I hereby certify that the work described in this thesis has been carried out under my general direction and supervision.

....
Lecturer, Metallurgy Department
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September 1981

I hereby certify that this thesis has not been previously submitted for a degree in any University, nor is it being submitted concurrently for a degree in any other University.

Geraint P. Williams
September 1981

THE HOT FORGING OF COPPER - 10% NICKEL POWDER COMPACTS

by

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SYNOPSIS

A research programme was set up to investigate the effects of a number of process variables on the structure and selected properties of a hot forged alloy of Copper - 10% Nickel powder compacts and to examine the processes occuring during the hot forging operation. X-ray analysis was also performed on a range of compacts to study any variations in homogeneity resulting from the use of different powders and process variables.

The variables selected for the investigation were those of forging temperature, initial powder particle size and initial powder particle size ratio. A systematic tabular method was employed in the analysis of the results to identify those variables and interactions of variables which had the most significant effect on the properties of the final forgings.

It was observed that the most significant factor involved in the operation was that of forging temperature, however serious consideration must also be given to the initial particle size. In some cases the effect of the latter variable was seen to be reversed by using different powders. The X-ray analysis proved that the homogeneity of the forgings made from both types of powder was almost identical. The differences in mechanical properties were considered as being due to the variation in preforging homogeneity between the compacts giving varying degrees of recrystallisation. Explanations are offered for the observations made.

The metallographic examination served to confirm the observations made from the results of mechanical testing and X-ray analyses.

CHAPTER 1: THE POWDER FORGING PROCESS

Section 1: Introduction to the Process

Powder forging is a process by which preforms made by conventional powder metallurgy techniques are hot forged in closed impression dies. It is considered as being one of the metal working techniques based on the use of powders which offers the greatest potential for widespread commercial exploitation. In a way it is a hybrid of the forging and conventional powder metallurgy techniques in that it combines the best aspects of both processes. The ability to easily pour metal powders into a closed die and produce a shape which can subsequently be heated and forged again in a closed die illustrates the basic nature of the process and reveals that its major advantage over conventional methods is one of material saving. The basic route for the powder forging process is shown schematically in Fig. 1.

Preparation of the powder to be compacted is the first step; this involves, if required, addition of internal lubricant such as graphite and alloying elements to the base powder and carefully blend them to give a homogenous mix. A carefully designed green preform is then made from this powder by conventional powder metallurgy techniques. The next stage involves the sintering or heating of the preform in a controlled atmosphere and then either cooling, reheating and then forging, or forging the preform direct from the furnace while it is still hot. The sintering treatment depends on the length of time required for diffusion of the alloying elements and whether or not there is a need for lubricant burn off. During the hot forge the preform assumes the geometry of the die, and the porosity is reduced to

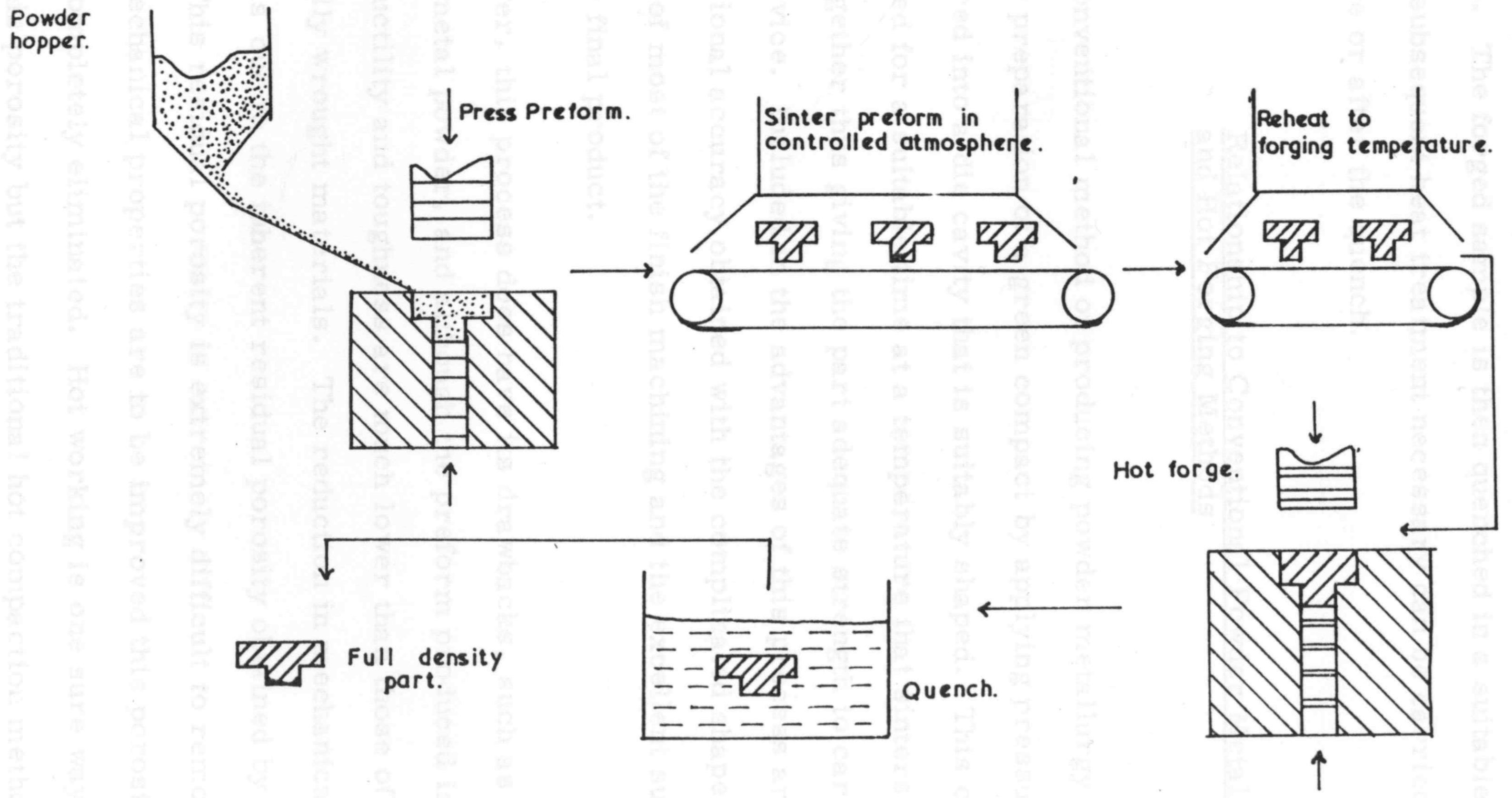


Fig.1 Schematic route for powder forging.

almost zero. The forged sample is then quenched in a suitable media; any subsequent heat treatment necessary can be carried out either before or after the quench.

Section 2: Relationship to Conventional Powder Metallurgy and Hot Forging Methods

The conventional method of producing powder metallurgy parts involves the preparation of a green compact by applying pressure to powder poured into a die cavity that is suitably shaped. This compact is then heated for a suitable time at a temperature that sinters the particles together thus giving the part adequate strength to carry loads in service. Included in the advantages of this process are the high dimensional accuracy obtained with the complicated shapes formed, elimination of most of the finish machining and the excellent surface finish of the final product.

However, this process does have its drawbacks, such as the cost of the metal powder, and although the preform produced is quite strong its ductility and toughness are much lower than those of the conventionally wrought materials. The reduction in mechanical properties is due to the inherent residual porosity obtained by this process. This residual porosity is extremely difficult to remove but if the mechanical properties are to be improved this porosity must be almost completely eliminated. Hot working is one sure way of removing this porosity but the traditional hot compaction method is inefficiently expensive and would not be applicable to producing large quantities of engineering components. It is a slow and cumbersome process which involves heating the powder by heating the die in which it is enclosed. Not only does this consume time but a great deal of

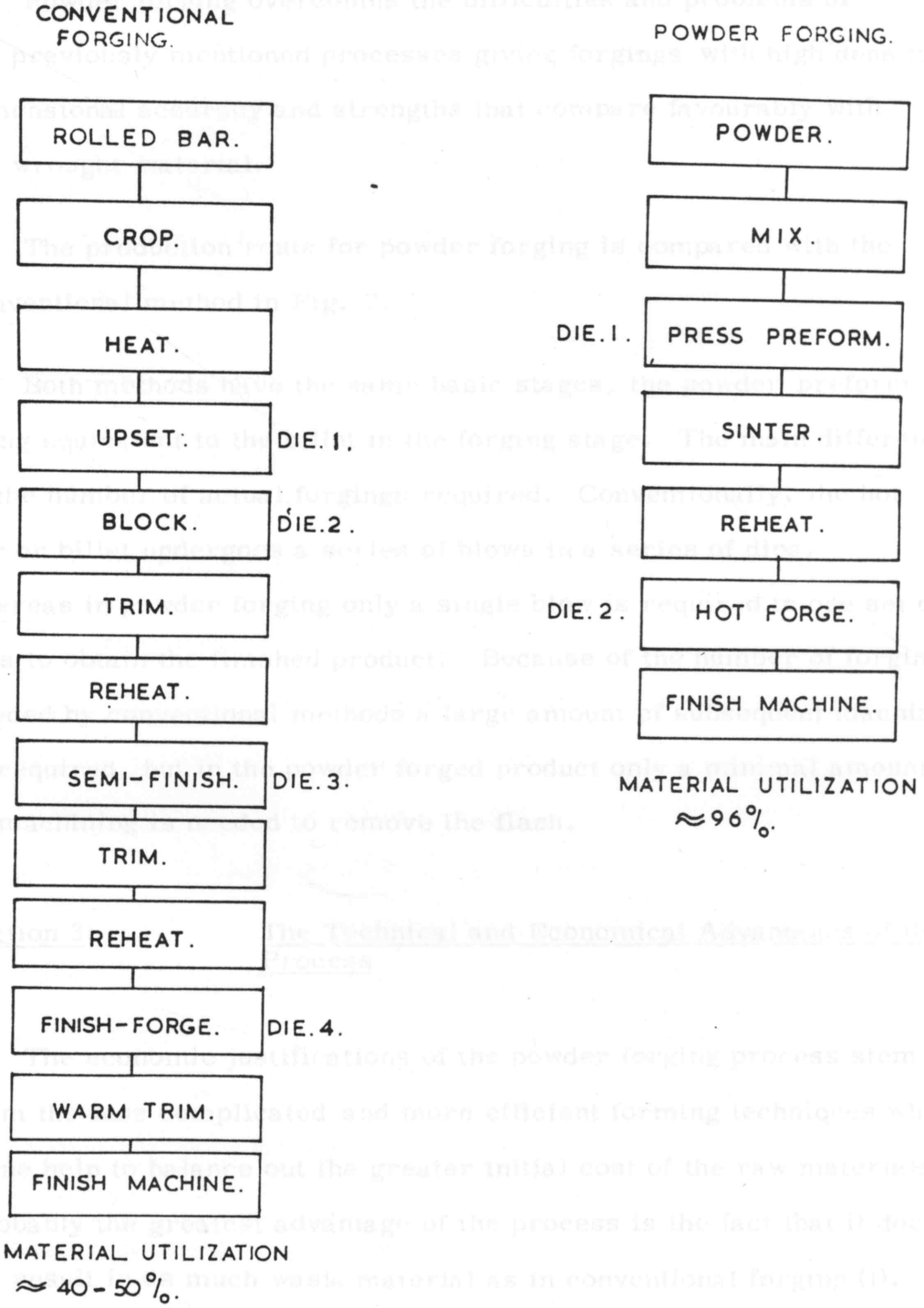


Fig. 2 Schematic comparison of conventional and powder forging routes.

energy is wasted as well. The conventional method of producing high strength materials is hot forging, but this again has its limitations in that the final forgings are not always dimensionally accurate and the material utilisation is poor.

Powder forging overcomes the difficulties and problems of the previously mentioned processes giving forgings with high density, dimensional accuracy and strengths that compare favourably with the wrought material.

The production route for powder forging is compared with the conventional method in Fig. 2.

Both methods have the same basic stages, the powder preform being equivalent to the billet in the forging stage. The main difference is the number of actual forgings required. Conventionally, the hot bar or billet undergoes a series of blows in a series of dies, whereas in powder forging only a single blow is required in one set of dies to obtain the finished product. Because of the number of forgings needed by conventional methods a large amount of subsequent machining is required, but in the powder forged product only a minimal amount of machining is needed to remove the flash.

Section 3: The Technical and Economical Advantages of the Process

The economic justifications of the powder forging process stem from the less complicated and more efficient forming techniques which alone help to balance out the greater initial cost of the raw materials. Probably the greatest advantage of the process is the fact that it does not result in as much waste material as in conventional forging (1). This is because in powder metallurgy forging, the material flows into pores, whereas conventionally, forging dense material to a smaller volume moves the material to the outside resulting in waste. It is this saving on waste material that can reduce or even eliminate the initial price difference between the powder and wrought bar or billet. This is especially pronounced in the case of the more expensive materials such as titanium and aerospace alloys. (2)

The simplified forging route means that a single forging stroke can replace the three or more forging blows required in conventional forging. Not only does this increase production rates but it also gives rise to other advantages. These include less die and tool wear due to the decrease in number of forging strokes, also, as the compactability of a porous billet is greater than a solid one the temperature and pressure of compaction need not be so high - this again reduces wear. Because of this reduction in forging pressure and temperature, smaller presses can be used.

