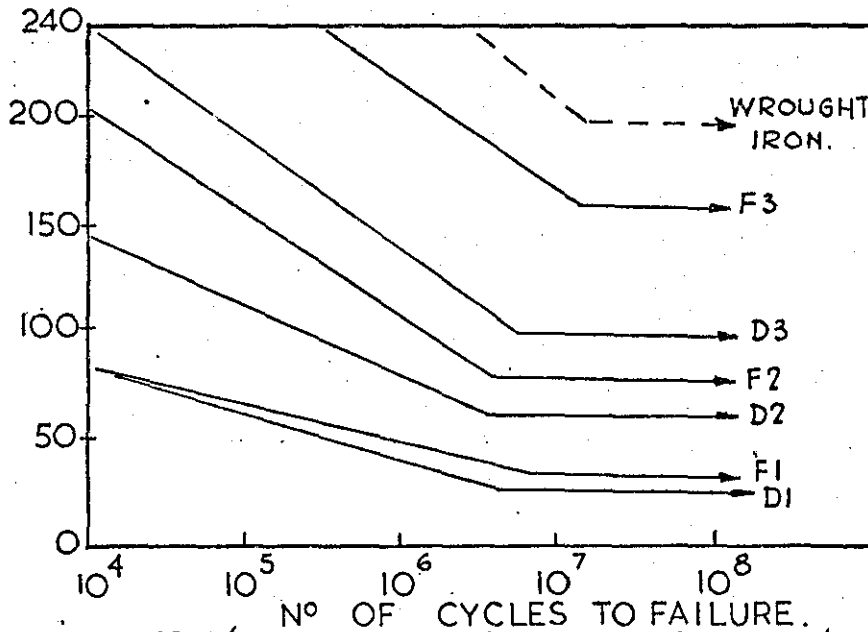
THE COMPACTED SHEET PROCESS
FOR ALUMINIUM ACCORDING TO DAUGHERTY.

FIG. N°17



S/N CURVES FOR
SINTERED COPPER.

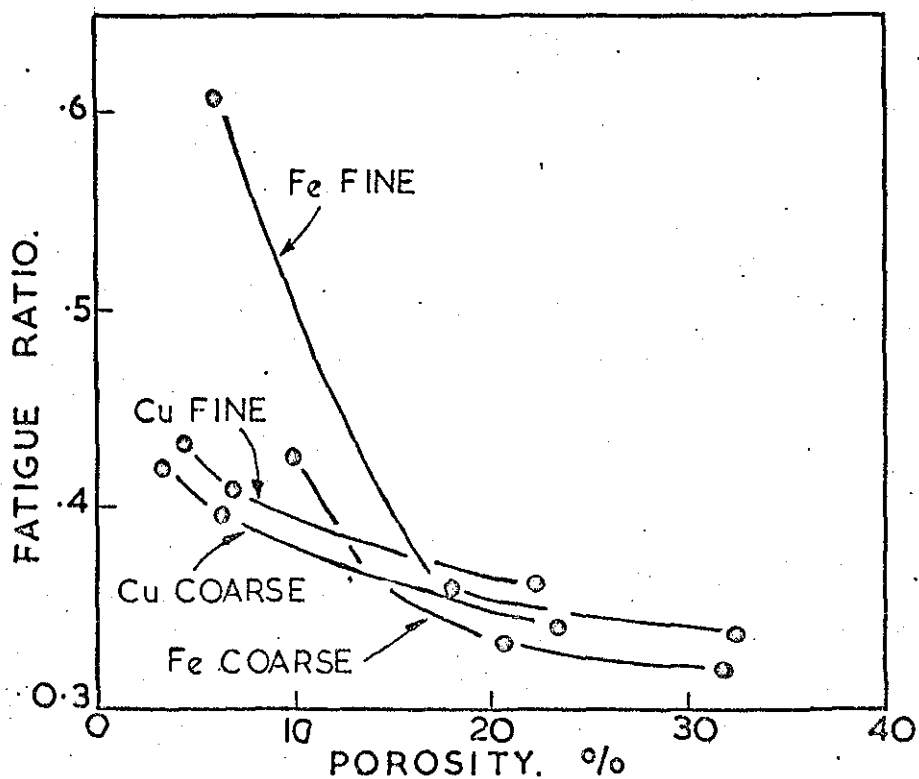
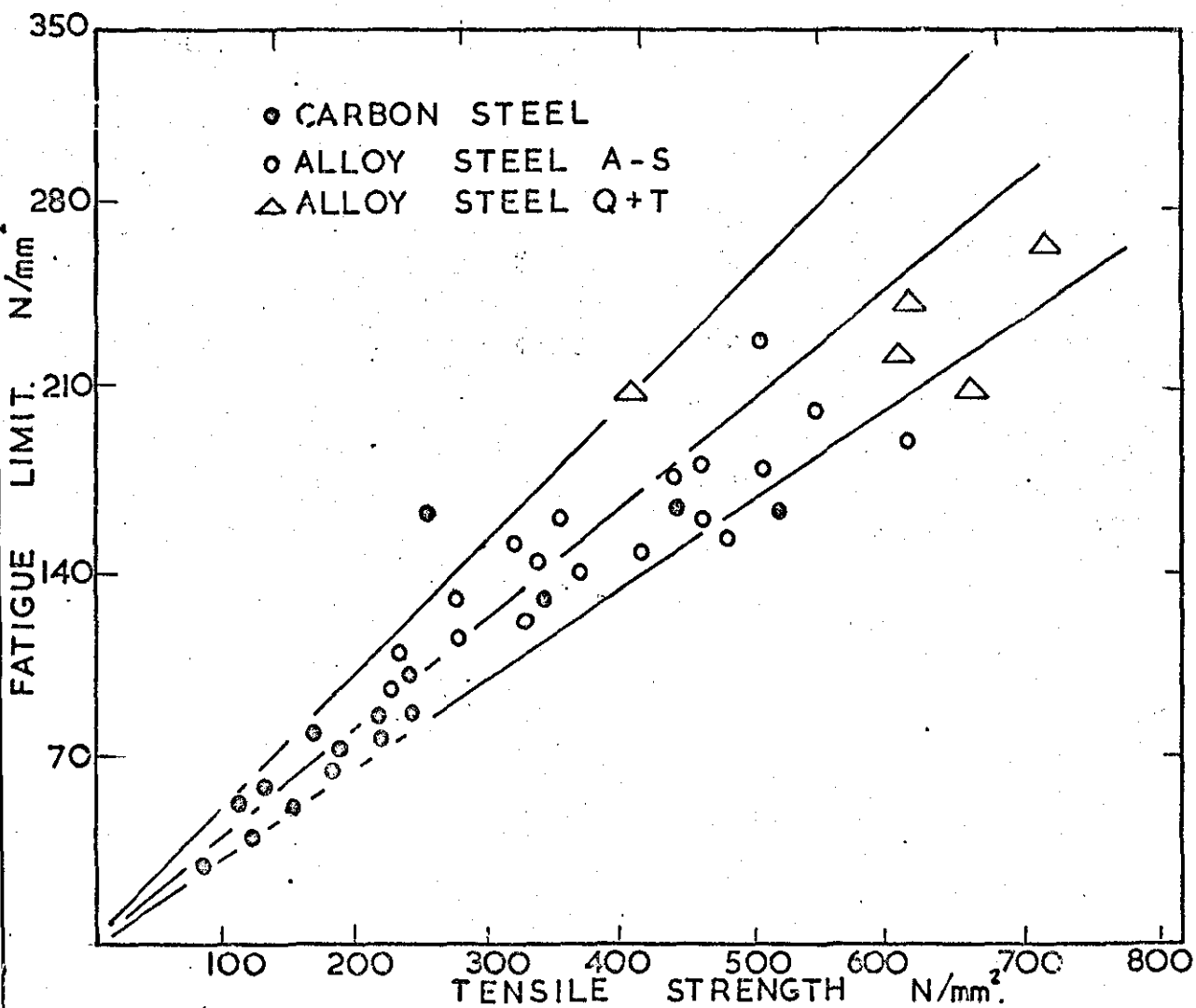
Curve A1 = 23.5% coarse porosity. Curve C1 = 22.5% fine porosity.
Curve A2 = 6.5% coarse porosity. Curve C2 = 7.0% fine porosity.
Curve A3 = 3.5% coarse porosity. Curve C3 = 4.5% fine porosity.



S/N CURVES FOR
SINTERED IRON.

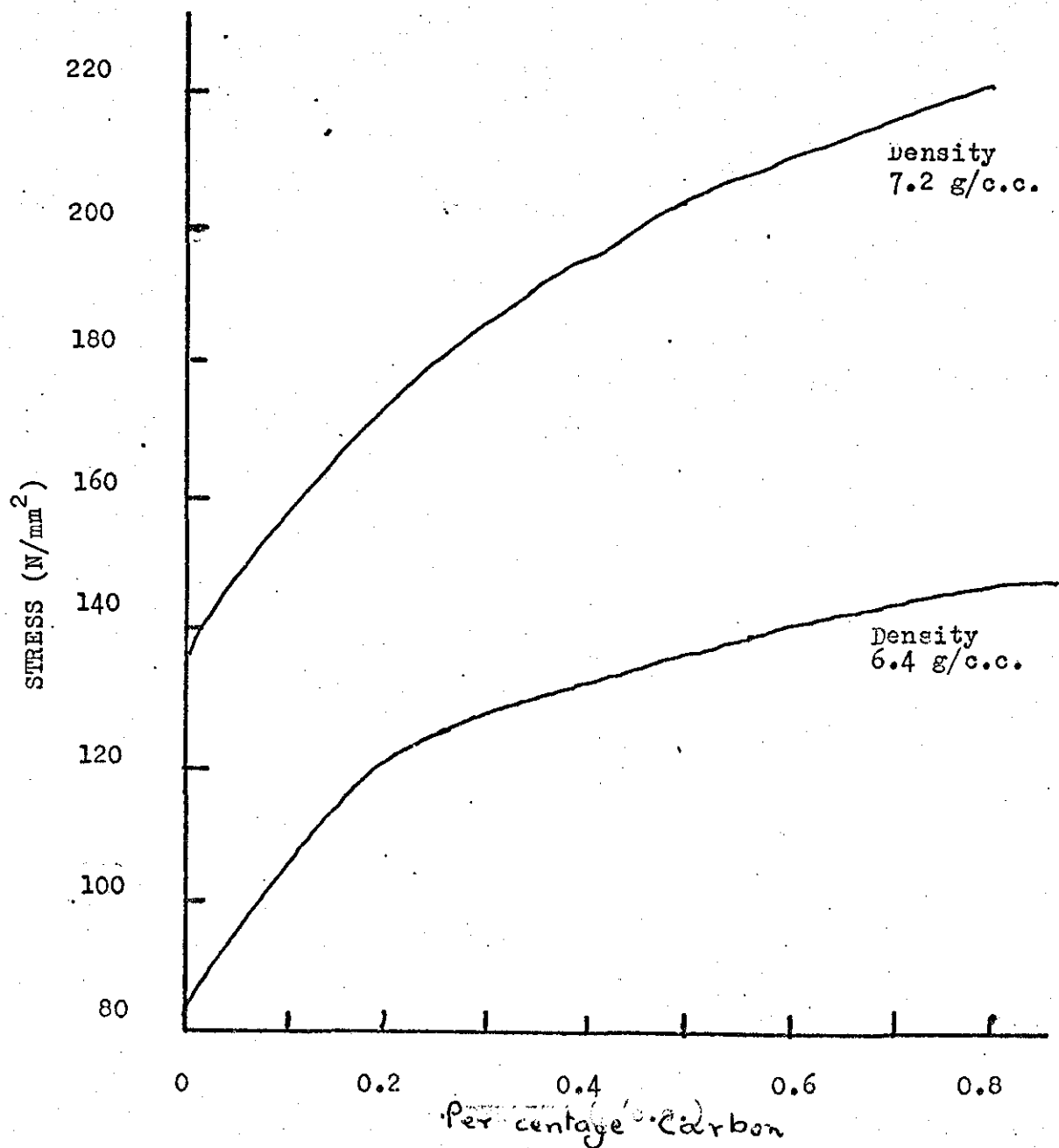
Curve D1 = 32.0% coarse porosity. Curve F1 = 32.5% fine porosity.
Curve D2 = 20.8% coarse porosity. Curve F2 = 18.3% fine porosity.
Curve D3 = 10.0% Coarse porosity. Curve F3 = 6.0% fine porosity.

FIG. N° 18.



THE EFFECT OF TENSILE STRENGTH AND POROSITY
UPON THE FATIGUE RATIO

FIG N^o 19



THE EFFECT OF CARBON UPON THE FATIGUE LIMIT
OF SINTERED IRON.