Diminishment in the amount of shop noise is another result of the reduction in number of forging strokes. Multisequence tools and the intermediate heating apparatus used conventionally would not be needed in powder forging giving yet another cut in tool costs. However, the tools and dies used in powder forgings are more complex and expensive and this must be considered while assessing the overall economics of the process. Component detail and surface finish of powder forgings such as in gear teeth and connecting rods, is much better for a given load than in conventional forging and sometimes completely eliminates the need for any finish machining. Again, as a protective atmosphere is used during the heat treatment of porous powder preforms, any surface decarburisation or oxidation is greatly reduced so that no surface finishing is required. In this way machining costs are substantially lowered.

Forgings with accurate, consistent weights can be produced because the amount of powder used to produce the preform can be accurately monitored. (3) Compared with conventional forging this involves a substantial saving e.g., for the manufacture of an automative gear, less than 400 gms. would be used for the powder forging whereas nearly 1200 gms. of steel would be required to make the same blank conventionally.

One disadvantage of the process is the need to install protective atmosphere apparatus necessary during the heat treatment of the preforms to reduce oxidation which might give grain boundary aligned inclusions on forging, thus reducing toughness. The conversion of the shop floor to accommodate suitable apparatus would not only involve a great expense, but also there might be a need for additional floor space to 'house' this equipment. At present, however, the main disadvantage of powder forging is still the higher price of metal powder. Nevertheless, it is expected that as the demand for powder metallurgy products increases, and the price of wrought steel billet rises, there will eventually be a decrease in the initial price differential between the two processes.

Section 4: Applications and Properties of Powder Forgings

The products of powder metallurgy forging are basically those that have been produced by conventional methods. Included in these are automative gears, drive shaft flanges, and connecting rods, (3) (4) (5), other components now manufactured as powder metallurgy products are side gear blanks, toothed belt pulleys, universal joint inner races and many others. (6)

There are many reports on the properties of powder forged materials and many of these reports include comparison between powder forged and wrought products. Generally the properties of powder forgings have been shown to be at least as good as the wrought product for many materials. (5) (7) (8) (9) (10)

Ductility, fatigue and impact performance of powder forged steels have been found to compare favourably with the wrought material as long as the non metallic inclusion level is controlled and full density is obtained. (5) (11) (12) (13) (14) (15)

A recent report by the Aluminium Company of America (10) shows that the properties and performance of some aluminium powder forged parts compare excellently with their wrought counterparts and in some cases were event better than those produced by conventional methods.

The fatigue life of gear teeth produced by powder forging (16) is greater than those machined from conventional forgings, because of the ability to forge close tolerance radii at roots of gear teeth and around stress areas in functional parts. Another important factor in the increased fatigue life of powder forged components is the smooth surface obtained from the low temperature forging.

Tauenblat and Goller (17) have shown that parts with excellent electrical conductivity can be made by powder metallurgy techniques, other electrical and mechanical properties of these parts are similar to those of the wrought material. Included in these products are armature bearing blocks and contacts for circuit breakers.

The grain structure of a powder preform is inherently smaller than that of a casting and this difference prevails in the completed forging (18) (19) (20). The plastic flow associated with conventional forging gives a highly oriented grain structure resulting in reduced impact and ductile properties in direction transverse to rolling. Planes of weakness also occur due to the presence of elongated sulphide and silicate inclusions. Powder forged products however have a randomly oriented grain structure giving isotropic properties. The relatively small grain size of powder forgings is normally advantageous for strength and ductility at low temperatures, whereas for materials such as superalloys used at elevated temperatures this is a serious disadvantage as a larger grain size minimises grain boundary sliding. This leads to an improved high temperature creep strength.

At present, however, many experts believe that the main market for powder forgings will be in the higher price range alloys. (18) (21) Several manufacturers of tool steels have developed powder metallurgy methods for producing billets based on isostatic pressing and then hot working by rolling or extruding. In conventional forging the segregation tendancy increases as the alloy content increases hence resulting in a decrease in ductility during hot forming. However, in powder forged billets there is virtually a complete absence of segregation as each particle is in fact acting as a miniature ingot. Highly alloyed materials also incur the problem of a decrease in the temperature range available for forging as the alloy content increases - this is because the resistance to deformation at forging temperature increases with higher alloying content.

An important group is the Co and Ni based superalloys used for gas turbine blading. With the increase in demands on creep strength and oxidation of turbine blades, alloying compositions have become much more difficult to forge - so much so that some of the most refractory materials available at present cannot be forged at all and must be cast.

Powder metallurgy techniques have overcome these difficulties and there are examples of previously unforgeable alloys that can now be rolled from stock prepared by powder compaction. (18) There has been a great deal of interest shown in the development of 'superplastic forging', especially in the field of dispersion hardened alloys which can only be produced by powder metallurgy. (23) (24) The formation of turbine discs from IN - 100 is an example of this application.

Nevertheless it should be observed that superalloy components produced from powders are made by a process based on the forging of extruded stock and not on the hot compaction of powder directly to the desired shape. The problem of interparticle bonding or deoxidation of the particle surfaces is much greater in the case of direct forging than in extrusion because of the lower amount of shear occuring. However, some alloys with acceptable properties have been formed without the use of extrusion.

CHAPTER 2: IMPORTANT VARIABLES OF THE PROCESS

Section 1: The Effect of Preform Shape

One of the most challenging variables in the powder forging process is that of preform shape, it largely determines whether the component can be successfully forged or not. The design adapted is dependent on a number of factors; the component complexity determines the extent to which the preform must correspond to the completed forging. Load requirements are influenced by the extent to which metal must be redistributed, hence the press capacity must be considered. Other considerations include the forgeability and green density of the preform, powder characteristics and forging temperature and die wear are also important and must be taken into account when the economics of the process are examined (3).

Technically and economically, simply shaped preforms are the most desirable but for the forging to be successful a more complicated shape is often required. It is clear therefore that the two major types of preform are plain slugs that bear no resemblance to the final forging or a well shaped preform that closely resembles the final shape (4), (25), (26). A plain shaped preform is easier and cheaper to produce than a complicated one; for the final shape to be generated by flow of the material a low density preform is advisable so that the material flows under the lightest loads. It has been suggested that the use of large deformation is advantageous in healing porosity and that the fact that a great deal of flow can take place at relatively low forging loads is another desirable attribute (27). This plastic flow is thought to assist in the early elimination of porosity and seal up cracks that are formed in the early stages of deformation in such a way that they are virtually invisible (4). However, planes of weakness could result if any oxide present on the particle surfaces was not completely reduced prior to the forging operation. It could also be argued that the mechanical properties of forgings produced from low density preforms are lower than those produced from preforms of higher density and that excessive flow during the deformation could cause hot tearing. Since the danger of cracking outweighs the advantage of easy flow at low pressures, it may be best to use a preform with intricate detail and close tolerances. When complex components are to be forged, the problem of balancing the forces so that uniform density results at the end of the stroke arises. In this case a high density preform would be more suitable as the movement of material in sheltered areas need not be so high. The actual forging step and necessary tooling would be simplified substantially, better dimensional control could be achieved but it would generally be more expensive to produce this type of preform. It is therefore clear that a compromise must be achieved between the two possibilities to obtain a forging which has the optimum desired properties.

Davies and Dixon (32) maintain that one of the main criteria of preform design is to avoid the flow of metal between two different sections of the component (such as barrel and stem) during the forging operation. If this cannot be avoided then there is a possibility that cracks or folds may occur at the change of section. This means that the mass of metal in the barrel must be the same as that required in the final forging so that it is the initial preform density that controls the height of the barrel; in a similar way the width of the stem can also be controlled.

Halter (28) used a plasticine model to find the optimum preform shape to forge a gear so that the best properties could be obtained. He observed that with a slug type preform a high flow gradient occured which could lead to the formation of shear cracks and the occurrence of lapping; this would mean excessive die wear. On the other hand, a fully developed preform did not give enough flow resulting in a density distribution across the gear. From these results, Halter concluded that because of ease of handling and best flow pattern, the intermediate shape of a partially bevelled preform would yield the best forgings.

Similar work has been carried out by Kuhn and Downey (29) on both the slug and embryo type preforms. In the former case they propose that not only does the shearing action aid the normal pressure in closing up pores, but it also increased the bond strength between the opposite sides of the collapsed pores, thus giving an enhancement in the soundness of the forging. Tests showed that impact resistance increased as the amount of flow involved in reaching full density increased. However, until the expanding surfaces reach the die walls there is an increased likelihood of fracture by the development of tensile hoop stresses. Once again a compromise between the basic shapes is recommended for the production of a sound forging with optimum properties.

Recent reports by Bockstiegel and Bjork (30) and Kuhn and Downey (31) have shown that valuable indications of the features to be incorporated or avoided in preform design can be obtained from simple and relatively inexpensive model experiments similar to those previously mentioned (28), (32), (29).

Section 2: The Influence of Preform Density and Weight Control

The influence of preform density is closely related to that of preform shape and these factors must be given considerable attention if powder forging is to succeed. Preform weight is also a critical factor in that it must be controlled to within ± 1% if the desired dimensional control and density are to be achieved. Too little powder would result in forgings that are not fully dense, too much powder would damage the apparatus. The preform density must be adequately controlled to minimise dimensional and density variations of the hot preform. The former is particularly important with regard to rapid insertion of the hot preform into the die.

Preform density or rather the porosity is the advantage this process has over conventional forging in that the forgeability of the material is vastly improved. It is the presence of porosity and concomitant work hardening that are responsible for this improved forgeability. Two processes are involved in the compaction of metal powders (33); bulk movement of the individual particles at relatively low pressures, and then plastic deformation of the individual particles. To achieve high green densities, a significant degree of plastic deformation is expected, and has been assumed by many to occur. Increasing the compaction pressure increases plastic deformation and also the resultant work hardening of the particles which means that unless compaction is done at elevated temperatures, achievement of high green densities becomes increasingly more difficult.

The problem of oxide penetration has been thoroughly investigated by Cook (34); he observed that rapid oxidation occurred at 1140° C (1413 K). For low density preforms oxidation had taken place on all particle surface within 2 seconds at this temperature. High density preforms were only oxidised to a depth of 0.070 inches for the same time at the same temperature. He recommended coating the preforms with graphite before forging as a solution to this oxidation problem. Retention of the preheating or sintering atmosphere in the preform porosity gives same protection from oxidation and if the preform is forged quickly enough this atmosphere can protect both the interior and surface of the preform.

Work done by Dower and Miles (72) suggested that up to a limit this internal oxidation is beneficial to the properties of the final forging. They observed a decrease in tensile strength and yield point of extrusion forged powder billets (Fig. 3) it was suggested that this was due to a dispersion hardening effect caused by internal oxidation. Because of the low porosity of the more dense forging, less oxide penetration occurred giving a decrease in strength. Similar observations were made at higher temperatures - increased strength was noticed due to the greater oxidation tendencies at elevated temperatures.

However, most workers have concluded that preform density has little, if any effect on the properties of hot forged compacts (25), (35), (36), (37), (38). Cull (26) discusses the optimum density for powder forging and points out that a few workers favour the high density range, especially when forging powders which do not contain combined carbon or those without a protective coating. Use of low density preforms permits greater movement within the die cavity i.e., greater plasticity is exhibited than at higher densities. However, in this case problems of hot shortness occur due to the tensile forces set up during the forging deformation, shear stresses can also cause cracking of low density preforms. A compromise must be found as flow is often desirable because it facilitates the design of complicated preforms and permit the use of less expensive compacting tools (38). It must also be realised that porous preforms cannot give the same degree of strength as a solid one. The former is more likely to crack when subjected to tensile stresses. These stresses can be kept at a minimum by matching the design of the hot forming tool with an adequately designed preform.