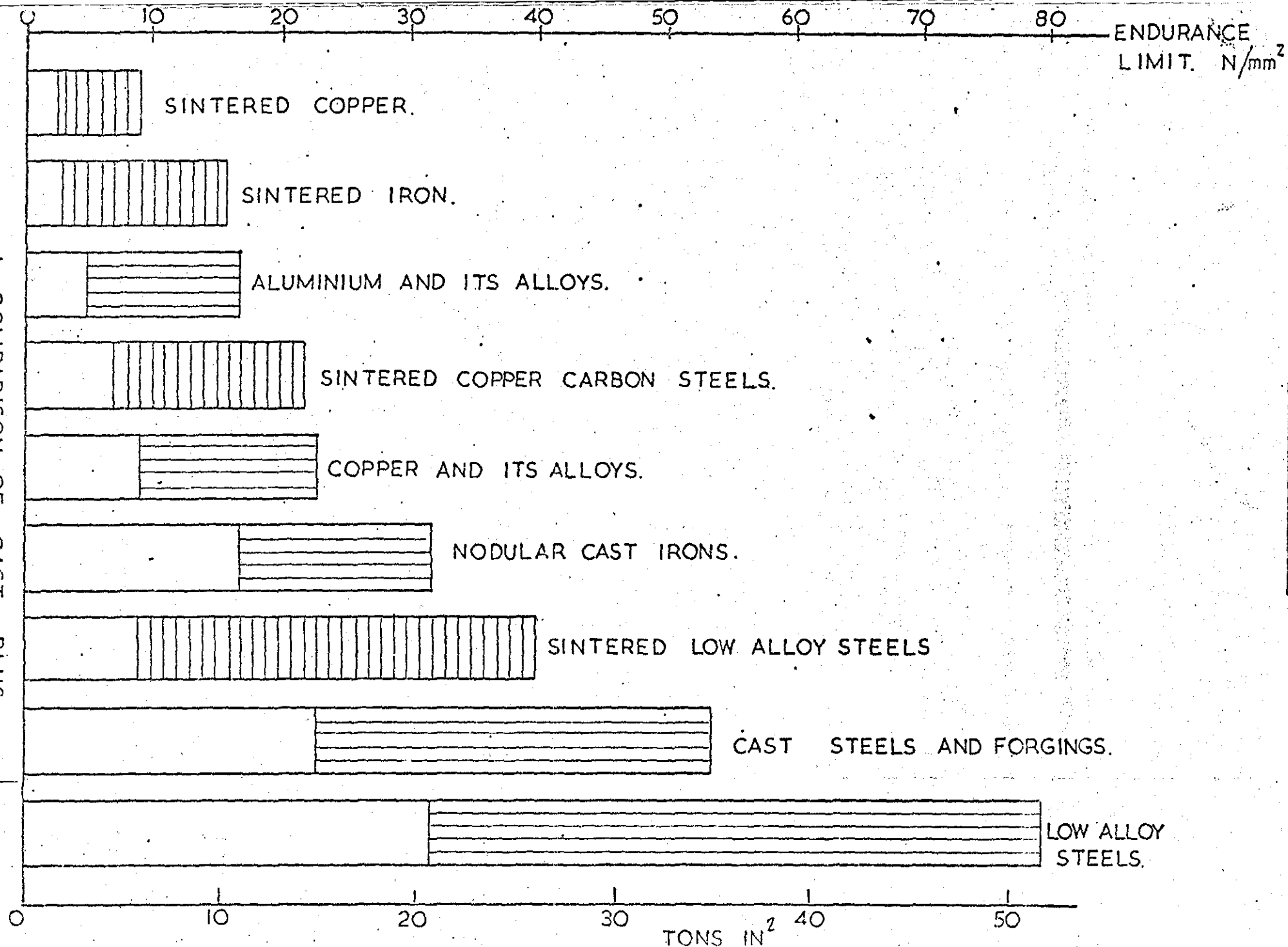
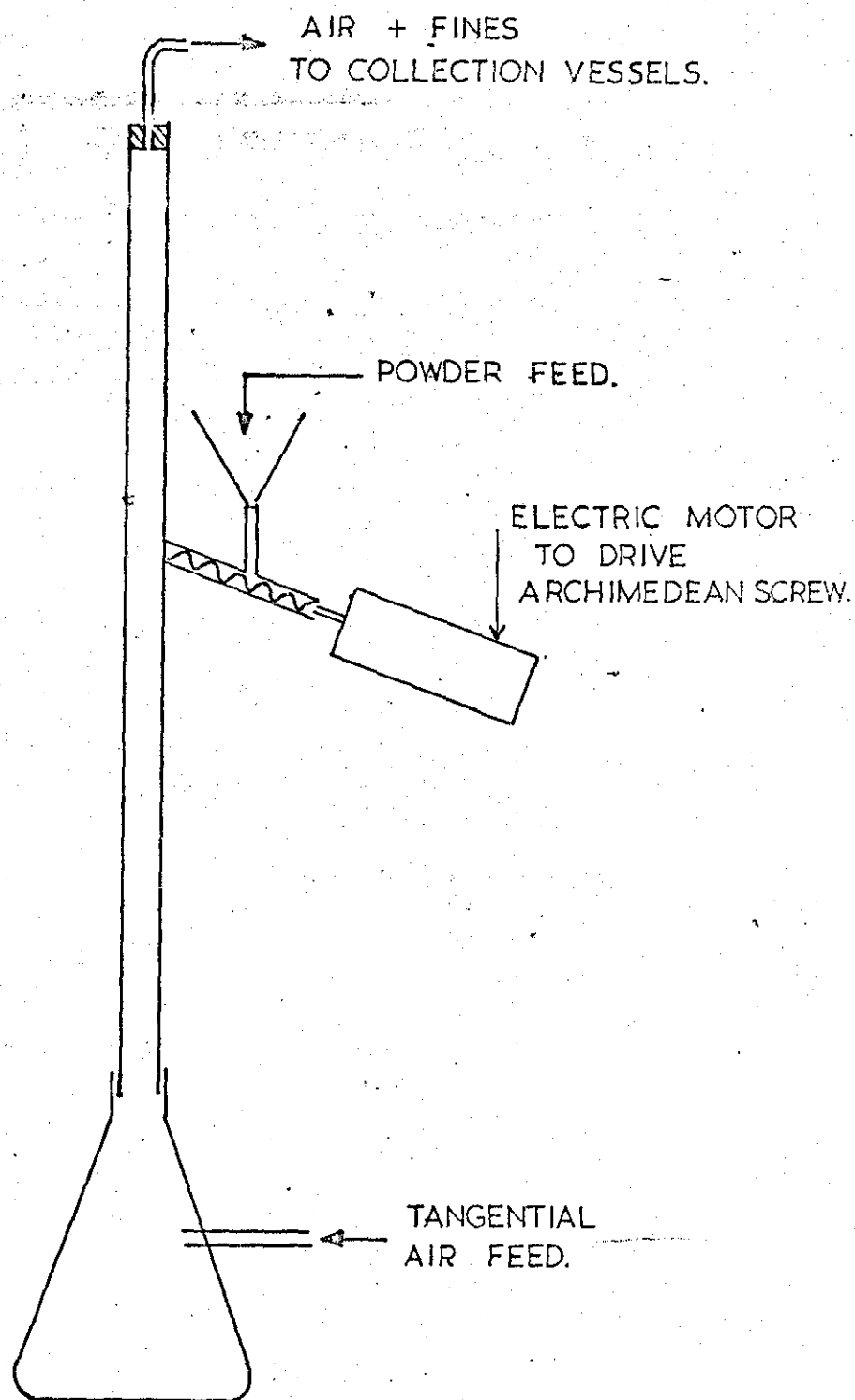


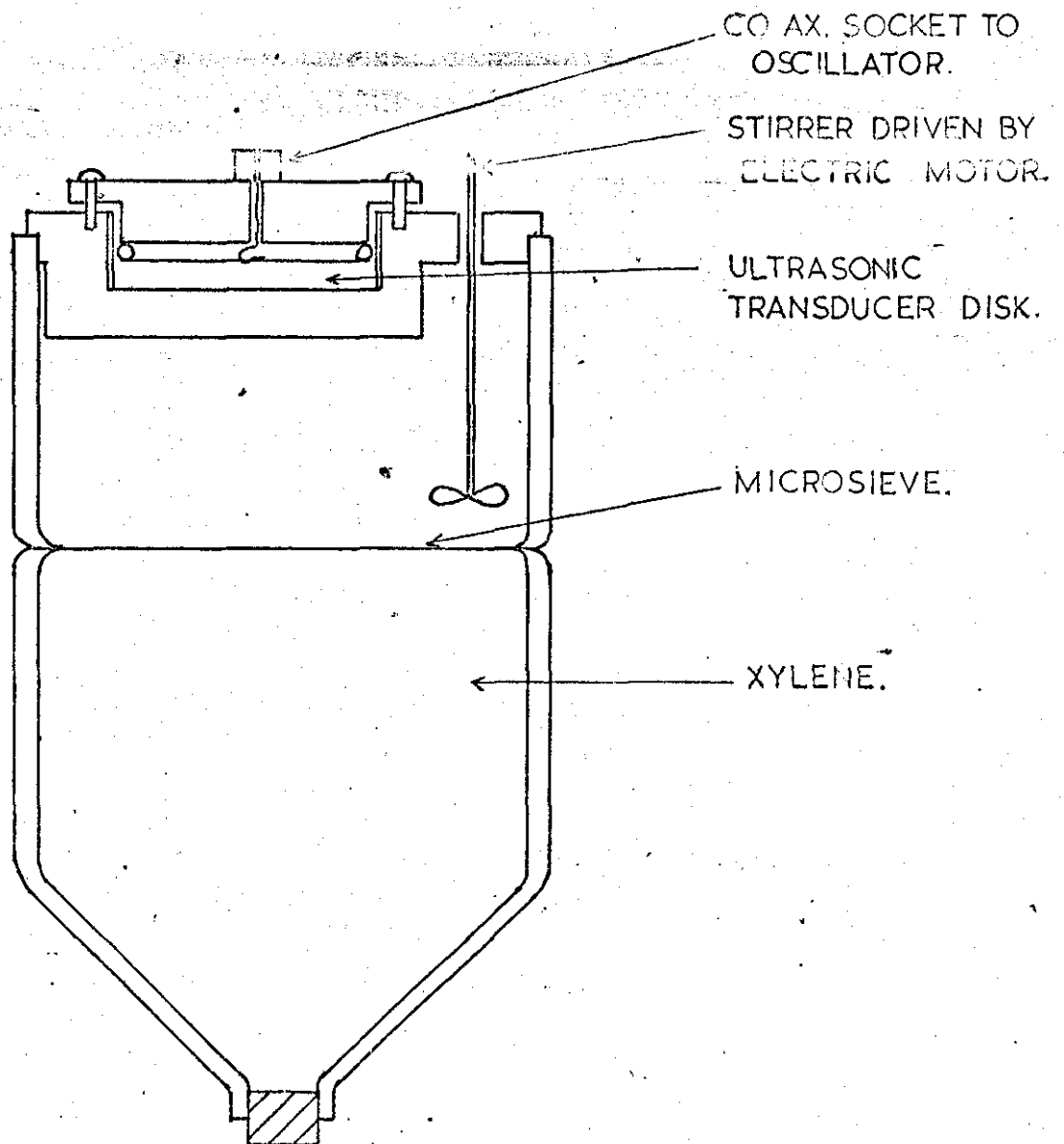
FIG No 20.

FIG. N° 21.



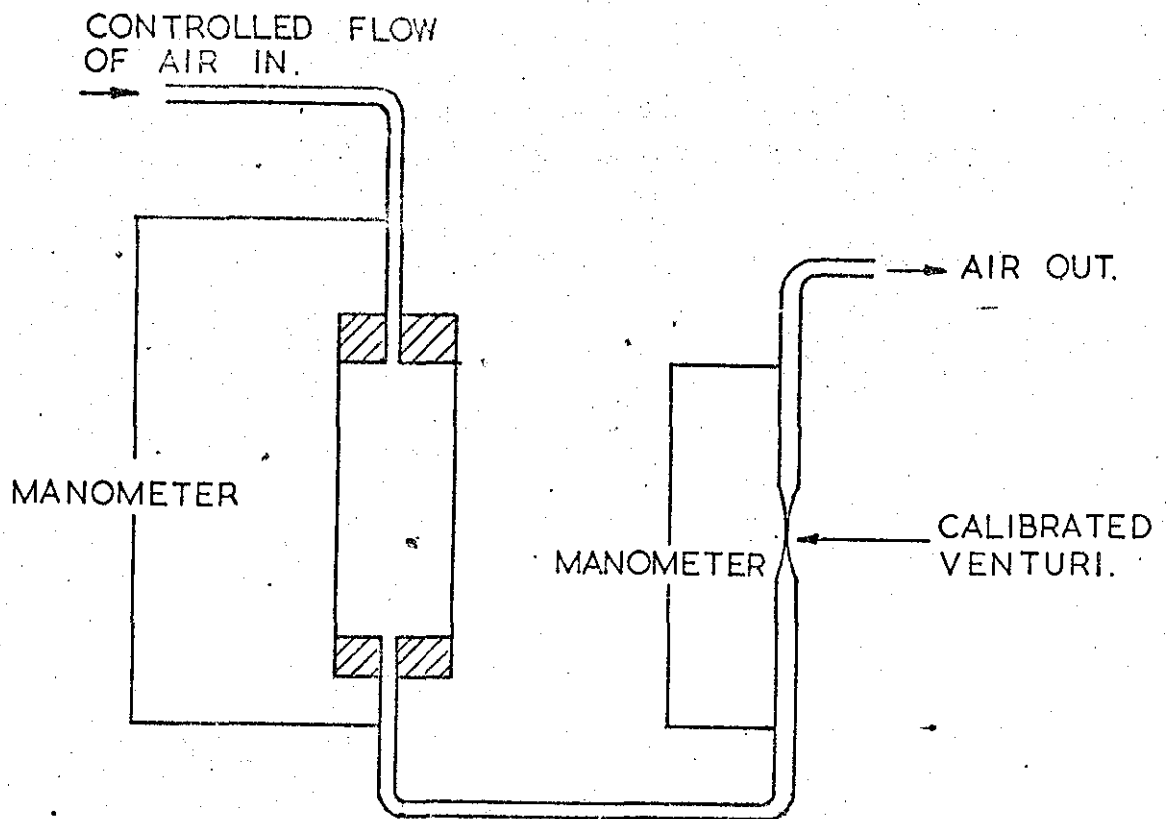
THE AIR ELUTRIATION COLUMN

FIG. N° 22.



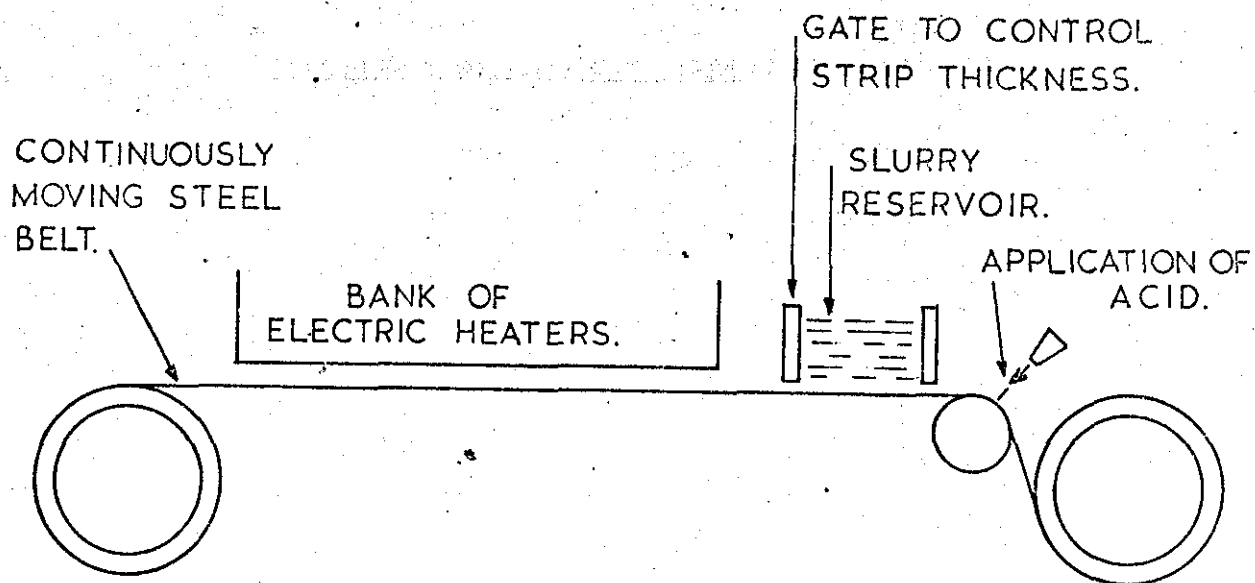
ULTRASONIC MICROSIEVING
APPARATUS.