Results obtained by Moyer (39) are in agreement with these observations. He realised that flow is an important criteria in the hot forging of powder compacts so he investigated the effect of forging

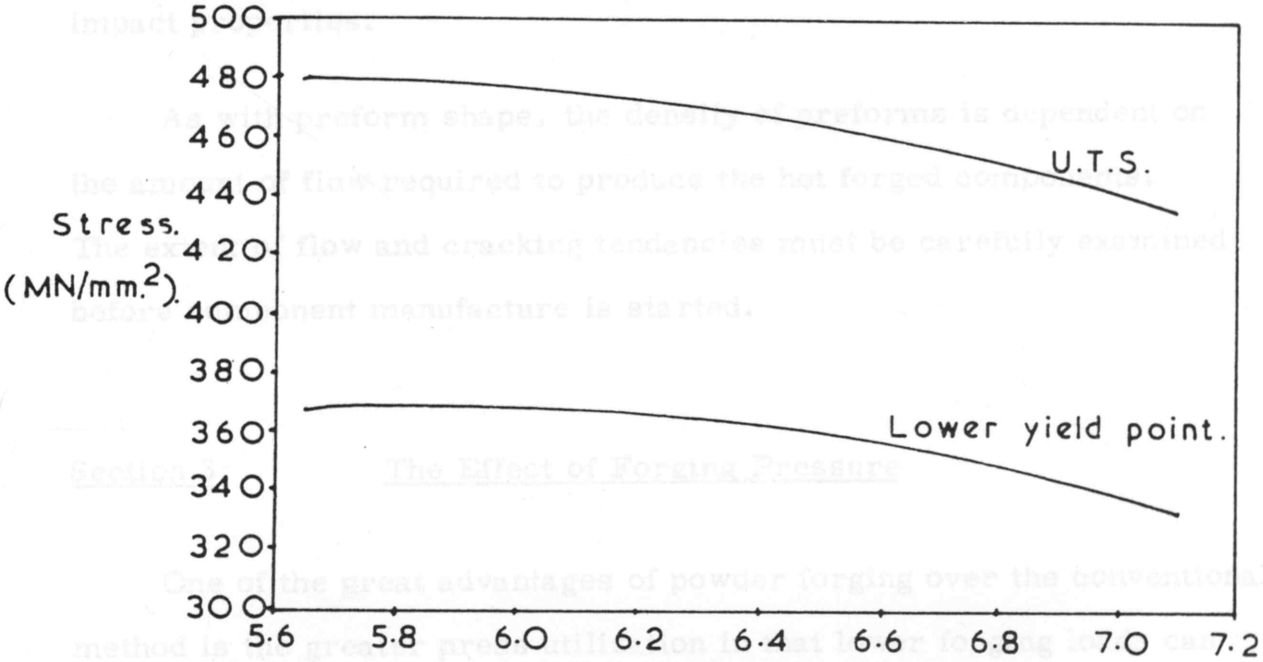


Fig. 3 Effect of initial density on the tensile properties of specimens extrusion-forged from green billets of sponge iron.

(Ref. 72)

different density preforms. Higher density preform densities were found to achieve close to full density with lower strain, therefore needing additional work to create the necessary flow. Flow is needed to promote bonding at interfaces resulting from pore closure. In the case of low density preforms most of the work given initially is spent in collapsing porosity. Although the amount of deformation given to the forging is severe, little flow or shear occurs within the deformed preform and again insufficient bonding occurs. It is clear then from the above how an intermediate density preform gave the best impact properties.

As with preform shape, the density of preforms is dependent on the amount of flow required to produce the hot forged components. The extent of flow and cracking tendencies must be carefully examined before component manufacture is started.

Section 3: The Effect of Forging Pressure

One of the great advantages of powder forging over the conventional method is the greater press utilization in that lower forging loads can be used than those needed for solid billets.

Although the absence of flash is a strong contributor to the reduced load requirements other factors also implement this reduction. Material distribution is minimised as metal can be distributed in the preform to where it is required, and the marked difference between forging characteristics of a hot powder preform and wrought steel also induce a reduction in load requirements. The load required depends on a number of factors such as forging temperature, material used and the shape and density of the preform. Halter and Rajan (76) pointed out that the pressure needed to achieve a specific density could be significantly raised by using the correct die wall lubricant during the operation. Fig. 4 shows a comparison of pressure/density relationship for a test piece and a component.

A preform in the low density condition and at the appropriate temperature starts to deform under relatively low forging pressures and it is in this early stage of hot compaction that most of the component detail is achieved. In fact very little flow takes place in the final stages. As the theoretical density is approached then the forging requirement rises - indeed, there is need for a significantly rapid rise in load exserted to remove the last 2 or 3% porosity as the pore size decreases (26), (40). Temperature has a strong influence on the load requirements (38), (41), (42) and will be discussed later; the density/pressure curves level off at values that are increasingly below the theoretical density as the temperature of forging decreases.

During their studies of the forging of Aluminium preforms Buckovecky and Rearick (78) observed that the terminal pressure had an effect on the properties of the forgings. Preforms forged at 20,000 p.s.i. had lower values for tensile strength and elongation than those forged at 50,000 p.s.i. The lower pressure was enough to cause lateral metal flow to the die walls and increase the densities to at least 98.5% of the theoretical value. They suggested that interparticle bonding as well as the density was affected by the forging pressure and this pressure must be adequate to completely densify the preform. The interparticle bonding must also be sufficiently strengthened to achieve consistently reliable properties in the forging.

Fischmeister etal (43) found that the pressure forging density curves for hot forging differ from those obtained in normal cold compaction through the existence of a yield pressure below which no flow of material and hence no densification occurs. This pressure can be represented by the following equation:

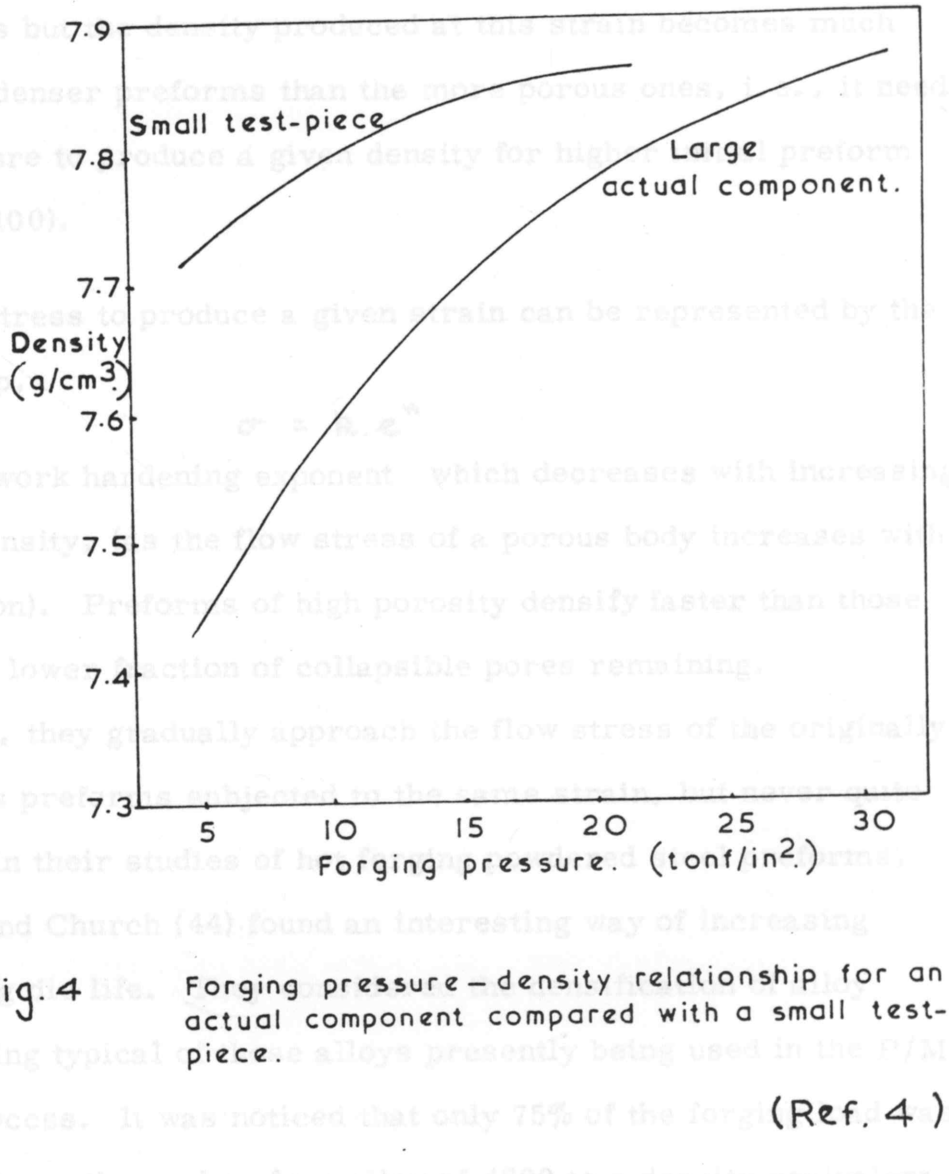


Fig. 4 Forging pressure – density relationship for an actual component compared with a small test-piece.

(Ref. 4)

$$P_y = 1.15\ (\sigma_y - K.f_p^{2/3})$$

σ_y = yield stress of the pore free material, K is a constant and f_p = volume fraction of the pores. The factor 1.15 derives from the Von Mises yield criterion for plane strain. The effect of preform density becomes more apparent as the forging pressure increases. Denser preforms require a higher pressure to achieve a given strain than more porous ones but the density produced at this strain becomes much higher for denser preforms than the more porous ones, i.e., it needs less pressure to produce a given density for higher initial preform densities (100).

The stress to produce a given strain can be represented by the relationship,

$$\sigma = k.e^{n}$$

where n = work hardening exponent which decreases with increasing preform density, (as the flow stress of a porous body increases with densification). Preforms of high porosity densify faster than those that have a lower fraction of collapsible pores remaining. In doing so, they gradually approach the flow stress of the originally less porous preforms subjected to the same strain, but never quite reach it. In their studies of hot forging powdered steel preforms, Donachie and Church (44) found an interesting way of increasing P/M forging die life. They considered the densification of alloy 4640 as being typical of those alloys presently being used in the P/M forging process. It was noticed that only 75% of the forging load was needed to forge the carbon free alloy of 4600 to a density equivalent to that of 4640. For carbon free modified 4600, only 40% of the forging load needed to produce an equivalent density in 4640 was used. Similar results had been found by Pietrocini (59) who reported that die life was increased from 20,000 parts per die to 50,000 parts per die by reducing the forging load from 70 t.s.i. to 30 t.s.i.: (57% reduction). A density change from greater than 7.8 gm. cm^{-3} to 7.6 gm. cm^{-3} was observed with this reduction. However Donachie and Church (44) state that an alloy having the flow stress of modified 4600 would allow an equivalent increase in die life without an accompanying density diminishment or degradation in properties of the forged component. However, certain cirteria must be met before the forgings can achieve useful mechanical properties.

Section 4: Forging Temperature

Important contributions are made to the final forging temperatures by both the die and preheat temperatures. In some cases where the preform is relatively small compared to the die, the effect of preheat temperature can be ignored, but otherwise this factor must be taken into account.

Much work has been done on the effect of forging temperature on the final properties of the forged material and the reports are varied and some times conflicting. However, it is generally agreed that the correct choice of temperature is essential if the desired properties are to be obtained and maximum die life achieved. Failure to recognise the importance of forging temperature would result in poor mechanical and physical properties and high tool costs. The useful range over which powder compacts may be forged is governed at both ends of the temperature scale by a number of factors. At the lower end of the scale the material exhibits reduced flowability to such a level that shear cracks might be induced, higher loads would be necessary and detrimental surface porosity would occur (28). The higher pressures needed could lead to die erosion which would affect the close tolerances required over a long run in the forming of delicately shaped gear teeth (4). On the other hand, high temperatures give rise to lubrication problems and thermal fatigue causes reduction in die life when there is too great a temperature difference between the die and preform (26), (28). Another difficulty is controlling the carbon content in the heating furnace - this would mean a more expensive furnace.

Working with iron powder Aren etal (41) found that the pressure needed to give constant strain decreased steadily with increase in temperature with no particular variation in the vicinity of the transformation temperature. Also noticeable was the decrease in U.T.S. and Yield Stress with increase in temperature again with no variation in the vicinity of the phase change region. The grain size was found to be strongly dependent on forging temperature, lower temperatures giving a finer grain size. It was noticed that the strength and grain size were related in accordance with the general shape of the Hall Petch equation $\sigma = \sigma_0 + K_0 . \frac{1}{\sqrt{d}}$ so that changes in strength with temperature can be accounted for by grain size effects.

Similar changes in mechanical properties with the temperature have been observed by others (32), (35), (36), (45) and as expected an increase was observed in both the reduction in area and in the elongation to fracture with a rise in temperature.

Maclean (42) also examined the effect of forging temperature on the mechanical properties of both sinter forgings and powder forgings. At the lowest powder forging temperature not all the carbon was taken into solution, this demonstrates the temperature dependence of powder forgings, i.e. higher forging temperatures had to be employed before the powder forged properties were comparable to the sinter forged samples.

Varying results have been reported by researchers examining the hot compaction of iron powder (32), (41) (46). Whereas Aren et al (41) failed to find any variations in mechanical properties in the phase change region, Davies and Dixon (32) observed a rapid increase in U.T.S. between 900° and 950° C which was followed by a progressive decrease in strength as the temperature was raised further. A similar decrease in micro hardness was observed at the same temperature which was in keeping with the U.T.S. results obtained for open die forging.

For closed die forging however it was noticed that the lowest temperature gave the highest strength and lowest ductility (Fig. 5) - the changes in mechanical properties in this case were attributed to microstructural changes and not the $\alpha \rightarrow \gamma$ transformation as in the open die tests.

Bosse et al (46) also observed changes in the mechanical properties in the phase transformation region and noted that deformation of the iron preforms was easier at around 850° C in the α phase than at higher temperatures in the γ phase. Corresponding to this maximum density, the hardness is also a maximum and the grain size is at a minimum for the same temperature. It was proposed that these results were due to plastic deformation occurring at this temperature.

Mixtures of iron powder and carbon have also been used to observe the temperature of forging on the properties of the forged material. Bockstiegel and Olsen (38) found that in the case of Ni - Mo alloyed steel compacts, the densification achieved at temperatures above 900° C appeared to be greater for compacts containing combined carbon compared with those containing uncombined carbon, i.e. graphite. They explained that this was probably due to the fact that graphite obstructs densification and as its specific weight is lower than the surrounding steel, the overall density of the compact is reduced. Keeping this in mind, they suggested that it would be advantageous in certain cases to forge (repress) at around 850° C and to avoid the formation of uncombined carbon by heating the compacts sufficiently fast to repressing temperature and omitting the presintering stage. However, post operation heat treatments could cause the formation of combined carbon resulting in the occurrence of deleterious cavities.

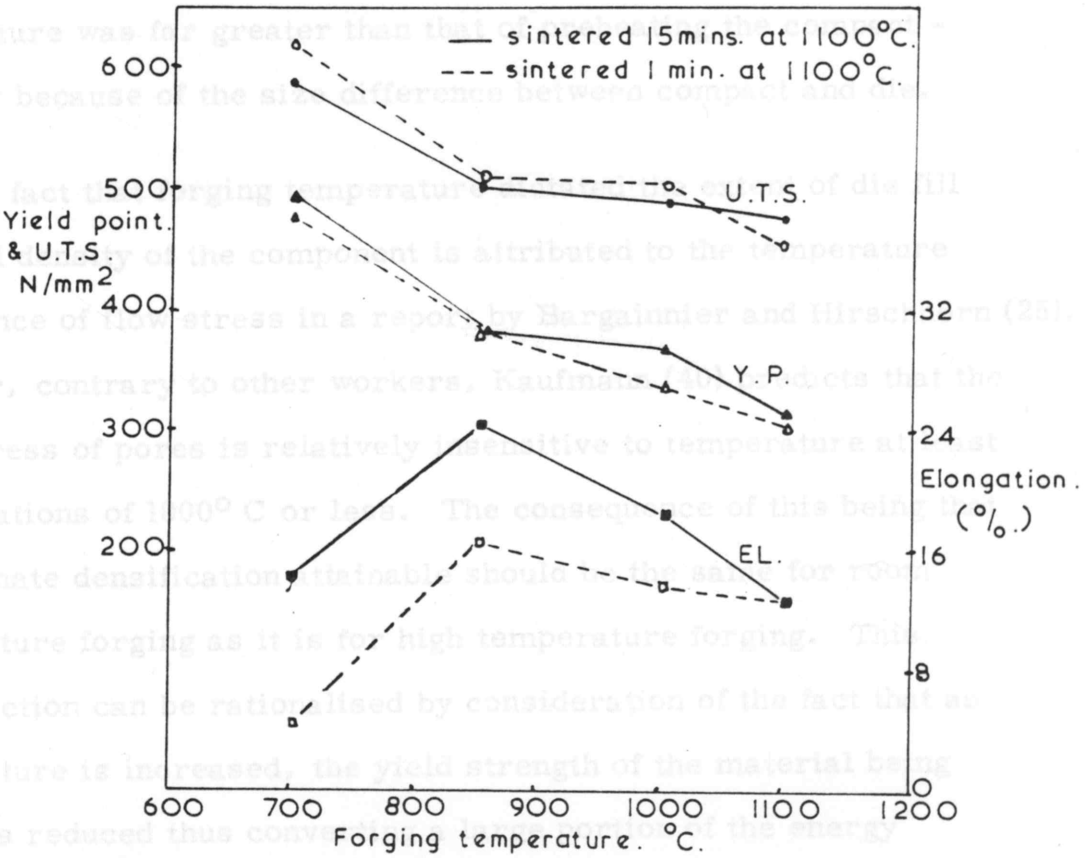


Fig. 5 The effect of forging temperature on the properties of forged preforms.

(Ref. 32)

Observations made by Niessen (35) are consistent with the above results, but in certain alloys he failed to improve on densities attained at 850° C by increasing the temperature further. Zapf (47) noted the effect of die temperature while hot recompacting an alloy of iron and copper powder. The final density was greatly increased with rise in die temperature, and as can be seen from Fig. 6 the influence of die temperature was far greater than that of preheating the compact - probably because of the size difference between compact and die.

The fact that forging temperature dictated the extent of die fill and final density of the component is attributed to the temperature dependence of flow stress in a report by Bargainnier and Hirschhorn (25). However, contrary to other workers, Kaufmann (40) predicts that the yield stress of pores is relatively insensitive to temperature at least for variations of 1000° C or less. The consequence of this being that the ultimate densification attainable should be the same for room temperature forging as it is for high temperature forging. This contradiction can be rationalised by consideration of the fact that as temperature is increased, the yield strength of the material being forged is reduced thus converting a large portion of the energy introduced by the forging sytem into plastic deformation, hence increasing deformation. There is however a limit to the pore size that can be removed and this is independent of temperature (40).

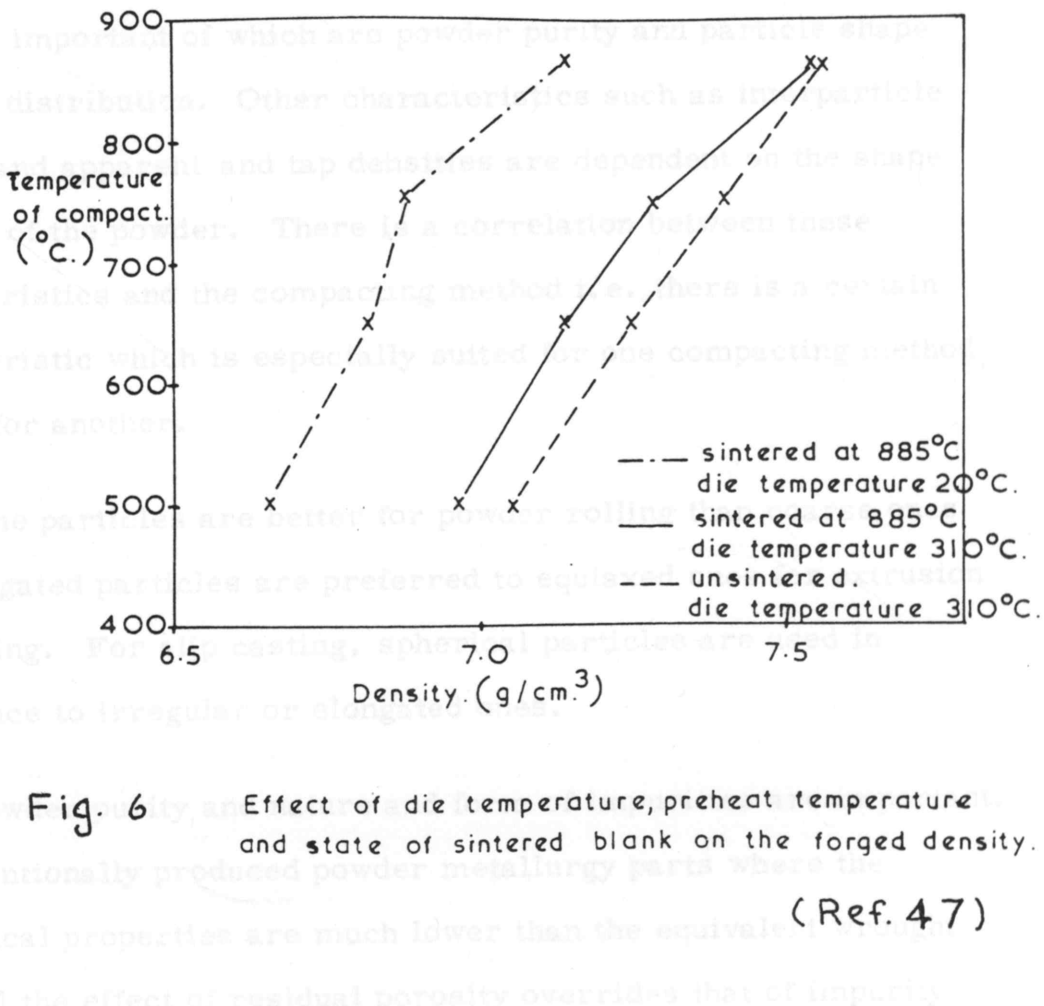


Fig. 6 Effect of die temperature, preheat temperature and state of sintered blank on the forged density.

(Ref. 47)

CHAPTER 3: POWDER CHARACTERISTICS

Section 1: Introduction

One of the main problems in the manufacture of a preform is selection of the powder and consideration of the characteristics of that powder. Powders exist with completely different characteristics, the most important of which are powder purity and particle shape and size distribution. Other characteristics such as interparticle friction and apparent and tap densities are dependent on the shape and size of the powder. There is a correlation between these characteristics and the compacting method i.e. there is a certain characteristic which is especially suited for one compacting method and not for another.

Fine particles are better for powder rolling than coarse ones and elongated particles are preferred to equiaxed ones for extrusion or swaging. For slip casting, spherical particles are used in preference to irregular or elongated ones.

Powder purity and nature and form of impurities are important. In conventionally produced powder metallurgy parts where the mechanical properties are much lower than the equivalent wrought material the effect of residual porosity overrides that of impurity inclusions; whereas in powder forging the effects of impurity and tramp inclusions are much more important.

Powder particle size distribution also plays an important role, in that choice of the correct distribution is essential if optimum compaction is to be achieved - e.g. slip casting and vibratory compaction require two completely different size distributions.

Section 2: Powder Purity

The degree of powder purity and the precise nature and form of impurities present in powder metallurgy preform are very important in that they strongly affect the properties of the final forgings. Most important is the reduction of ductility and impact strength due to the presence of impurity inclusions. These impurities tend to be introduced during the manufacture of the powder; this is usually the case for reduction techniques, as in the reduction of iron ore or mill scale. However, metals produced by other methods such as atomisation of the molten metal or by electrolytic deposition have a much higher degree of purity with hardly any inclusions (Fig. 7) Powder forging alloy powders are often formed by the atomisation technique but the use of hydrogen during the manufacturing process poses difficulties when powders with combined carbon are prepared. This is due to the extensive decarburisation that can occur.

Carbon is sometimes added in the form of flake and when this process is used the preform is sintered in the conventional manner i.e., no non oxidising or non decarburising atmospheres for a certain length of time so that the carbon can diffuse into the iron lattice. Any residual flake graphite that might exist can act as a stress raiser and cause fatigue failure early in the life of the component especially if it exists near the surface.

However, the impurity which causes most problems is that of unreduced oxide normally introduced by manufacturing processes. The effect of this oxide is clearly seen in Fig. 7 ; this shows the dependence of impact strength on density for two types of iron powders manufactured by different processes. Work done by Peissker (52) shows that unreduced oxide has a similar detrimental effect on the properties of alloy powders produced from elemental mixes or prealloyed.