FIG. N° 23.



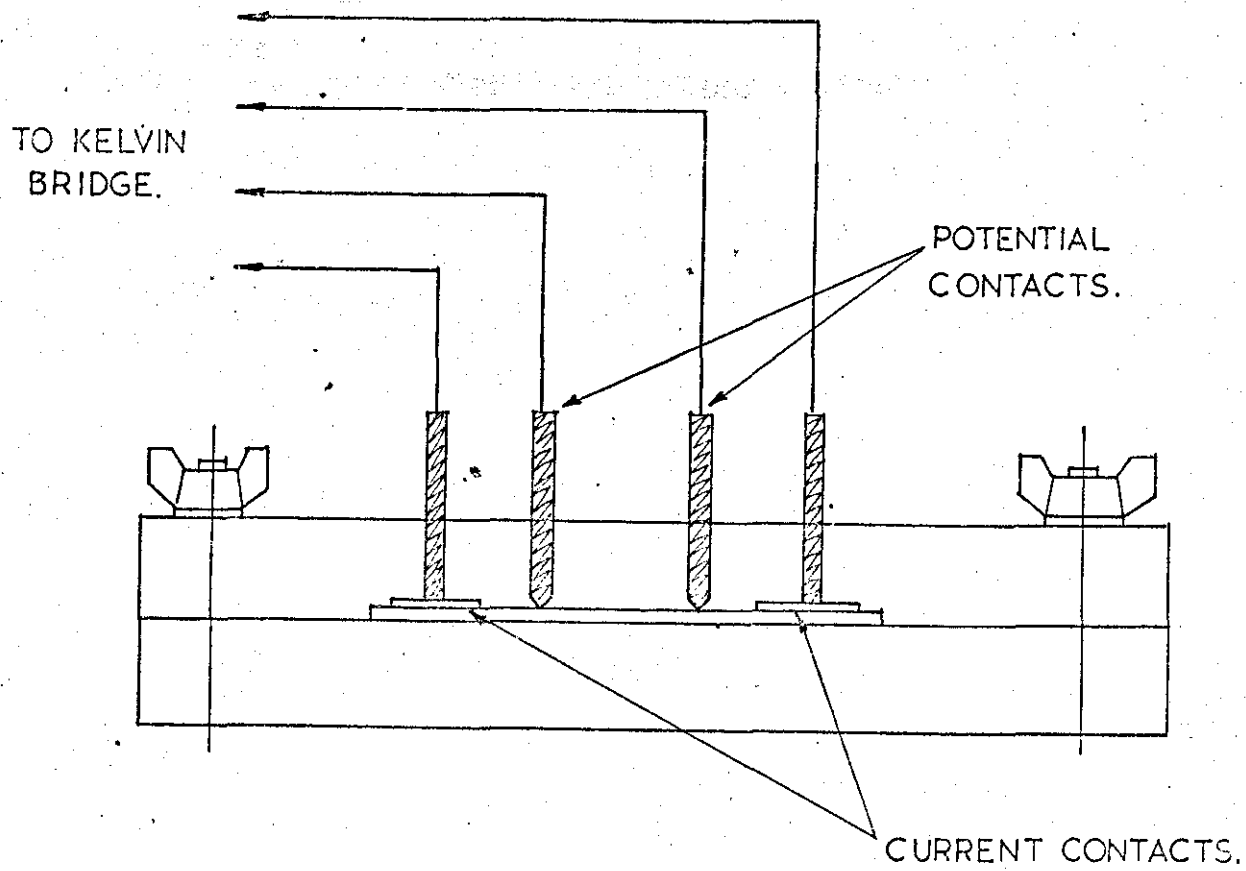
THE LEA AND NURSE APPARATUS.

FIG. N° 24.



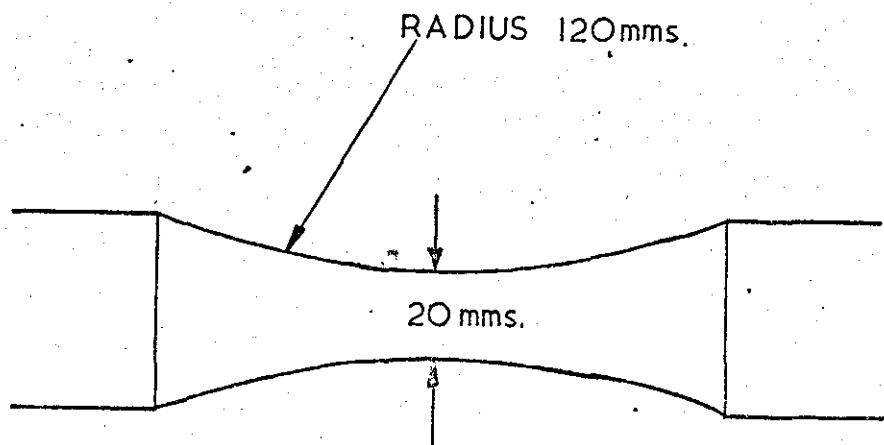
B.I.S.R.A. PILOT PLANT FOR
'FLEXISTRIP' PREPARATION.

FIG. N° 25.



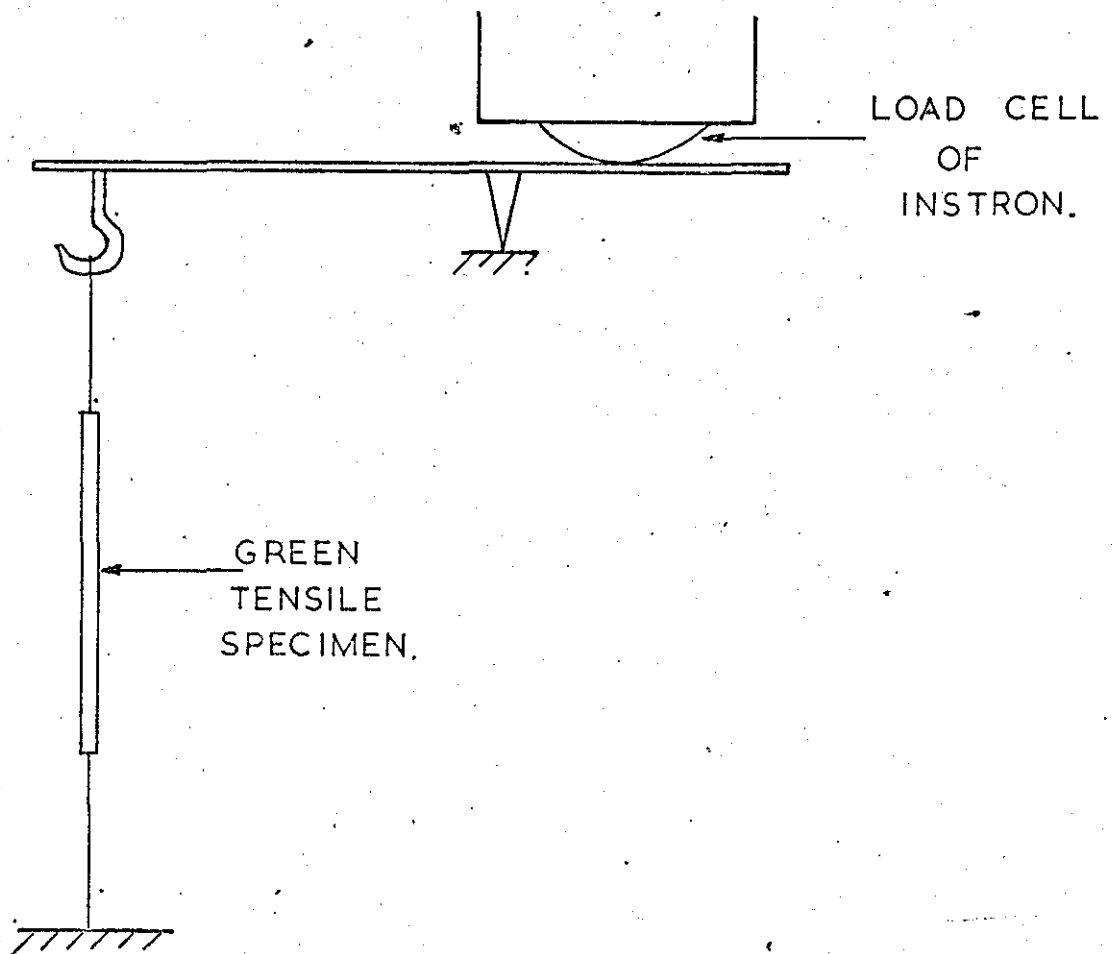
RESISTIVITY APPARATUS.

FIG. N° 26.



SHAPE OF TENSILE SPECIMENS
FOR DETERMINING GREEN STRENGTH.

FIG. N° 27.



ARRANGEMENT FOR DETERMINING
GREEN STRENGTH.

FIG. NO. 28.



MAG.
x200



MAG.
x200.

'STEREOSCAN' PHOTOGRAPHS OF J.J.M. 300
AND WOODALL DUCKAM POWDERS.

FIG. NO. 29



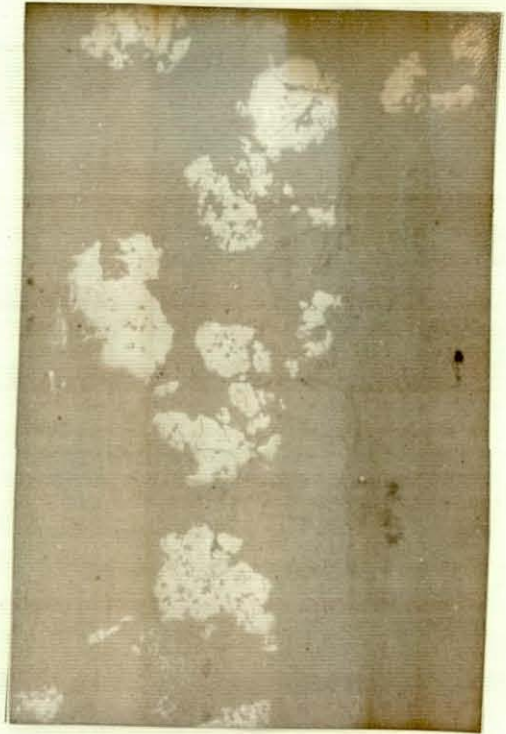
MAG.
x 500

'STEREOSCAN' PHOTOGRAPH OF
SINTREX ELECTROLYTIC POWDER.

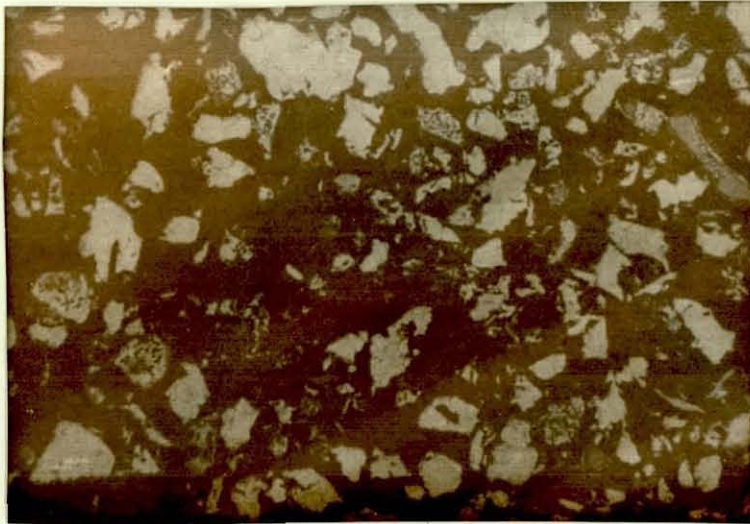
FIG. NO. 30.