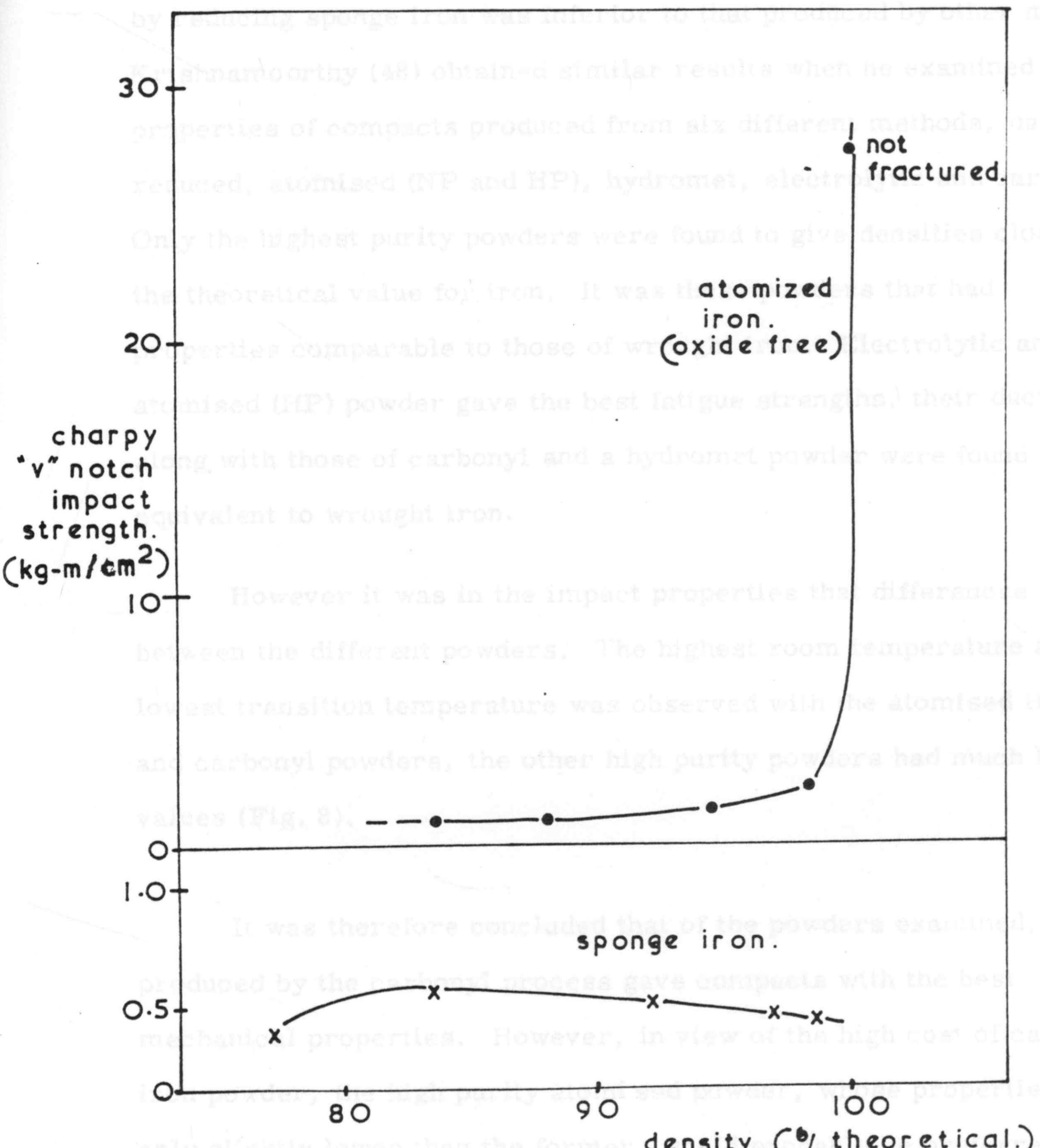


Fig. 7 Effect of density on the impact properties of two types of powder forged iron.
(Ref. 9)

Numerous researchers have examined the properties of compacts made from iron produced by different manufacturing processes (13), (47), (48), (49).

Early work by Koehring (50) revealed that iron powder made by reducing sponge iron was inferior to that produced by other methods. Krishnamoorthy (48) obtained similar results when he examined the properties of compacts produced from six different methods, namely reduced, atomised (NP and HP), hydromet, electrolytic and carbonyl. Only the highest purity powders were found to give densities close to the theoretical value for iron. It was these powders that had properties comparable to those of wrought iron. Electrolytic and atomised (HP) powder gave the best fatigue strengths, their ductilities, along with those of carbonyl and a hydromet powder were found to be equivalent to wrought iron.

However it was in the impact properties that differences arose between the different powders. The highest room temperature and lowest transition temperature was observed with the atomised HP and carbonyl powders, the other high purity powders had much lower values (Fig. 8).

It was therefore concluded that of the powders examined, that produced by the carbonyl process gave compacts with the best mechanical properties. However, in view of the high cost of carbonyl iron powder, the high purity atomised powder, whose properties were only slightly lower than the former, would probably be preferred commercially.

By promoting high purity and removing interstitials and inclusions such as oxide one ensures that the individual particle can plastically deform to a greater extent than with lower grade powders

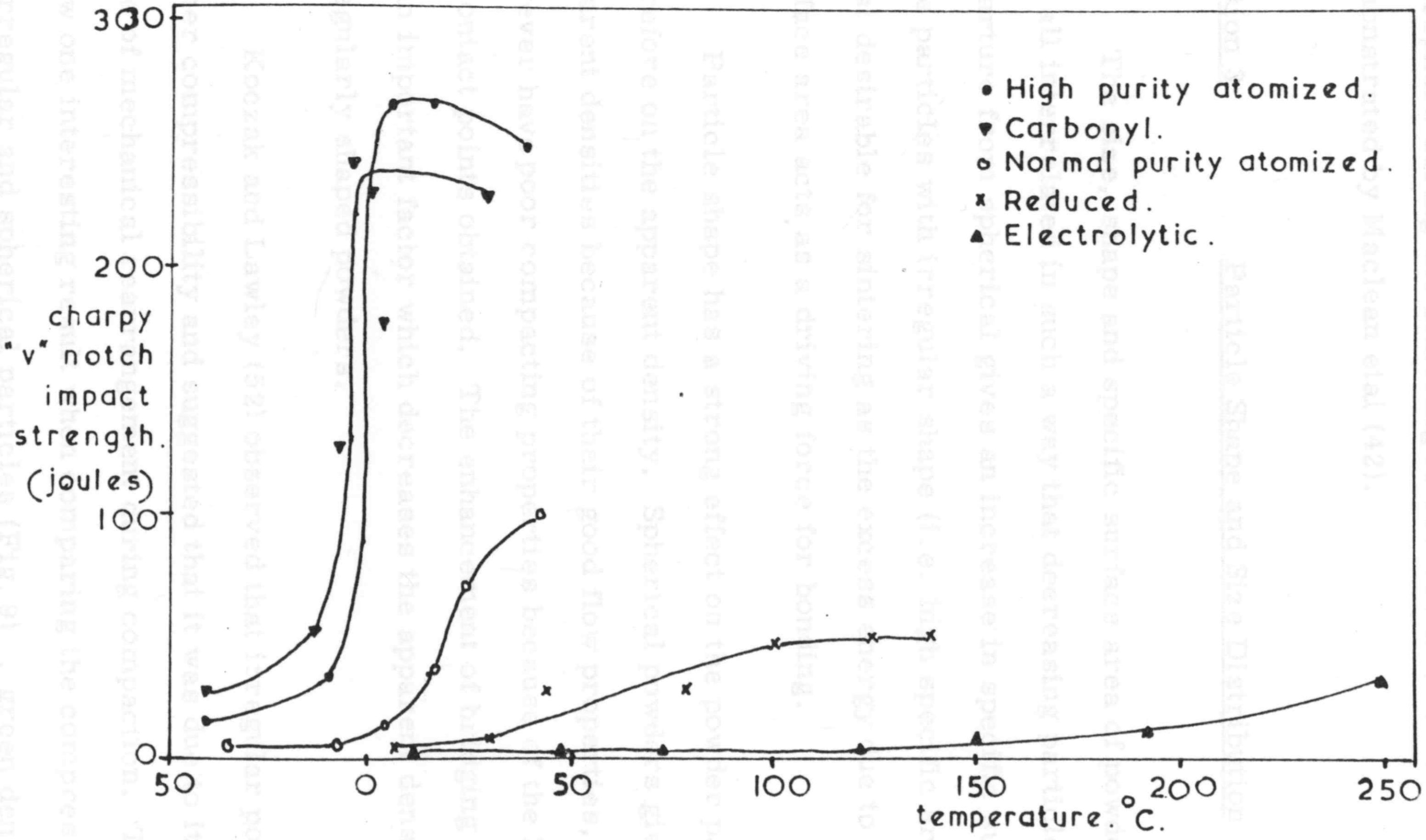


Fig. 8 Effect of temperature on the impact properties of fully dense iron compacts made from different iron powders.

(Ref. 48)

thus promoting bonding of the particles and enhancing alloying during the sintering stage. From this the benefits of using a reducing atmosphere during the sintering is obvious and has been clearly demonstrated by Maclean etal (42).

Section 3: Particle Shape and Size Distribution

The size, shape and specific surface area of powder particles are all interrelated in such a way that decreasing particle size or departure from spherical gives an increase in specific surface area. Fine particles with irregular shape (i.e. high specific area) are the most desirable for sintering as the excess energy due to the high surface area acts as a driving force for bonding.

Particle shape has a strong effect on the powder packing and therefore on the apparent density. Spherical powders give the best apparent densities because of their good flow properties, these however have poor compacting properties because of the low number of contact points obtained. The enhancement of bridging by irregularities is an important factor which decreases the apparent density of irregularly shaped powders.

Koczak and Lawley (52) observed that irregular powder showed higher compressibility and suggested that it was due to its greater ease of mechanical rearrangement during compaction. They also show one interesting result when comparing the compressibility of irregular and spherical particles (Fig. 9) , green density increases with decreasing particle size for the irregular powder, whereas for the spherical powder the green density is increased by increasing the particle size.

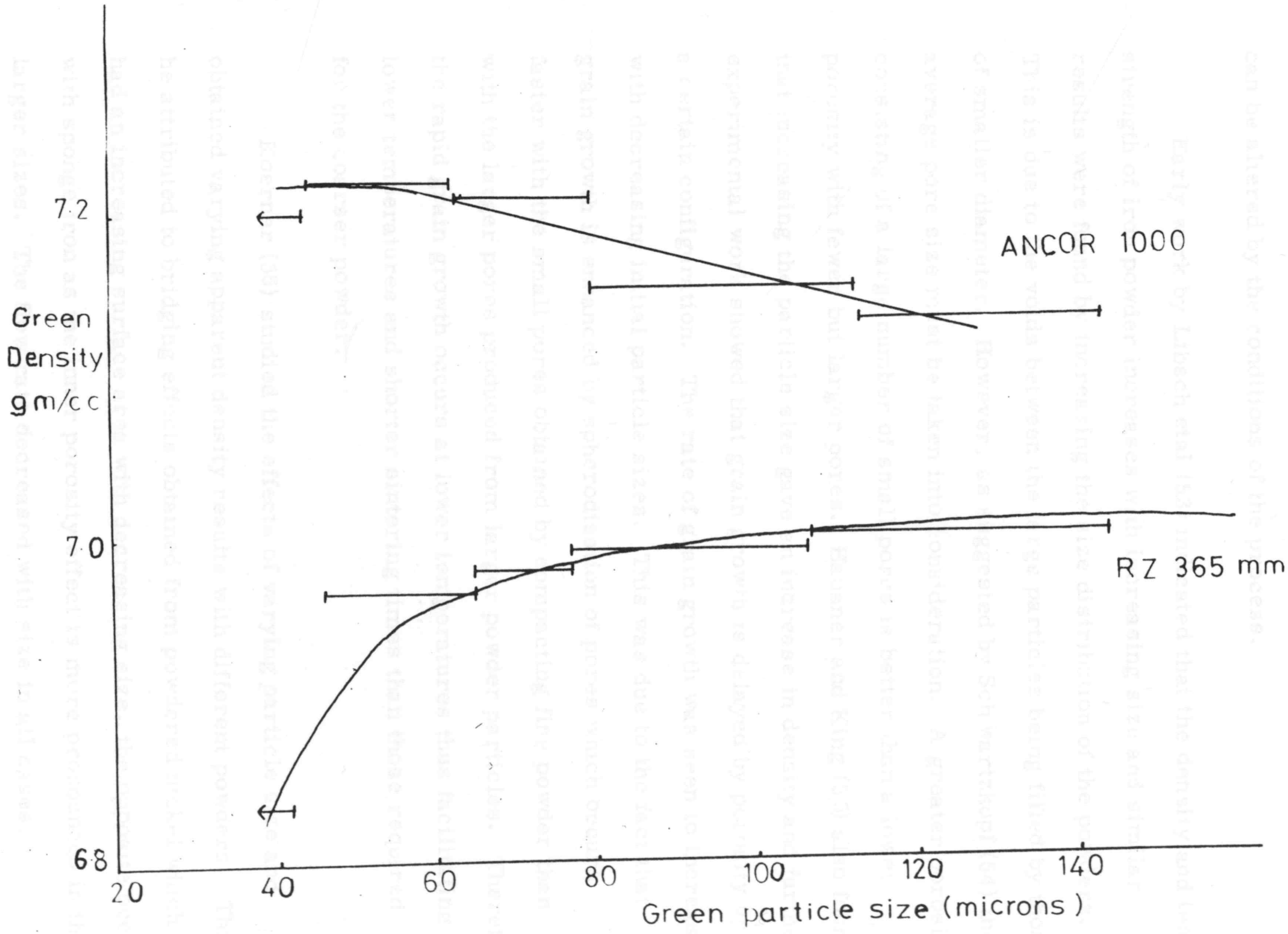


Fig. 9 Green Density vs green particle size from an irregular powder and a more spherical powder

(Ref. 52)

The shape of the powder particles is usually dependent on the manufacturing process whereas the particle size and size distribution can be altered by the conditions of the process.