x 50



x 100

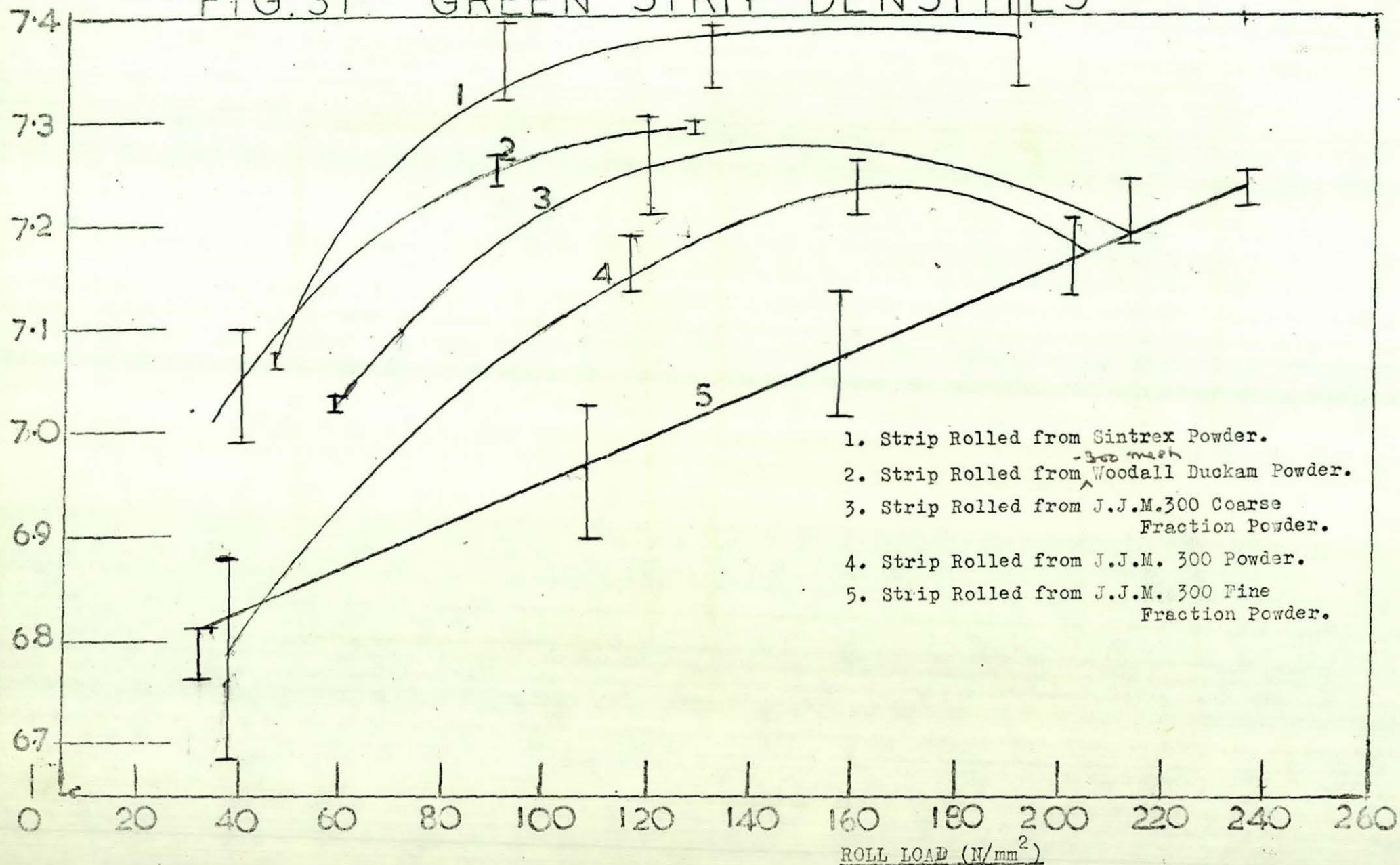


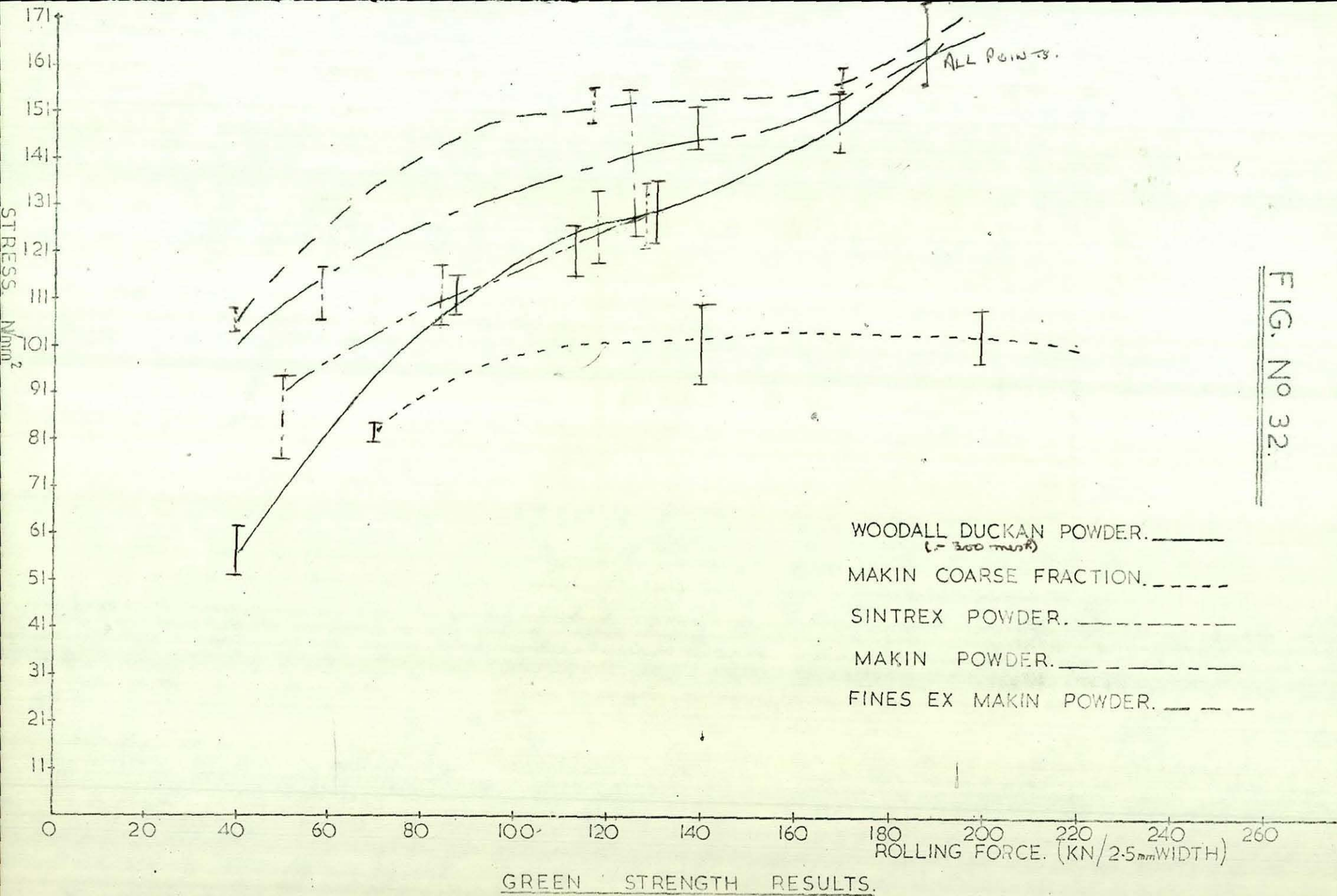
x 50

MICROSTRUCTURES OF SINTREX,
WOODALL DUCKAM AND MAKIN
POWDERS.

DENSITY
(g/cc)

FIG. 31 GREEN STRIP DENSITIES





RESISTIVITY
OHMS-C.M.

↑ S rolled at 95 kN/25 mm $\approx 2-2.6$ ohm-cm. WD = WOODALL DUCKAM PDR. (-300 mesh)
MP = AS RECEIVED NAKIN PDR.
S rolled at 130 kN/25 mm $\approx 5.0-8.3$ ohm-cm. MF = FINE ELUTRIATED FRACTION.
MC = COARSE ELUTRIATED FRACTION.
S = SINTREX ELECTROLYTIC POWDER.

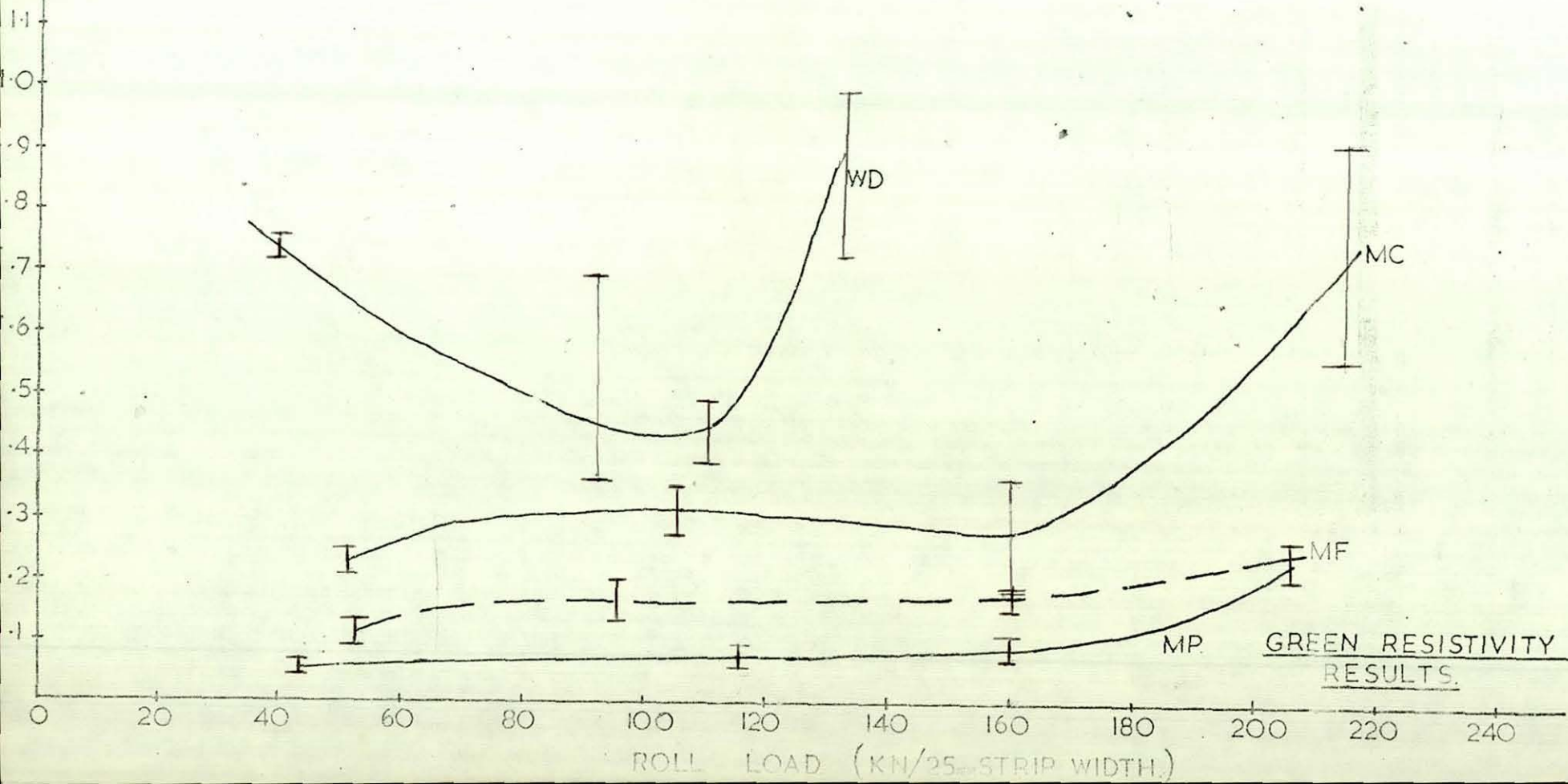


FIG. No 33

FIG. No 34.

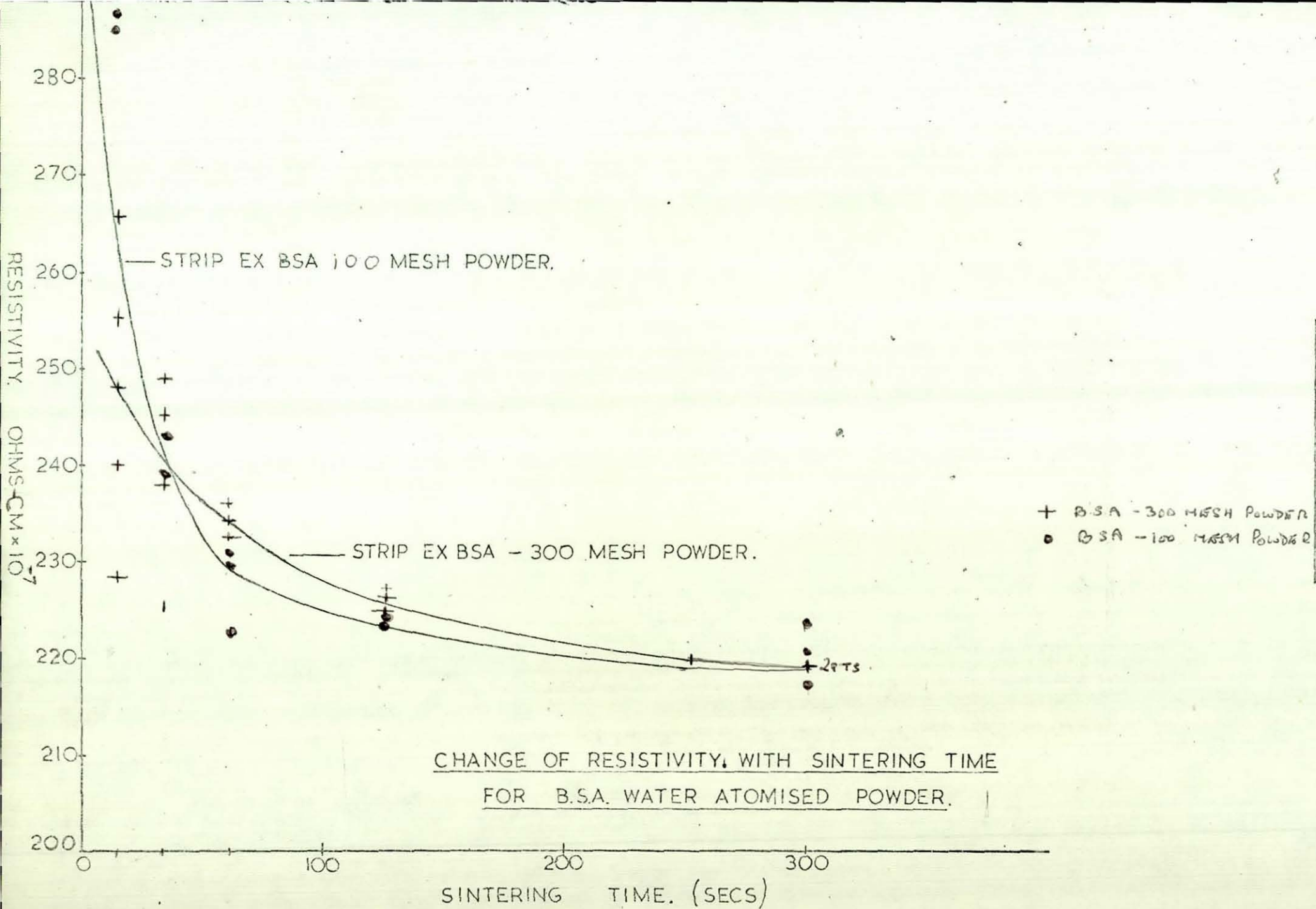


FIG. N° 35.

CHANGE IN RESISTIVITY WITH SINTERING
FOR VARIOUS POWDERS AT A N° OF ROLL LOADS.

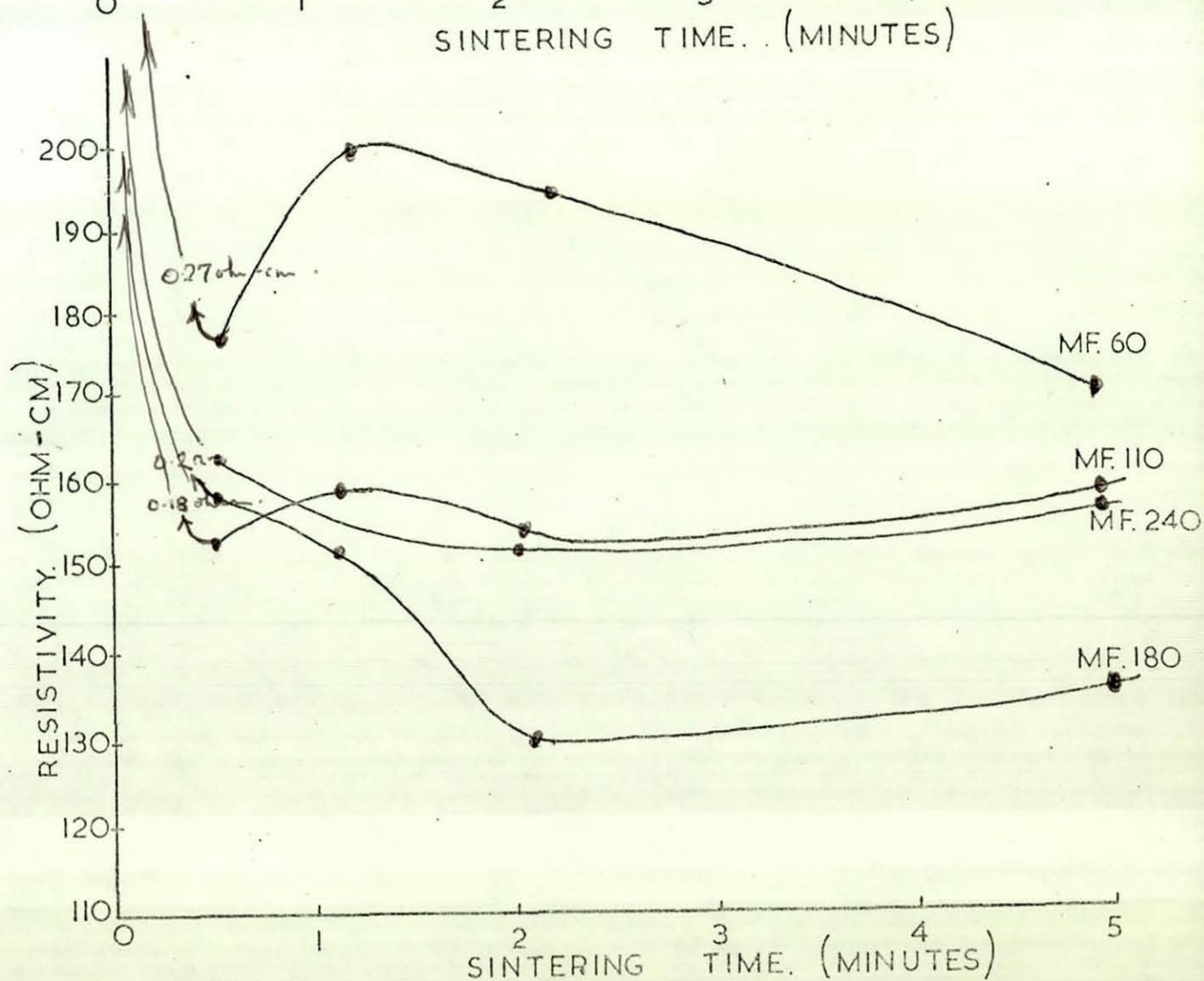
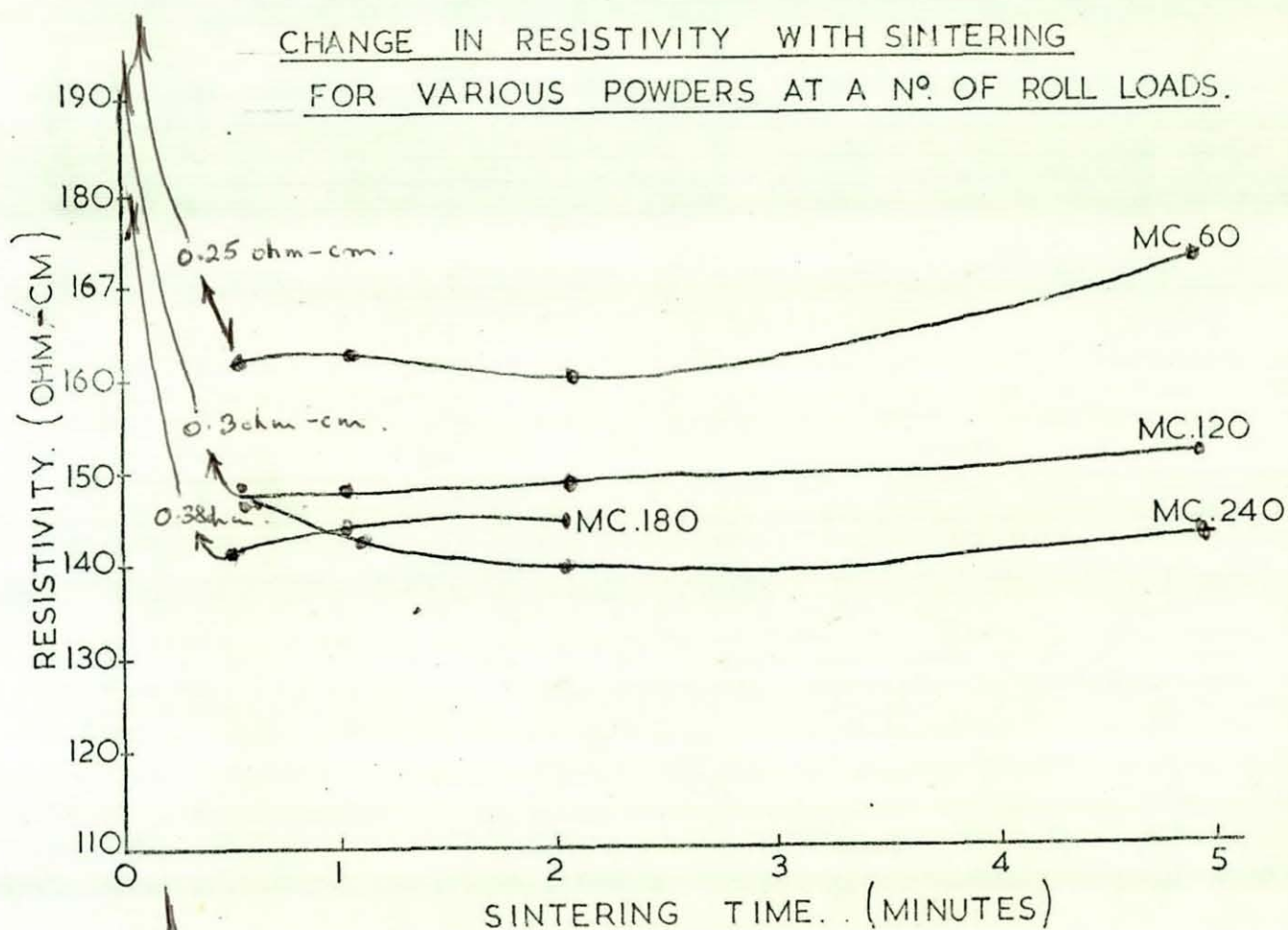


FIG. N° 35. CONT.

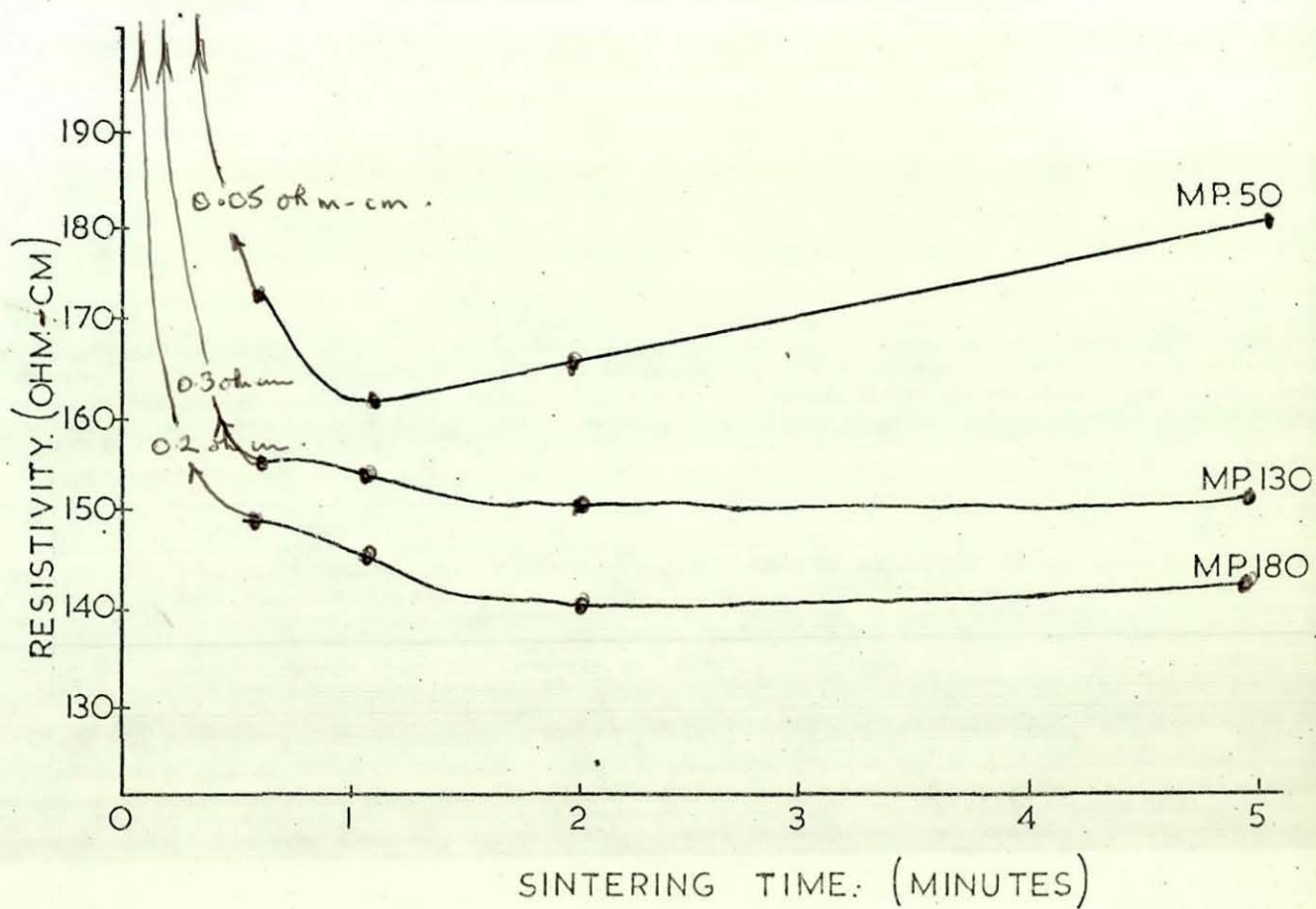
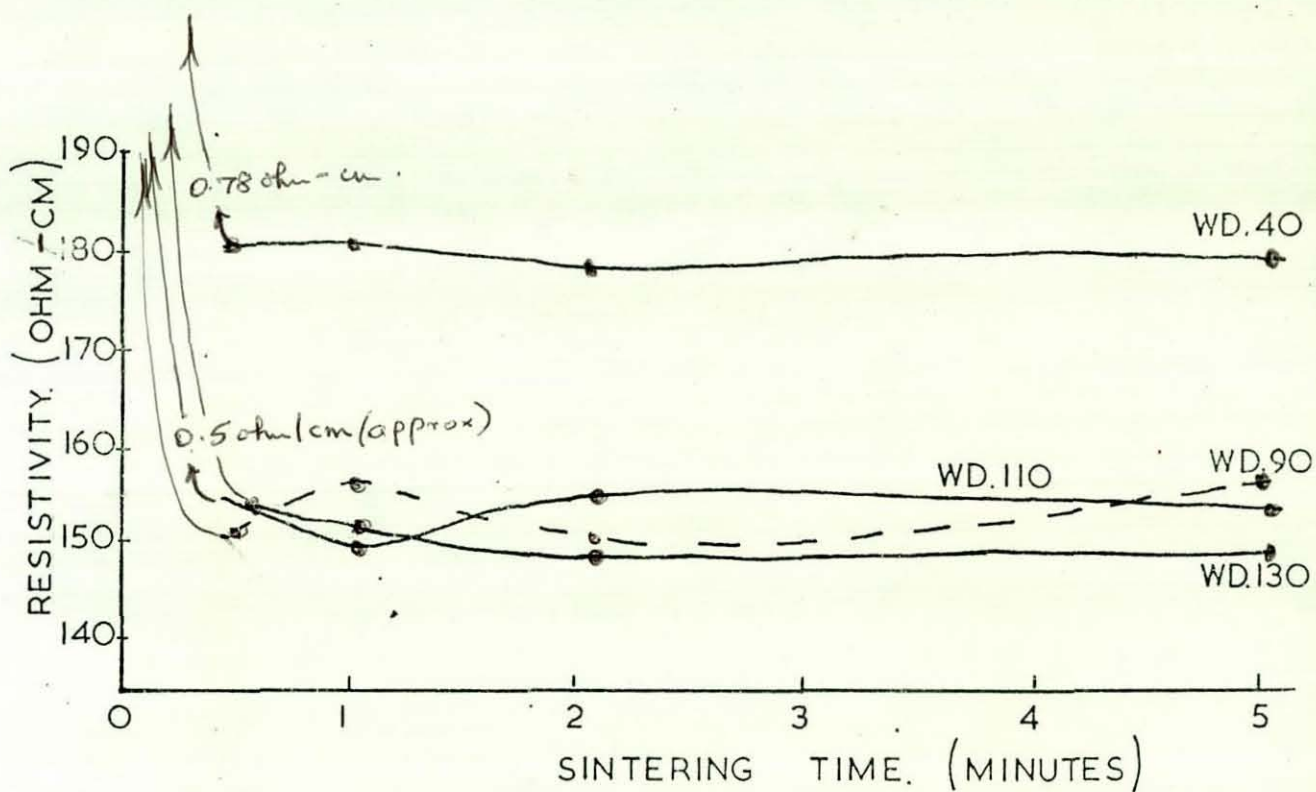
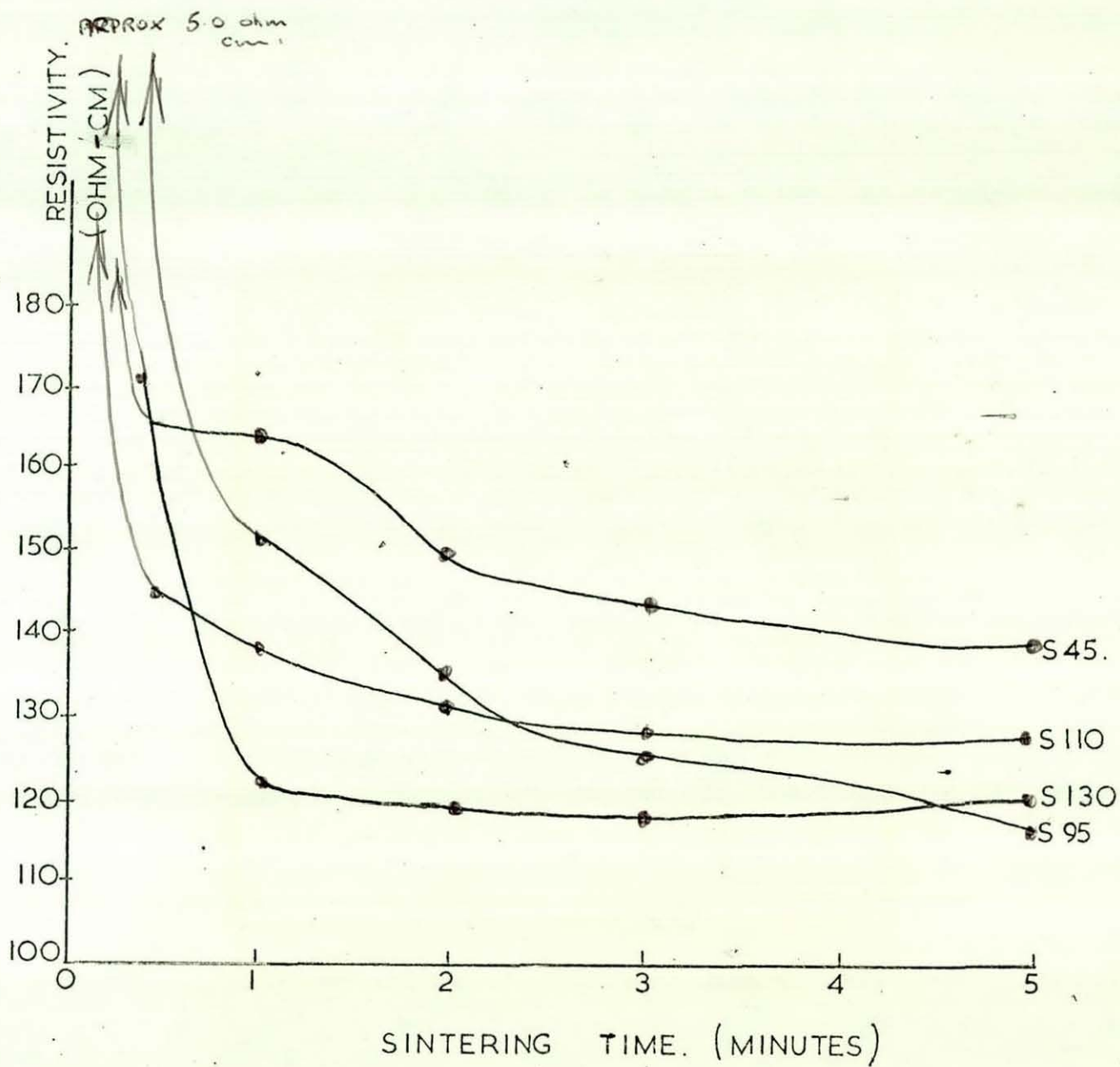