Early work by Libsch etal (53) indicated that the density and tensile strength of iron powder increases with increasing size and similar results were found by increasing the size distribution of the powders. This is due to the voids between the large particles being filled by those of smaller diameter. However, as suggested by Sch wartzkopf (54) the average pore size must be taken into consideration. A greater porosity consisting of a large number of small pores is better than a lower porosity with fewer but larger pores. Hausner and King (55) also found that increasing the particle size gave an increase in density and further experimental work showed that grain growth is delayed by porosity up to a certain configuration. The rate of grain growth was seen to increase with decreasing initial particle sizes. This was due to the fact that grain growth is enhanced by spherodisation of pores which occurs faster with the small pores obtained by compacting fine powder than with the larger pores produced from larger powder particles. Therefore the rapid grain growth occurs at lower temperatures thus facilitating lower temperatures and shorter sintering times than those required for the coarser powder.

Koerner (56) studied the effects of varying particle size and obtained varying apparent density results with different powders. This he attributed to bridging effects obtained from powdered nickel which had an increasing surface area with decreasing size, the opposite occurs with sponge iron as the inner porosity effect is more pronounced in the larger sizes. The flow rate decreased with size in all cases.
According to Zapf (47) there is a slight increase in elongation to

fracture as the particle size increases, whereas Byrne and Ferris (57) only found minimal variations in properties on altering the powder size. What variation did occur was to increase ductility and mechanical properties on decreasing particle size, using specific surface as their variable.

Others have also found fine powders to be beneficial (59), (58) Bargainnier and Hirschhorn (25) found, as did others, that size was not a crucial factor and only small changes in mechanical properties occurred when the powder particle size was altered.

A recent investigation carried out by Rusnak (60) revealed that the total energy to achieve a given green density is greater for coarse powder over the density range shown (Fig.10). At densities of 93% theoretical density and above the curves approach each other indicating that relatively lower energy is required for coarse powder compaction as the density increased. The higher total energy requirement of the coarse powder for compaction is accounted for by its poor packing efficiency and not because of lower compactability. The lower energy requirements of coarse powders are more evident in the final stages of compaction.

Fine powders have a higher specific surface area which results in greater frictional forces than coarse powder. Because of this, fine powders have reduced compactability in the low density areas where particle rearrangement and localised plastic flow is important. In the later stages, densification proceeds by bulk compression and closure of isolated pores; as stated previously coarse powder will give fewer but larger pores than the fine powder because of its poorer packing efficiency.

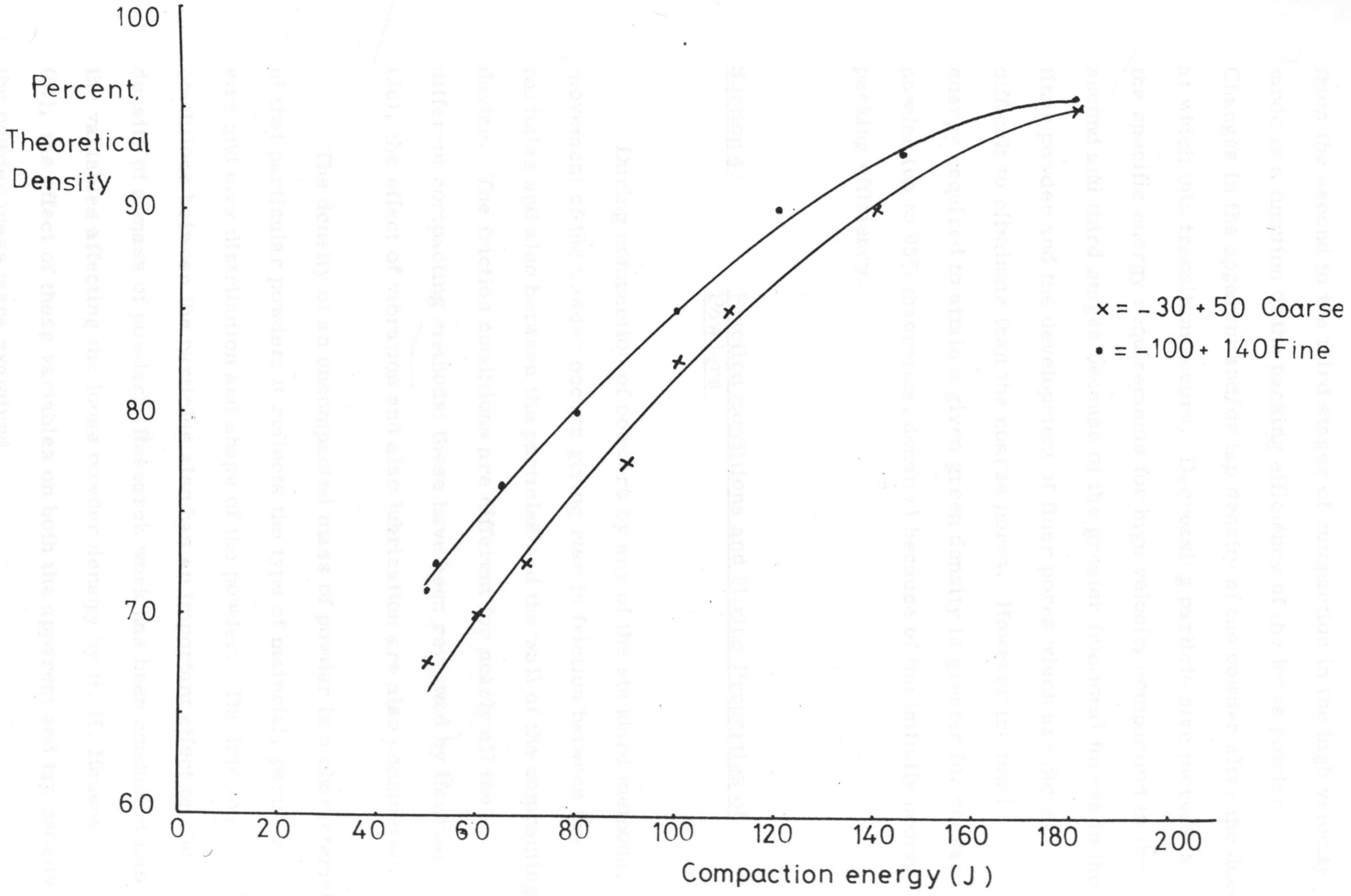


Fig.10 Compaction densification as a function of the energy expended in the high velocity compaction of coarse and fine copper powders

(Ref 60)

The conclusions drawn from this work was that the transition from the second to the third stages of compaction in the high velocity mode is a function of the packing efficiency of the loose powder. Changes in the apparent and/or tap density of the powder alter the density at which this transition occurs. Decreasing particle size increases the specific energy requirements for high velocity compaction in the second and third stages because of the greater frictional forces in the finer powder and the development of finer pores which are more difficult to eliminate than the coarse pores. However the total energy required to attain a given green density is greater for coarse powder (up to 95% theoretical density) because of the initially poorer packing efficiency.

Section 4: Friction Conditions and Mixing Properties of Powders

During compaction of powders by any of the standard methods, movement of the powder occurs giving rise to friction between the particles and also between the particles and the wall of the compacting device. The friction conditions are different for nearly all the different compacting methods; these have been reviewed by Hausner (20), the effect of vibration and also lubrication are also considered.

The density of an uncompacted mass of powder is a characteristic of that particular powder; it reflects the type of material, particle size and size distribution and shape of the powder. The friction conditions between the particles also has an important effect on the density of a mass of powder. Research work has been conducted into the variables affecting the loose powder density by H. H. Hausner (61), the effect of these variables on both the apparent and tap density of the powder mass were examined.

Apparent density was highest for spherical powder but only a slight increase was observed on tapping; on the other hand apparent density was lowest for flake powder but it was this type that showed the greatest increase on tapping. This shows that there was greatest friction between the flake powder and that it was overcome by tapping; this type of powder therefore gives a high dt / da ratio.

He found difficulty in assessing the effect of particle size due to the different levels of porosity in the powders he used. However, Rusnak discovered that both the apparent and tap densities decreased as the particle size increased, illustrating the better packing efficiency of fine powders.

Other factors such as oxide films, lubricants and media around the powder particles are also discussed by Hausner. However, another examination into the effect of particle size distribution on the friction conditions in a powder mass (62) disclosed that there is no correlation between these two factors.

Friction plays an important part in the mixing of powders. Mixing must be efficient, i.e. it must produce a homogenous mixture in the least possible time, not only for economical reasons but also because work hardening of the particles can occur and other characteristics can be altered with unduly long mixes. Three types of mechanisms normally occur during mixing, namely: 'convective mixing' where groups of adjacent particles are transferred from one location to another, 'diffusive mixing' by distributing the particles over a freshly developed surface, and 'shear mixing' where slip planes are set up within the mass of powder.

Segregation can occur in mixes as a result of differences in properties of the components - of these properties only the particle size can be satisfactorily controlled. It has been found that only a certain size ratio between the components is acceptable for complete mixing without segregation. Excellent reviews of the topic have been given by Williams (63), and Lloyd and Yeung (64).

Section 5: Some Aspects of Alloy Powders and their Preparation

There are basically three methods by which alloy powders can be produced; from elemental mixes of the individual components, partially prealloyed powders or completely prealloyed powders in which each particle is the true alloy. All three methods have their advantage and drawbacks. The advantages of using mixed powders include better compressibility and higher green strength which is important particularly in the production of bearings with low sintered density. However, several workers (13), (65) have observed poor mechanical properties when testing sinterforgings made from elemental mixes. This was because of incomplete alloying giving rise to both metallurgical and chemical heterogeneity. The only way to eliminate this heterogeneity is to use very long, uneconomical diffusion times and even this is sometimes not sufficient (4). Nevertheless there are some cases where reasonable properties have been obtained from mixtures of iron and nickel (47) as long as a suitable post forging heat treatment was given to the forgings. For the powders used by Zapf (47) this constitutes a 1-3 hour sinter at a temperature of between 1050 - 1150° C; however diminishment of mechanical properties could result from this due to the subsequent grain growth.

Some alloy powders require the addition of free graphite (26); these partially prealloyed powders incur the same problems as those of elemental powder mixes in that again, long diffusion times are essential. This is especially true in the case of addition of flake graphite to alloy steel for if any residual flakes exist near the surface of the forging then it could lead to premature fatigue failure of the component. A recent report by Koczak (66) reveals that he successfully combined carbonyl nickel with a masteralloy to give a compact with good green strength, homogeneity, grain growth and excellent precipitation characteristics. This means that lower forming loads and temperatures will be needed thus facilitating processing and achievement of full density. He therefore concluded that an alternative route does exist by using partially prealloyed powders, i.e. a combination of masteralloy and elemental nickel.

Prealloyed powders also have their disadvantages, one of these is the reduced compressibility due to the increased strength and resistance to deformation of the individual particles. This handicap can now be overcome by making green compacts of relatively low density and achieving densification by hot forging rather than using the cold die compaction used conventionally. The lower sintering temperatures and shorter sintering times provide the economic benefits of using prealloyed powder.

Use of alloy powders rather than powder mixtures often causes an enhancement in mechanical properties especially in ductility. Experiments conducted by Volgina etal (67) established that the transverse rupture strength of sintered 1 to 6% Cr ferrous materials made from 40 Kh and Kh6 prealloyed powders is much higher than that of materials of the same composition produced from mixed chromium and iron powders of the same porosity. Elongation results were also higher for prealloyed powders than those prepared from elemental powder mixtures. These results were attributed to the greater homogeneity and finer grain size observed in the prealloyed powders.

Knopp (69) obtained similar results when he compared the properties of prealloyed steels and those prepared from primary blends (Fig.11). Prealloyed materials gave higher values for tensile and yield strength but the elongations were lower than for elemental mixes. These results were echoed when similar tests were performed on prealloyed 70/30 brass, stainless steel and straight aluminium.

For the manufacture of prealloyed powders the method of atomisation is preferred over those of chemical or direct reduction methods. The difficulty with the method of water atomisation is that oxidation occurs while the particle is being quenched. After atomisation it is usual practice to anneal the powder in hydrogen not only to soften the particles but also to reduce the oxide layer formed on the surface of each particle. However, the oxides of manganese and chromium are difficult to reduce whereas oxides of other elements in alloy steel reduce quite readily. This unreduced oxide could lead to bonding difficulties during forging, but as long as the percentage of unreduced oxide is kept at a minimum no great decrease in properties should occur. As mentioned previously difficulties also arise with alloys containing combined carbon during the hydrogen reduction as rapid decarburisation takes place (26).

To overcome these problems there now exist a series of atomisation processes (70), (69) by which powders can be formed that are perfectly clean, spherical and fairly coarse. This production method is advantageous for special alloys such as superalloys, titanium alloys, tool steels etc., (nevertheless not all the problems have been conquered especially for forging of non ferrous powders as has been demonstrated by Farrell (19) and Branstedt (71)).

Overaging has been observed in some instances with the tendency to counteract the beneficial hardening during forging.