FIG. N° 35.



MC 60	=	J.J.M.	300	COARSE	FRACTION	ROLLED	AT	60 kN/25	mm.	WIDTH		
MC120	=	"	"	"	"	"	"	120	"	"	"	"
MC240	=	"	"	"	"	"	"	240	"	"	"	"
MF240	=	"		FINE	"	"	"	240	"	"	"	"
MF180	=	"	"	"	"	"	"	180	"	"	"	"
MF110	=	"	"	"	"	"	"	110	"	"	"	"
MF 60	=	"	"	"	"	"	"	60	"	"	"	"
MP 50	=	"		"AS RECEIVED"	"	"	"	50	"	"	"	"
MP130	=	"		"	"	"	"	130	"	"	"	"
MP180	=	"		"	"	"	"	180	"	"	"	"
WD 40	=	-	300	MESH	WOODALL	DUCKAM	"	40	"	"	"	"
WD 90	=	"	"	"	"	"	"	90	"	"	"	"
WD110	=	"	"	"	"	"	"	110	"	"	"	"
WD130	=	"	"	"	"	"	"	130	"	"	"	"
S 45	=	SINTREX		ELECTROLYTIC	POWDER	"	"	45	"	"	"	"
S 95	=	"	"	"	"	"	"	95	"	"	"	"
S 110	=	"	"	"	"	"	"	110	"	"	"	"
S 130	=	"	"	"	"	"	"	130	"	"	"	"

FIG. NO. 36.



MAG
x 2.2K

B.S.A. WATER ATOMIZED POWDER

SINTERED FOR 10 SECONDS

FIG. NO. 37.

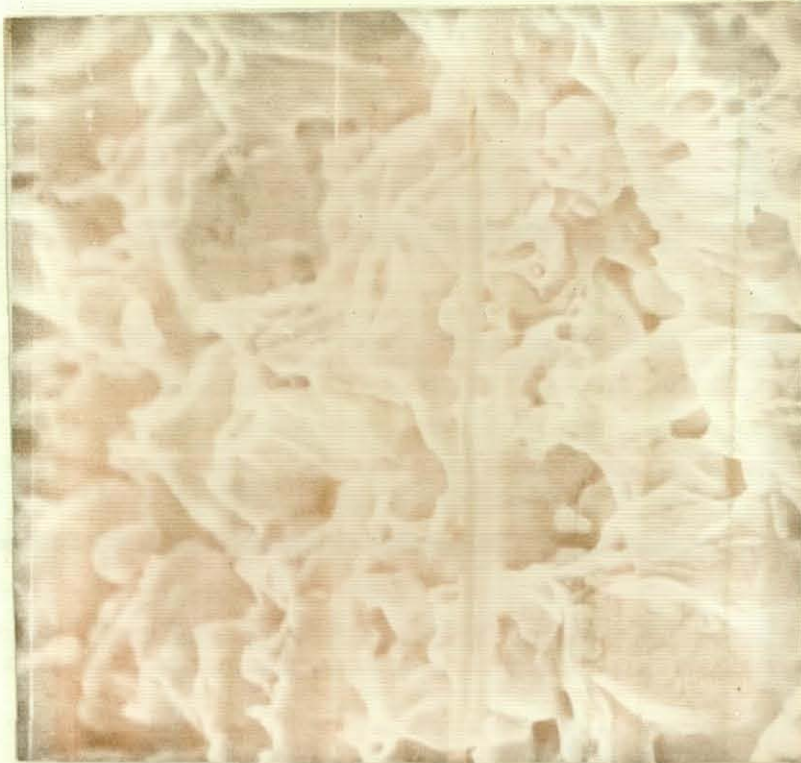


MAG.
x 1.5 K.

B.S.A. WATER ATOMIZED POWDER

SINTERED FOR 60 SECONDS

FIG. NO. 38.



MAG.
x1.5K.

B.S.A. WATER ATOMIZED POWDER

SINTERED FOR 300 SECONDS

FIG. NO. 39.

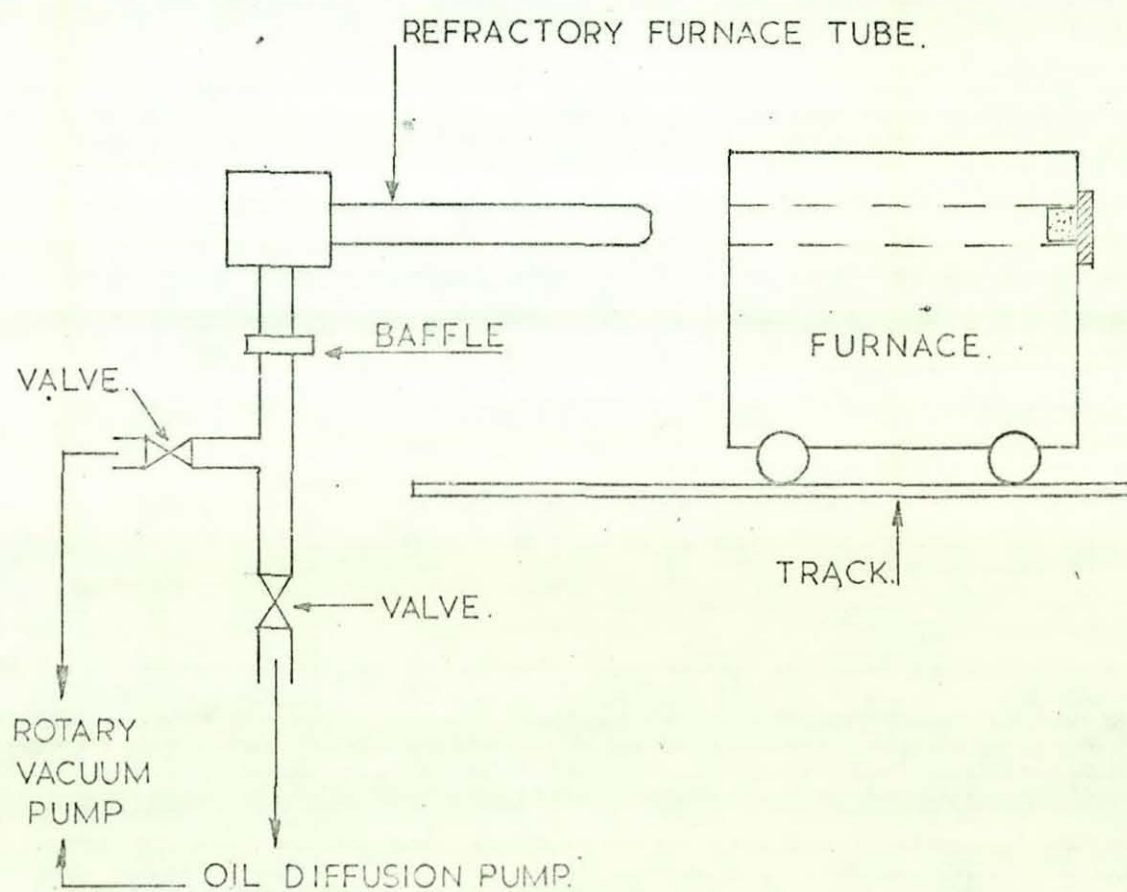


MAG.
x 500

B.S.A. WATER ATOMIZED POWDER

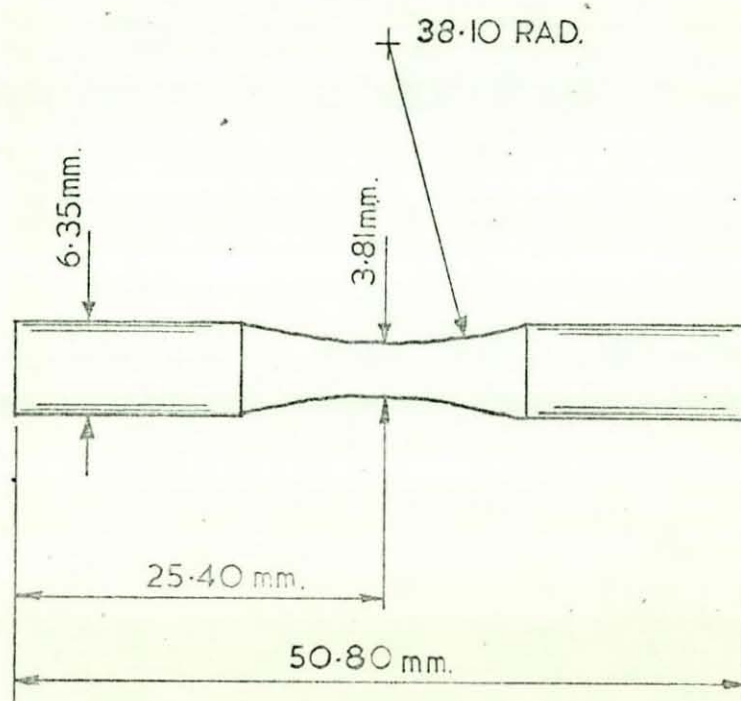
"AS ROLLED"

FIG. N°40



VACUUM FURNACE FOR SINTERING
NICKEL SPECIMENS.

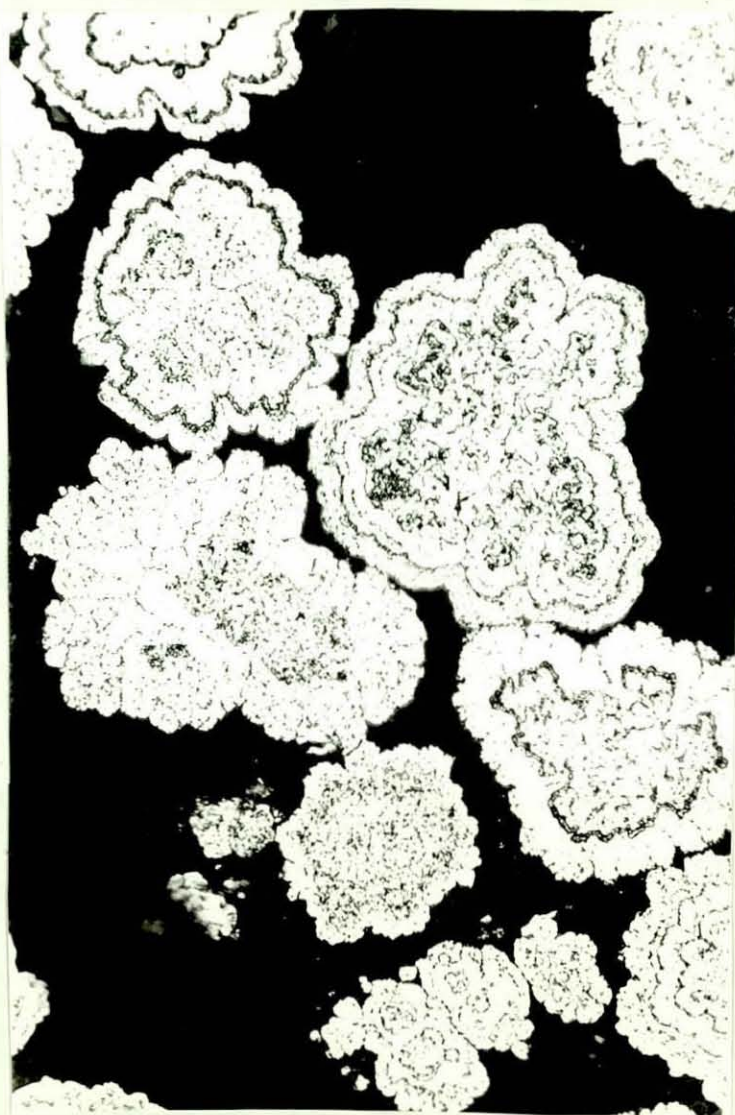
FIG. N°41



A K70B FATIGUE SAMPLE.

(TWICE FULL SIZE.)

FIG. NO. 42.

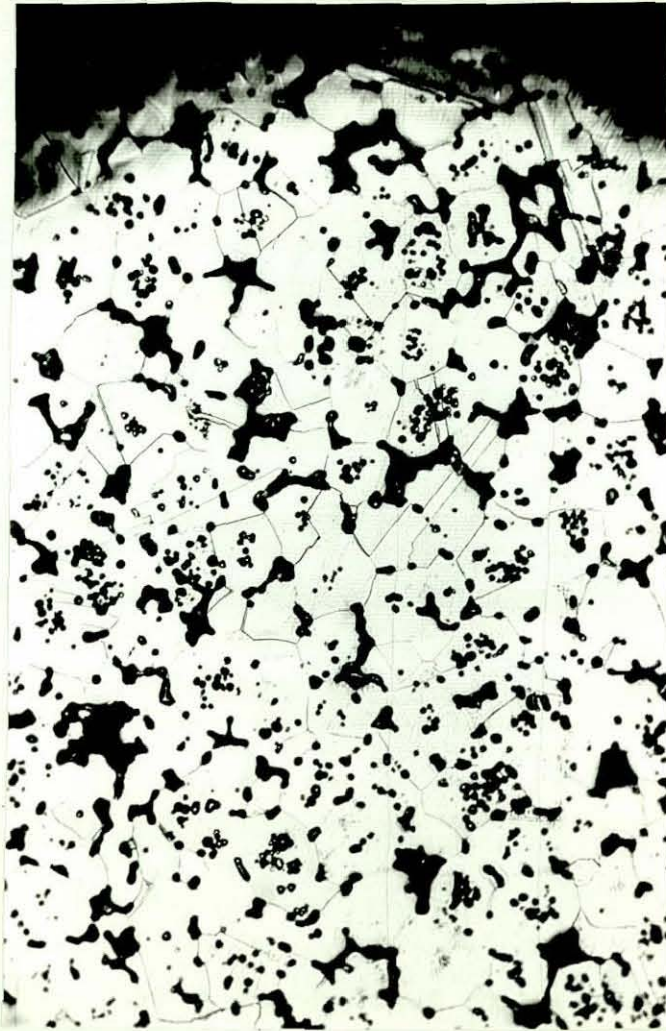


MAG.
x400

MICROSTRUCTURE OF "AS RECEIVED".

SHERRITT GORDON NICKEL POWDER.

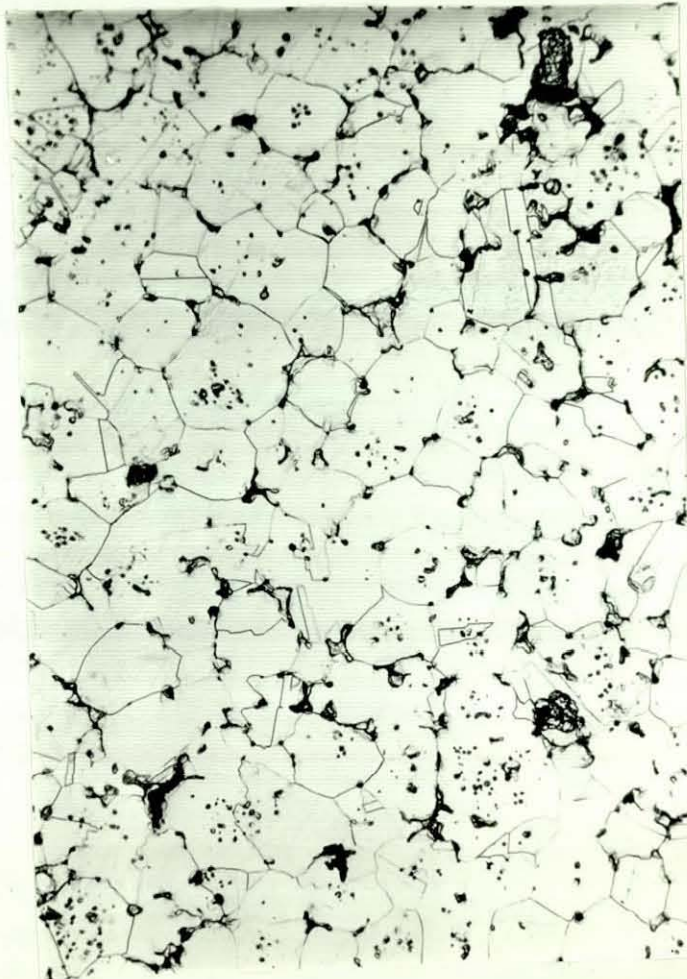
FIG. NO. 43.



MAG.
x150

FILM LIKE POROSITY IN 12% POROSITY SAMPLE
MADE FROM -200 + 200 MESH NICKEL POWDER
(5% SULPHURIC ACID ETCHED)

FIG. NO. 44.



MAG.
x150

FILM LIKE POROSITY IN 12% POROSITY SAMPLE

MADE FROM - 200 + 240 MESH NICKEL POWDER

(ION ETCHED)

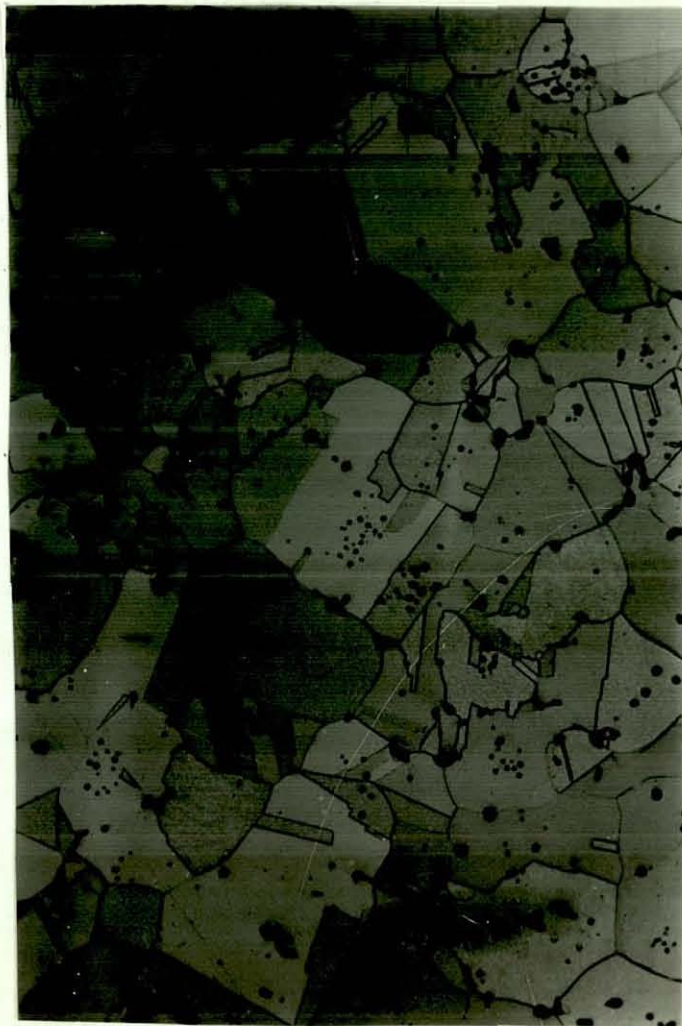
FIG. NO. 45.



MAG
X1100

SCANNING ELECTRON MICROGRAPH OF THE
FATIGUE FRACTURE OF 4% POROSITY SERIES
MADE FROM "AS RECEIVED" NICKEL POWDER.

FIG. NO. 46.



MAG
x150

MICROSTRUCTURE OF 4% POROSITY SAMPLE

PREPARED FROM -150 + 200 MESH NICKEL POWDER

(ION ETCHED)

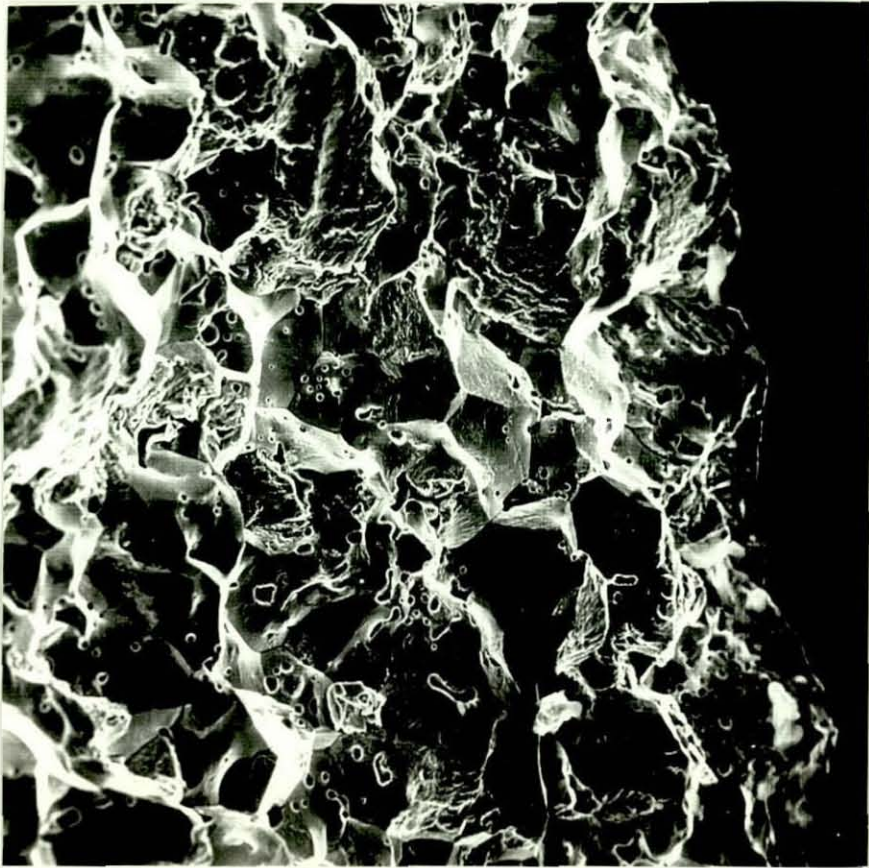
FIG. NO. 47.



MAG.
x600

SCANNING ELECTRON MICROGRAPH OF THE FATIGUE FRACTURE
OF 10% POROSITY SAMPLE MADE FROM - 150 + 200 MESH
NICKEL POWDER SHOWING UNDULATED AND FACETED SURFACES.

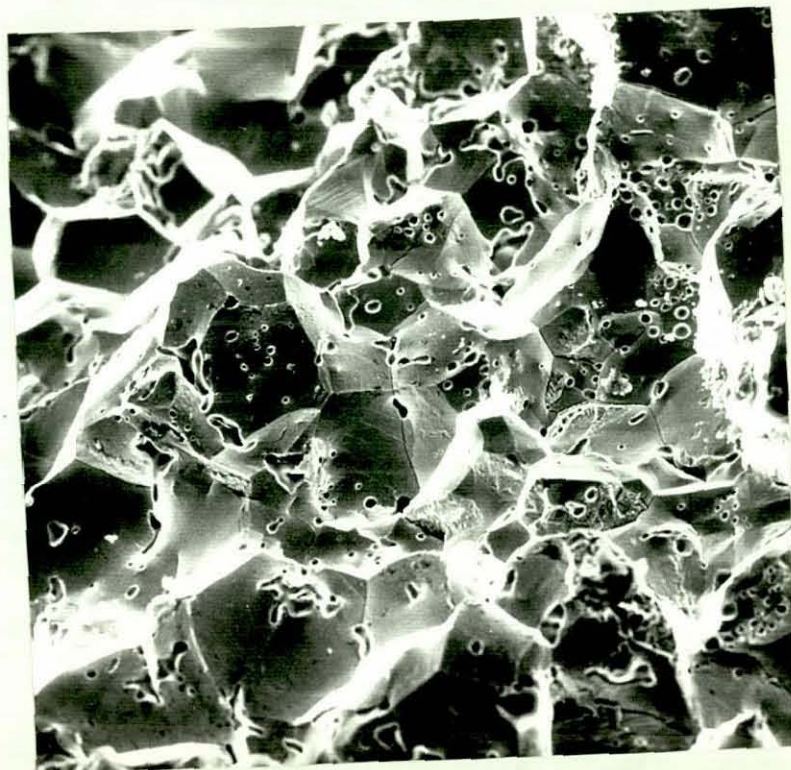
FIG. NO. 48.



MAG.
x250

SCANNING ELECTRON MICROGRAPH OF THE
FATIGUE FRACTURE OF A 4% POROSITY SAMPLE
MADE FROM -200 + 240 MESH NICKEL POWDER.

FIG. NO. 49.



MAG.

x200

SCANNING ELECTRON MICROGRAPH OF
THE FATIGUE OF A 4% POROSITY SPECIMEN
MADE FROM -150 + 200 MESH NICKEL POWDER

TABLE. 1.

POWDER	% +200 MESH	% -200 +240 MESH	% -240 +300 MESH	% -300 +350 MESH	% -350 MESH
J.J.M. 300	-	-	-	0.20	99.80
SINTREX ELECTROLYTIC POWDER	.43	.79	3.49	6.68	88.61
WOODALL DUCKHAM POWDER	20.85	17.10	15.87	14.00	32.18
COARSE PARTICLES EX J.J.M. 300	0.02	0.08	0.04	0.20	99.66
B.S.A. - 100 MESH POWDER	38.90	9.60	7.40	6.40	37.70
B. S. A. - 300 MESH POWDER	-	-	-	15.3	84.7

SIEVE ANALYSIS DATA.

TABLE 1.

POWDER	TIME SIEVED	PERCENTAGE + 31 MICRONS.
J.J.M. 300	3 hours	40.3
J.J.M. 300	5 hours	78.0
J.J.M. 300	8 hours	48.0
J.J.M. 300	8 hours	48.0
WOODALL DUCKAM POWDER - 300 MESH	8 hours	58.0
SINTREX ELECTROLYTIC POWDER	8 hours	46.0

MICROSIEVING DATA.

TABLE 2.

PARTICLE SIZE (microns)	PER CENTAGE DISTRIBUTION BY WEIGHT.
41.5 - 45	15.1
38 - 41.5	13.8
35 - 38	12.8
32 - 35	11.6
29.5 - 32	10.8
25.5 - 29.5	9.9
23.5 - 25.5	8.7
20 - 23.5	7.5
15 - 20	6.4
< 15	3.4

SEDIMENTATION DATA FOR J.J.M. 300

TABLE 3.