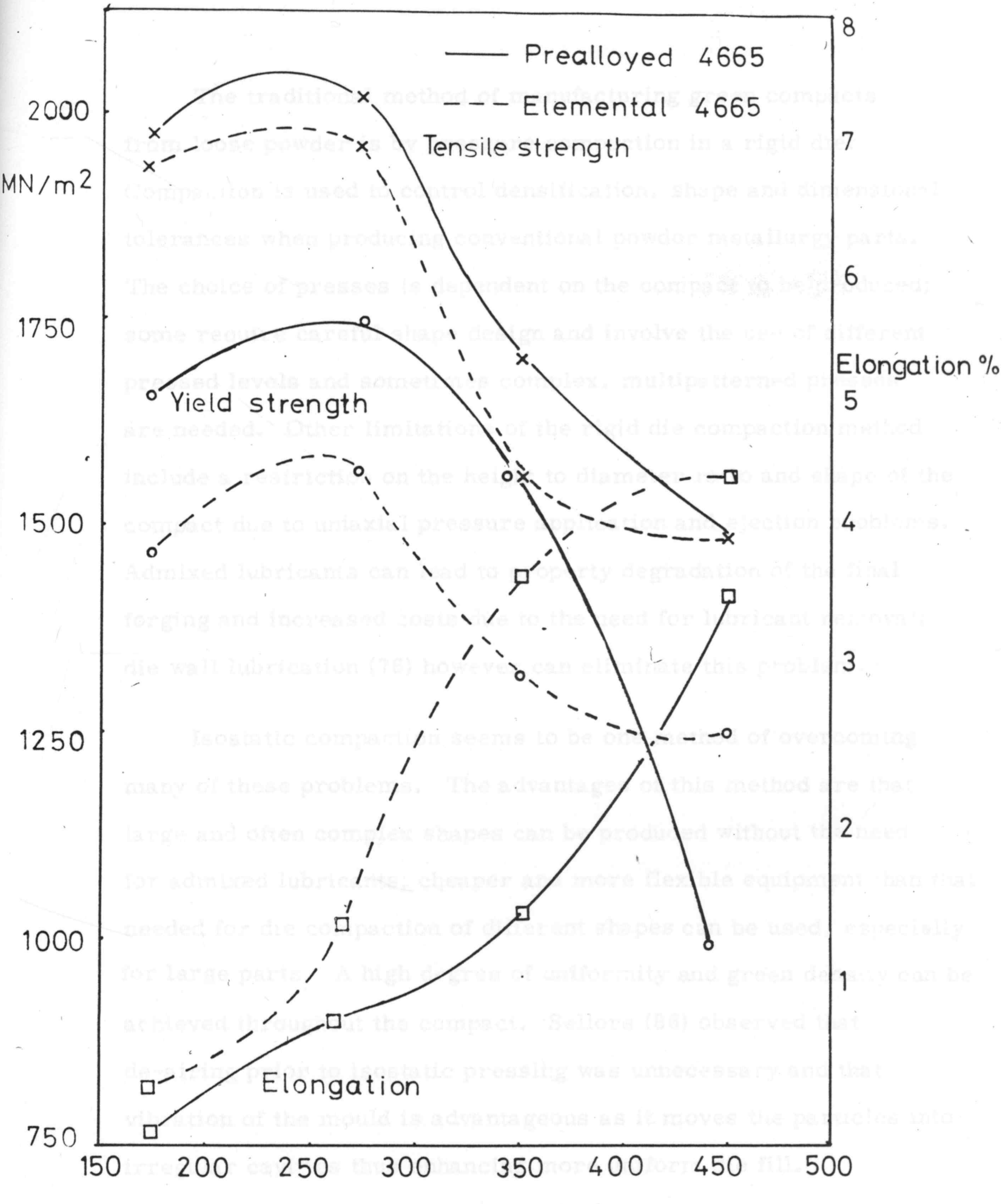


Fig. 11 Comparison of tensile properties of samples forged from steel preforms prepared from prealloyed and elemental powder

(Ref. 68)

CHAPTER 4: METHODS OF COMPACTION

Section 1: Manufacture of Preform

The traditional method of manufacturing green compacts from loose powder is by pressure compaction in a rigid die. Compaction is used to control densification, shape and dimensional tolerances when producing conventional powder metallurgy parts. The choice of presses is dependent on the compact to be produced; some require careful shape design and involve the use of different pressed levels and sometimes complex, multipatterned presses are needed. Other limitations of the rigid die compaction method include a restriction on the height to diameter ratio and shape of the compact due to uniaxial pressure application and ejection problems. Admixed lubricants can lead to property degradation of the final forging and increased costs due to the need for lubricant removal; die wall lubrication (76) however can eliminate this problem.

Isostatic compaction seems to be one method of overcoming many of these problems. The advantages of this method are that large and often complex shapes can be produced without the need for admixed lubricants; cheaper and more flexible equipment than that needed for die compaction of different shapes can be used, especially for large parts. A high degree of uniformity and green density can be achieved throughout the compact. Sellors (86) observed that de-airing prior to isostatic pressing was unnecessary and that vibration of the mould is advantageous as it moves the particles into irregular cavities thus enhancing more uniform die fill.

Basically, the process involved sealing the desired powder in a membrane or bag and subjecting the whole to a hydrostatic form of pressure. A liquid is usually used as the pressure transmitting medium, although other media such as plastics and gasses have been used. In cold isostatic pressing pressure can be applied by direct compression or from a pumped system. The former is more amenable for high pressure attainment and involves less complicated tooling and die design, however removal of tooling and compact extraction can be lengthy resulting in slow production rates. Because of this pumped systems are generally favoured. Two types of bag tooling are recognised, namely wet and dry bag tooling (87).

The former involves immersing the flexible mould which contains the powder into the pressing fluid in a pressure vessel. After compression the compact can be removed from the flexible mould; although not essential, de-airing is sometimes employed to prevent cracking in the more brittle type of powders. The attractions of this method are the low tools and equipment costs and the ability to press a variety of shapes in the same vessel simultaneously. However, the main drawback is the long time involved in the stripping and ejection of the compact.

In the dry bag technique, the bag is permanently sealed in the pressure vessel and the fluid is never contaminated with powder. The same bag can be used repeatedly and as long as a simple shape is maintained, high production rates can be achieved.

It can therefore be seen that poor dimensional control and low production rates are the main disadvantages of the isostatic pressing technique. The former problem can be overcome in the forging stage and the latter by advances in equipment design. A review of the use of hot isostatic pressing has been given by Dr. F. A. Thompson (88). In the superalloy field, powder metallurgy and hot isostatic pressing offer a number of advantages over conventional techniques. Hot isostatic pressing is preferable to cold isostatic pressing when prealloyed powders are used because of their reduced compressibility. Not only are the benefits metallurgical, but also economical, especially in the manufacture of turbine discs. The manufacture of gas turbine blades by this method is also rapidly becoming a reality.

Another method of powder consolidation is that of slip casting in which a slurry of powder and chemicals is poured into a permeable mould through which most of the liquid will pass without the application of pressure. Low density compacts which are usually treated to remove moisture and residual chemicals result from this method. Its main attraction is the ability to produce complicated shapes from powders with poor compressibility such as spherical prealloyed powders.

A new method for making preforms which are to be hot forged into fully dense high strength parts is the Loose-Pack process (89). The process uses low cost, unannealed powder and eliminates the pressing and associated stages employed in conventional methods. This is considered the main advantage of the technique. Essentially, the process involves pouring a mixture of powder and binder into a consumable mould and vibrating to maximum density - sintering and hot forging then follow to complete the process. Tests have shown (89) that low alloy steel forgings made from the Loose-Pack process were comparable to those made by conventional methods; the properties also compared favourably with those of wrought steel.

Ultimately it is the powder characteristics and desired shape and properties of the compact that determine the compaction method employed.

Section 2: Forging Methods

There are at least three methods by which a material can be hot forged, these are by; hydraulic pressing, mechanical pressing or by using a high energy rate press such as the Petro-Forge. Hirschhorn and Bargainnier (9) suggest that 'it may be best to have a rapid initial application of force to the preform followed by a relatively slow, high pressure squeezing action'. In this way the material can be moulded to its final shape due to the initial weakness of the preform. Cull (26) states that the most generally preferred compaction method is by mechanical pressing; hydraulic compaction being too slow and the high energy rate method delivers the maximum load at the point of impact and not when most of the required work has been done when the greatest load is needed.

Significant results have been obtained, however, with a Dynapak machine which delivers extremely high levels of energy to the powder mass within a few milliseconds (90). High green densities have been obtained even with powders most difficult to compact by conventional techniques, probably due to optimisation of particle plasticity. The advantage of this process is that cheaper and coarser grades of powder can be used and the cost of H.E.R.F. equipment is lower than that of conventional presses.

Davies and Dixon (32) reported that from their experiences with use of the Petro-Forge high speed machines, preforms could be forged within a very short time interval after removal from the controlled atmosphere furnace, thus reducing oxidation and chilling effects. This introduces the possibility of reducing the physical size of the equipment and of cutting the capital costs of the operation.

Samples compressed by H.E.R.F. and sintered to densities identical to those produced by conventional techniques show higher mechanical properties. This is probably because the high rates of loading lead to local plastic deformation on the contact surfaces, thus uncovering new surfaces resulting in a greater number of metallic contacts and hence better bonding (91). With explosive compaction the plastic deformation of the particles increases, leakage of metal occurs into the pores and there is evidence of some texturing. This increase in velocity causes the deformed particles to alter the flow of metal into pores in the form of a jet of material. The result of this is that any oxide present on the particle surfaces is severely disturbed and fragmented giving improved particle bonding. A technique has been developed that combines the advantages of hot pressing and direct explosive compaction methods of consolidating powdered materials (92). By hot explosive compaction above the recrystallisation temperature of the material, high density compacts with high mechanical properties can be obtained without the need for high temperature sintering. Another advantage is the possibility of compacting poorly preserved powders.

Guest et al (27) report that at high speeds more pressure was required to achieve a given density for low density preforms than higher density ones. The effect of speed was only noticeable at intermediate temperatures. It was generally observed that the more complex the part, the slower the compaction rate because of the difficulty in filling the die cavity and the complexity of the pressing motions. Others have reported (38) however, that the speed of compaction was significant only at the lower temperatures where a higher compacting rate resulted in less porosity.

CHAPTER 5: HEATING OPERATIONS

Section 1: Heating to Sintering Temperature

Here arises a problem that involves combining the old and new forging processes. Conventional metallurgy forging plants do not have, as a rule, controlled atmosphere furnaces, and neither do powder metallurgy plants have forging presses. It is therefore quite unusual to find both furnaces and presses side by side on the shop floor. This means that a great deal of trouble and expense is likely to be incurred during the conversion to powder forging. Not only this, but additional space would also probably be needed to house the new apparatus.

A simple and attractive solution to the problem would seem to be the use of induction heating. Several investigators have demonstrated the advantages of this alternative method of heating (73), (74), (75). Hirschhorn et al (73) observed that induction sintering for adequate periods develops tensile strengths equivalent to or greater than conventionally sintered materials at the same density levels. Induction heated specimens are also characterised by a finer grain size than that observed conventionally. The ductilities were found to be lower but the hardness greater than those of conventionally produced materials; this is explained by microstructural differences due to the shorter times at temperature and faster cooling rates. For many potentially useful alloys, temperatures of commercial furnaces are not high enough to produce adequate alloying in reasonable times. Therefore, by using induction sintering, many more alloys can be brought into powder forging.

Use of this rapid induction heating would appear to be a much more appealing alternative than to use the cumbersome, space consuming sintering furnaces used conventionally. This smaller heating unit would also save on atmospheric control costs. Knopp (75) made a comparison of furnace heating versus induction heating for both elemental and prealloyed powders. He found that the hardnesses were similar and that the carbon had diffused in the times used. This is of great importance as was discussed by Cull (26), because free graphite present in the final forgings could result in a serious degradation in the fatigue properties of the forging. The ultimate tensile strengths and yield strengths were similar for both the furnace and induction heated compacts. However, the ductility and impact values were lower for the induction heated preforms; probably due to the fact that metallic additives did not completely diffuse due to the short times used. A non uniformity was also observed in the grain structure of the induction heated preforms.

Vernia (74) has also examined the prospects of short cycle sintering by induction heating. His results revealed that a remarkable degree of strength and carbon solutioning could be obtained throughout a reasonable range of temperature in a very short time. Temperature limitations usually attached to conventional sintering are eliminated as only the sample and a very small amount of fixturing is heated to high temperature. It is therefore obvious that the advantages of induction heating go beyond those of reduction in capital equipment and ease of adaptation to automation. These include a potential for faster cooling (thus enhancing properties), reduction of furnace volume and improved cleanliness due to faster cooling. There are also reports that successful induction hardening can be carried out without a protective atmosphere if the compacts are firstly dipped into colloidal graphite. Maclean et al (42) commented that the rate of oxide reduction increases rapidly with temperature, thus the use of induction sintering at high temperatures for short times would appear to be beneficial especially if the minimum preform density tolerable is used.