POWDER	NO. OF FIELDS EXAMINED	NO. OF PARTICLES COUNTED	% - 10 MICRONS	% 10 - 20 MICRONS	% 20 - 30 MICRONS	% 30 - 40 MICRONS	% 40 - 50 MICRONS	% +50 MICRONS
J.J.M.300	30	869	26.8	12.7	37.2	12.5	5	5.8
J.J.M.300	28	756	27.9	8.9	31.3	16.0	8.7	11.0
FINES EX J.J.M.300	24	699	34.9	18.3	40.8	3.0	2.0	1.0
FINES EX J.J.M.300	25	900	39.0	17.6	36.2	5.8	1.4	-
SINTREX ELECTROLYTIC POWDER	31	1,177	27.8	12.8	35.8	10.4	4.8	8.4
SINTREX ELECTROLYTIC POWDER	7	163	22.7	8.0	34.5	11.6	9.4	14.0
COARSE EX MAKIN POWDER	46	1,955	17.8	3.2	23.6	21.8	14.0	19.6
-300 MESH WOODALL DUCKHAM POWDER	40	1,653	44.0	10.3	24.0	10.2	5.7	4.6

PARTICLE SIZE DISTRIBUTION BYQ.T.M. OF IRON POWDERS.

TABLE No. 4.

POWDER	AVERAGE PROJECTED AREA PER PARTICLE (mm ²)	DIAMETER CIRCLE OF SAME AREA (μ)
J.J.M. 300	.000295	18.4
FINES EX J.J.M.300	.000165	14.0
COARSE FRACTION EX J.J.M.300	.00055	26.5
SINTREX ELECTROLYTIC POWDER	.000363	21.4
-300 MESH WOODALL DUCKHAM POWDER	.00019	15.4
B.S.A. -100 MESH POWDER	.000216	16.6
B.S.A. -300 MESH POWDER	.000155	14.1

PARTICLE SIZE DERIVED FROM

O.T.M. SURFACE AREA DATA.

TABLE 5.

POWDER	SURFACE AREA (m ² /g)
J.J.M.. 300	0.13
ELECTROLYTIC POWDER	0.074
WOODALL DUCKAM	0.19
WOODALL DUCKAM - 300 MESH	0.24
B.S.A. - 100 MESH	0.25
B.S.A. - 300 MESH	0.25

SURFACE AREA RESULTS

(STEARIC ACID ADSORPTION)

TABLE 6.

POWDER	SURFACE AREA (m ² /g)
J.J.M. 300	0.31
SINTREX ELECTROLYTIC -300 MESH	0.14
WOODALL DUCKAM AS RECEIVED	0.19
WOODALL DUCKAM -300 MESH	0.22

B.F.T. SURFACE AREA DATA

TABLE 7.

POWDER	WEIGHT SPECIFIC SURFACE OF POWDER (m ² /g)	RELATIVE SPECIFIC SURFACE.
FINES EX J.J.M. 300	0.0157	1.00
J.J.M. 300	0.0102	0.65
COARSE FRACTION EX J.J.M. 300	0.0072	0.46
SINTREX ELECTROLYTIC POWDER	0.0092	0.59
WOODALL DUCKAM POWDER -300 MESH	0.0076.4	0.49
B.S.A. POWDER -100 MESH	0.0042.0	0.27
B.S.A. POWDER -300 MESH	0.0070	0.45

SPECIFIC SURFACE DATA

DETERMINED BY THE

LEA AND NURSE METHOD.

TABLE NO. 8.

POWDER	RELATIVE SURFACE AREA
J.J.M. 300	.68
FINES EX J.J.M.300	1.00
COARSE FRACTION EX J.J.M.300	.49
SINTREX ELECTROLYTIC POWDER	.61
-300 MESH WOODALL DUCKHAM POWDER	.86
B.S.A. -100 MESH POWDER	.77
B.S.A. -300 MESH POWDER	1.00

RELATIVE SURFACE AREA RESULTS BY Q.T.M.

TABLE 9.

POWDER	APPARENT SOLID DENSITY (g/c.c.)	PER CENTAGE CLOSED POROSITY
MAKIN POWDER (AS RECEIVED)	7.75	1.5%
MAKIN POWDER (FINE FRACTION)	7.51	4.6%
MAKIN POWDER (COARSE FRACTION)	7.87	-
SINTREX ELECTROLYTIC POWDER	7.69	2.3%
WOODALL DUCKEN POWDER	7.79	1.1%
B.S.A. -100 MESH POWDER	7.76	1.4%
B.S.A. -300 MESH POWDER	7.75	1.5%

APPARENT DENSITIES

TABLE 10.

POWDER	COMPACTION PRESSURE.					
	133 N/mm ² (3.6 t.s.i.)	176 N/mm ² (11.4 t.s.i.)	231 N/mm ² (15 t.s.i.)	310 N/mm ² (20 t.s.i.)	386 N/mm ² (25 t.s.i.)	463 N/mm ² (30 t.s.i.)
WOODALL DUCKAM - 300 MESH	4.77 g/cc	4.80 g/cc	5.43 g/cc	5.90 g/cc	6.25 g/cc	6.52 g/cc
SINTREX ELECTROLYTIC POWDER	5.06	5.10	5.67	6.20	6.54	6.50
J.J.M. 300	4.95	5.10	5.63	5.90	6.21	6.48
FINES EX J.J.M. 300	4.48	4.42	5.039	5.51	5.87	6.04
COARSE FRACTION EX J.J.M. 300	4.80	5.09	5.48	5.83	6.13	6.35

STATIC COMPACTION DATA

TABLE 11.

ELEMENT	PERCENTAGE BY WEIGHT.
C	0.15
S	0.025
P	0.002
Si	0.13
Mn	0.07
O	0.33
N	0.006

CHEMICAL ANALYSIS OF

B.S.A. -100 MESH ATOMIZED POWDER.

TABLE 12.

CHEMICAL ANALYSIS %	J.J.M.300.	SINTREX ELECTROLYTIC 300 POWDER	WOODALL DUCKAM POWDER
C	0.084	0.008	0.015
S	0.015	0.018	0.008
P	0.011	0.006	0.020
Si	0.07	0.01	0.03
Mn	0.37	0.04	0.25
O	0.85	0.15	0.18

CHEMICAL ANALYSIS DATA.

TABLE 13

POWDER	ROLL LOAD (kN/25 mm ²)	SINTERING TIME (SECONDS)	DENSITY (g/c.c.)
WOODALL	110	30	7.30
DUCKAM POWDER	110	300	7.31
SINTREX	130	30	7.27
POWDER	130	300	7.34

DENSITIES OF SOME ROLLED
& SINTERED SPECIMENS.

TABLE 14. TENSILE STRENGTHS OF COMPACTED AND SINTERED STRIP.

POWDER	INITIAL ROLL LOAD kN/25 m.m. Width.	T.S.AFTER 30 SECDS. N/mm ² .	T.S.AFTER 60 SECDS. N/mm ² .	T.S.AFTER 120 SECDS. N/mm ² .	T.S.AFTER 300 SECDS. N/mm ² .
Sintrex Powder	45	116	126	162	161
	95	-	125	132	185
	130	-	130	161	134
Woodall Duckam Powder	40	110	124	138	131
	90	153	211	234	196
	110	175	262	156	187
	130	135	189	197	148
J.J.M. 300 Powder	50	143	181	184	183
	130	183	205	203	209
	180	177	207	183	192
	220	183	2205	203	209
	240	-	178	198	-
J.J.M. 300 Coarse Fraction Powder	60	148	152	173	164
	120	132	184	188	201
	240	185	204	199	208
J.J.M. 300 FINE FRACTION POWDER	60	143	175	164	201
	110	143	203	217	213
	180	182	188	-	250
	240	203	214	-	-

TABLE 15.

POWDER	INITIAL ROLL LOAD	INITIAL SINTERING TIME	SECOND ROLL LOAD	SECOND SINTERING TIME	T.S.	el
	kN/25mm. width.	Seconds	kN/25mm. width	Seconds	N/mm ² .	%.
J.J.M.300 COARSE FRACTION	240	30	150	30	254	5
J.J.M.300	40	30	150	30	325	10
J.J.M.300 FINE FRACTION	120	30	150	30	353	14
J.J.M.300	120	30	150	30	298	10
WOODALL DUCKAM	40	30	150	30	197	11

TENSILE STRENGTH OF FINISHED STRIP.

Table 16.

Apparent Density, Tap Density and Flow Rate
for the As-received Powder and the Sieve
Fractions Used in the Prepared of Specimens

POWDER	APPARENT DENSITY g/cm ³	TAP DENSITY g/cm ³	HALL FLOW RATE seconds
As-received	4.47	5.02	22.5
-240 + 300# (61 - 53 μ m)	4.24	4.86	22.4
-200 + 240# (74 - 61 μ m)	4.27	4.89	21.4
-150 + 200# (104 - 74 μ m)	4.30	4.92	21.8

TABLE 17

COMPACTION STAGE	COMPACTION LOAD (N/mm. ²)	DENSITY (g/cc)
AS PRESSED	355	7.02
1ST COINING	355	7.74
2ND COINING	355	7.87
3RD COINING	355	8.01
4TH COINING	355	8.07
5TH COINING	355	8.14
6TH COINING	355	8.28
7TH COINING	355	8.33

MULTIPLE REPRESSING AND ANNEALING OF

"AS RECEIVED" ANNEALED POWDERS.

Table 18.

POROSITY AND GRAIN SIZE CHARACTERISTICS

SET	PCRE SIZE DISTRIBUTION FRACTION OF PORES WITH CHORD LENGTHS							MEAN* PORE CHORD LENGTH μm	POROSITY MEASURED BY QTM %	POROSITY MEASURED BY DENSITY %	GRAIN* SIZE μm
	>0 μm	>5 μm	>10 μm	>15 μm	>20 μm	>25 μm	>30 μm				
12F	1	0.63	0.27	0.11	0.05	0.03	0.02	6.2	8.4	11.8	25
12N	1	0.45	0.20	0.09	0.04	0.02	0.01	5.2	4.7	12.2	38
12C	1	0.49	0.20	0.03	0.04	0.02	0.01	4.9	4.9	10.1	31
4F	1	0.39	0.10	0.03	0.01	0.00(6)	0.00(3)	4.0	3.1	3.9	24
4N	1	0.36	0.11	0.04	0.01	0.00(6)	0.00(3)	4.0	3.3	4.0	29
4C	1	0.50	0.21	0.08	0.03	0.01	0.01	5.0	3.2	3.5	30
4D	1	0.43	0.11	0.04	0.01	0.00(5)	0.00(2)	4.3	3.3	4.5	22

*Number average

+Mean linear intercept

Table 19.

Tensile and Fatigue Properties of Sintered Sheritt

Gordon Nickel

SET	0.5% Proof Stress N/mm ²	Tensile Strength N/mm ²	Elongation %	Reduction in Area %	Endurance* Limit at 10 ⁸ cycles N/mm ²	Endurance Ratio
12F		194	18		70	0.36
12M	90	198	16	10	73	0.37
12C	100	161	8	8	59	0.39
4F	138	267	20	18	121	0.45
4M	142	262	20	15	118	0.45
4C	146	259	18	14	118	0.45
4D	151	264	20	17	118	0.45

* In all series the endurance limit at 10⁷ cycles was ~2 N/mm² higher than that at 10⁸ cycles.

Table 20.

Fatigue Data for Sintered Nickel Specimens

Density g/cm ³	Porosity* %	Maximum Stress N/mm ²	Cycles to Failure
<u>Set 12F</u>			
7.84	11.7	108	3.6×10^4
7.90	11.0	93	1.4×10^6
7.90	11.0	93	1.5×10^6
7.77	12.5	85	7.9×10^5
7.84	11.7	74	2.9×10^6
7.75	12.7	69	$>10^8$
7.82	11.9	62	$>10^8$
Estimated Endurance Limit at 10^8 cycles ± 70 N/mm ²			
<u>Set 12M</u>			
7.97	10.2	108	1.4×10^5
7.70	13.3	93	5.4×10^5
7.79	12.2	85	5.0×10^5
7.80	12.2	77	1.1×10^6
7.71	13.2	72	$>10^8$
Estimated Endurance Limit at 10^8 cycles ± 73 N/mm ²			
<u>Set 12C</u>			
8.11	8.7	108	7.1×10^4
7.82	11.9	93	4.0×10^4
8.00	10.0	93	1.8×10^5
7.96	10.4	77	6.8×10^5
8.05	9.3	71	2.0×10^5
7.96	10.4	62	1.1×10^6
8.00	10.0	57	$>10^8$
Estimated Endurance Limit at 10^8 cycles ± 59 N/mm ²			

Density g/cm ³	Porosity %	Maximum Stress N/mm ²	Cycles to Failure
<u>Set 4F</u>			
8.54	4.05	139	2.6×10^6
8.63	3.04	139	1.2×10^6
8.53	4.16	124	5.5×10^7
8.52	4.27	120	$>10^8$
Estimated Endurance Limit at 10^8 cycles ± 121 N/mm ²			
<u>Set 4M</u>			
8.54	4.05	170	2.0×10^5
8.57	3.70	155	2.2×10^5
8.55	3.94	139	1.7×10^6
8.51	4.48	124	6.6×10^6
8.55	3.94	120	2.2×10^7
8.55	3.94	116	$>10^8$
8.55	3.94	93	$>2 \times 10^7$
Estimated Endurance Limit at 10^8 cycles ± 118 N/mm ²			
<u>Set 4C</u>			
8.62	3.14	154	6.3×10^5
8.58	3.60	139	5.3×10^5
8.57	3.70	135	1.8×10^6
8.56	3.82	131	5.5×10^7
8.58	3.60	124	3.2×10^6
8.59	3.49	120	2.6×10^7
8.60	3.38	116	$>10^8$
Estimated Endurance Limit at 10^8 cycles ± 118 N/mm ²			

Density g/cm ³	Porosity* %	Maximum Stress N/mm ²	Cycles to Failure
<u>Set 4D</u>			
8.53	4.16	154	1.5×10^4
8.47	4.83	147	2.3×10^5
8.40	5.63	139	1.8×10^5
8.52	4.27	131	8.3×10^5
8.41	5.50	127	1.3×10^5
8.61	3.26	116	$>10^8$
Estimated Endurance Limit at 10^8 cycles ± 118 N/mm ²			

* Assuming density of pore free nickel is 8.90 g/cm³