Probelms do arise with induction sintering, however. Parts that depart from spherical pose heating problems and the high rate of cooling often induces cracking, especially in unsintered preforms. This might be due to evaporation of the lubricant, if so, a die wall lubricant can be used as suggested by Halter and Rajan (76). Since the outside gets heated more than the inside, time must be allowed for temperature equalisation after removal from the furnace, this might cause oxide absorption to the outer layers.

Presently, however, many workshops prefer to use conventional methods for heating using a controlled atmosphere. In this case, if transfer from the furnace to the die is sufficiently rapid, gas entrapped in the outer pores protects the compact from any undue oxidation.

Section 2: Lubricant and Binder Burn Off

Most conventional powder metallurgy establishments have a sintering apparatus which incorporates a "burn off" stage to accommodate for the removal of admixed lubricants introduced during the cold compaction stage, especially when the preform is made by compression in a rigid die or by slip casting. The operation and principles of the burn off stage have been reviewed excellently by Marshall (7). It is essential that these admixed lubricants and other chemicals are removed as they are deleterious to the properties of the final forgings. Although these lubricants aid the compaction and ejections of the preform, they have many disadvantages. The amount of lubricant removed decreases as the compacting pressure increases due to the closure of interconnected porosity. Any chemicals or lubricants that remain on the particles reduce the number of particle contacts giving poorer interparticle bonding. This in turn leads to lower sintered strength and also promotes a large amount of interconnected porosity on completion of the sintering. Because of this high amount of interconnected porosity, severe internal oxidation may occur if the hot compact is exposed to air prior to the forging operation (34). On forging this oxide and residual chemicals would remain on the grain boundaries causing a serious degradation in the mechanical properties of the forged product. The larger the compact, the longer is the time required to remove the lubricant which means that larger furnaces must be used and more heat and energy expended. Admixed lubricants are especially undesirable in the case of induction heating operations as the high rate of heating causes the lubricants to boil and often produces cracking especially in unsintered compacts (23).

Halter and Rajan (76) recommended the use of die wall lubrication as an alternative. Not only is there a saving in the volume of lubricant used, but there are also other desirable advantages. The most important of these is the almost complete elimination of the "burn off" stage; as all of the lubricant is situated at the surface it can be removed easily and quickly. This means that more space is available due to reduction in furnace capacity and more important, there is a great saving on energy expenditure. Higher production rates would also result from the elimination of this previously slow and uneconomical stage.

Isostatic pressing is another solution to the problem because it can be performed without the need for lubrication as has been previously mentioned.

Section 3: Sintering and Homogenisation Treatment

The main purpose of the sintering operating is to transform the green compact, which consists of individual particles, to a mass in which the particles have lost their identity, and recrystallisation and grain growth have taken place across the former grain boundaries. When compacts are made from alloys of elemental blends then sintering can also act as a homogenisation process. In an excellent review of the topic, Thummler and Thomma (77) proposed a definition for sintering: 'By sintering is understood the heat treatment of a system of individual particles or of a porous body, with or without the application of external pressure, in which some or all of the properties of the system are changed with the reduction of the free enthalpy in the direction of the porosity free system. In this connection, at least enough solid phases remain to ensure shape stability.'

During sintering, metal particles of different orientations come into contact with each other producing areas of lattice instability, these areas act as recrystallisation nucleii. In the case of solidified metals, boundaries are produced at the temperature of solidification which means they are stable at or below the solidifcation temperature. In compacts produced from powders at room temperature, increased atomic mobility at increased temperature will enable the atoms to assume a more stable arrangement at the contact areas than the ones produced by accident during compacting. This means that recrystallisation effects are induced in the same way as with instabilities from work hardening (55). The number of recrystallisation nucleii is therefore dependent upon the amount of cold work introduced during compaction which in turn depends on the pressure, plasticity size and shape of the particles and the pressure distribution within the compact. Grain growth is hindered by the presence of oxide films and voids; but as sintering continues the process of necking reduces the amount of connected porosity giving stronger particle bonds and producing a more coherent skeleton (41).

Thummler and Thomma (77) observed three distinct stages in the sintering process which merge continuously into one another. In the first stage the particles remain as individuals since grain growth beyond the original particle is not possible. The tensile stresses from the surface tension maintain the grain boundaries between the particles that are adjacent in the plane of contact but the formation of an occasional new grain is possible. The second stage witnesses the greatest amount of shrinkage with the formation of a coherent network of pores and a continuation of grain growth. Further spherodisation of pores occurs in the final stage and densification proceeds so slowly that it is often difficult to decide whether the process is complete and the residual porosity is permanent (40), (77).

Many diffusion mechanisms are operational during the sintering procedure (77) and in general, the diffusion rate of an individual metal into a given lattice is higher, the closer the temperature is to the melting point. Indicating that in a mixture of elemental powders the component with the lowest melting point will diffuse fastest. It has been observed by many (78), (79), (80) that during the sintering of mixed powders, the particle size is a critical factor. Homogenisation rate is fastest when the particles of the minor constituent are smallest, because the number of large pores is less providing less obstacles, thus enhancing diffusion. Gas films also act as barriers to diffusion but these can be removed by employing a suitable atmosphere. The extent of homogenisation can be measured by the electrical resistance of the material (79), the maximum occurring just before complete homogenisation. In some cases, homogenisation is not completed and sintering is interrupted to impart the desired properties on the compact. An alloy of 90 - 10 Cu - Ni powder was found to be completely homogenised after sintering for approximately 30 hours at 1000° C (1273K) (79). This observation was made for particles of -300 +325 mesh, increasing the particle size increased the time needed for complete homogenisation.

Aren et al (41) studied the effect of presintering in an open die forging assembly and came to the same conclusion as others (35), (47) that there was no beneficial effect of sintering. The effect was, in fact, to the contrary, that forging pressure had to be raised and the risk of oxidation increased as presintering proceeded. The resistance to deformation increases with presintering and the effect becomes quite appreciable with long presintering times. Nissen (35) observed, as did Zapf (47) that green preforms required a substantially lower amount of pressure to achieve a given density than did sintered preforms. Cundill et al (13) noticed that increasing sintering time and temperature led to an increase in strength but a decrease in ductility of blended alloys. No change was observed in the impact resistance. However, Badia et al (37) found that both the tensile and impact strength were improved with increasing sintering treatment. Other researchers investigating the effects of sintering have also observed similar results (52), (81), (42).

Therefore, a well bonded, ductile structure in the sintered preform is needed to ensure good forgeability and uniform properties in the forged product. For this, controlled sintering conditions must be followed and a suitable atmosphere employed. The function of the sintering atmosphere is generally twofold; undesired reactions such as oxidation and carburisation must be avoided, and desired reactions such as the reduction of oxides must be performed (55). The most frequently employed atmosphere is one that can reduce unwanted oxides from the surfaces of the powder particles. If left unreduced these oxides would seriously affect the properties of the final forging. Not only does this controlled atmosphere perform the necessary reactions during sintering, but it also helps to protect the compact during transfer from the furnace to the forging press (34).

The economics of the sintering operation is a limiting factor affecting profitability in powder metallurgy. Delubrication, presintering and sintering are three important steps in the production of powder metallurgy parts and if the cost of these could be reduced then the economics of the process would be improved. Hallemeier (82) has suggested a solution to the problem; he proposed that use should be made of a vacuum induction furnace able to perform the three operations and provide rapid cooling under optimum metallurgical conditions. In this way yield and quality would be improved and cost factors reduced.

It is generally agreed that prealloyed powders cut sintering time considerably, but these powders can prove costly. On the other hand elemental mixes need long diffusion times and oxidation problems are incurred with cheap alloying elements such as Manganese. Fischmeister and Larsson (83) describe a way of overcoming these problems. The alloying addition needed for hardenability must be made in the form of a low melting point master alloy so as to shorten diffusion times. The conditions are that the alloy must wet the base material in question (Fe in this case) reducing the diffusion distance to one particle radius, it must also penetrate quickly along the Fe grain boundaries, again to reduce diffusion distances. Low melting point alloys of copper and manganese satisfy these conditions.

The optimum situation would of course be to avoid cooling and reheating to forging temperature by combining the sintering and preheating and forging the compact once it has cooled sufficiently from the sintering temperature (9) (82).

Section 4: Post Forging Heating Operations

Various reports have shown that post forging heat treatments can be both beneficial and deleterious to the final properties of a forged powder compact. Work done by Zapf (47) revealed that a post forging treatment of between 1 - 3 hours imparted the optimum mechanical properties on the steel in question. Also apparent was that unsintered compacts were much more compressible than those sintered before forging. Nissen (35) also has a similar recommendation after observing that sintered preforms required 35% to 70% greater load for compaction than green ones, thus increasing die and tool wear. A post forging homogenisation process has been successfully tried on Hastealloy B sheet (84) yielding acceptable properties, the major disadvantage being the additional heat treatment required to achieve the necessary amount of homogenisation by interdiffusion. Prealloyed powders respond to post forging heating operations much more readily than blends of elemental powders; it has been found that prealloyed ferrous powders can be heat treated just like wrought steels (85), (9). Inconel 713C alloy can also be heat treated to give an improvement in properties as long as the recommended procedures are followed (21). The tensile, fatigue and impact properties of other prealloyed powders have also been seen to improve with suitable heat treatments (13).

It is therefore in the blended powders that problems arise during post forging heat treatments (13), (26), it was proposed that this was due to the lack of hardenability that eliminated the possibility of the beneficial heat treatments. Cull (26) suggested that as the powder forging process is one of pure hot compaction to unit solid density in which little or no lateral movement occurs, then post forging heat treatments are unnecessary. Even if they are employed, the slightly higher austenitising rate in powder forged steels will not produce a substantial amount of grain growth. Because of the finer grain size of powder forgings, the carburising of powder forged steels does not respond in the same way as with wrought material. Inferior case hardening occurred when the alloyed steels contained slight heterogeneities but the results were quite satisfactory when the alloying elements were uniformly distributed (26).

In some cases, subsequent heat treatments such as normalising, austenitising or case hardening can be deleterious due to the formation of combined carbon. This leads to the formation of cavities that detrimentally effect the properties of the final forging.

CHAPTER 6: DENSIFICATION AND LATERAL FLOW

One of the main characteristics of importance in the forging of materials is the extent of lateral spread. During pure compression, without the effect of friction, this characteristic is expressed as the ratio of transverse strain to axial strain and is termed the Poisson's ratio of the material, ν_p (41) (93).

$$\nu_p = \frac{1}{2} \frac{d \Sigma w}{d \Sigma h}$$

In elastically deforming materials, this ratio is constant during the deformation and is a property of the material. For a solid, conventional material experiencing no volume change during plastic deformation, this ratio is equal to 0.5. However, during the plastic deformation of powder compacts, density changes do occur and less lateral spread occurs than with solid materials. This is due to the fact that some of the work of compression is used to reduce the pores in the material and flow occurs in the direction of the punch initially. Therefore during the plastic deformation of porous materials the Poisson's ratio is less than 0.5 and has been found to be a function of the current density of the material, not the initial density (94). This relationship can be written in the form:

$$\nu = 0.5 \left(\frac{\rho}{\rho_T} \right)^{1.92}$$

where $\left(\frac{\rho}{\rho_T} \right)$ = fraction of theoretical density.

Although Fischmeister et al (95) claim that the strong initial strain dependence of ν_p is not taken into account, tests on various materials showed that this relationship is obeyed in all cases except for iron powder sintered below the $\alpha \rightarrow \gamma$ transformation.

Similar tests done on sintered Aluminium powder forged at 371° C gave the results shown in Fig. 12. Here, the Poisson's ratio for a given density is slightly lower than that for room temperature deformation. This is probably due to the lower values for the work hardening exponents of hot worked materials. Lower 'n' values give lower Poisson's ratio, or more densification per increment of strain. As the density of the powder approaches the theoretical value, lateral flow is enhanced and the Poisson's ratio approaches that of a fully dense material (Fig. 13).

Therefore it can be seen that powder compacts differ from conventional forging stock in that they are compressible. Flow is produced by shear, uniaxial stresses and a hydrostatic component of the stress system (95). Thus, adaptation of the Von Mises equation and borrowing a modified Mohr-Coulomb criterion from soil mechanics, Kuhn and Downey proposed that the yield criterion:

$$f = \left[3J_2' - (1 - 2\sigma) J_2 \right]^{\frac{1}{2}}$$

where J_2 is a function of J_2', the second invariant of the stress deviator satisfied the requirements of yielding of a sintered powder material.

As has been mentioned previously, the relationship between strain and overall density was found to be unaffected by temperature even though the pressure to reach a given strain is increased (41). The effect of temperature on flow stress seems to be rather ambiguous as some found no variation with temperature (95), (41), whereas others (32) observed a sudden drop in flow stress as the $\alpha \rightarrow \gamma$ transformation was exceeded.

Kuhn et al (96) studied the macroscopic behaviour of sintered powdered metals, and from their observations they established a plasticity theory for these materials. They also made a model study of the impurity

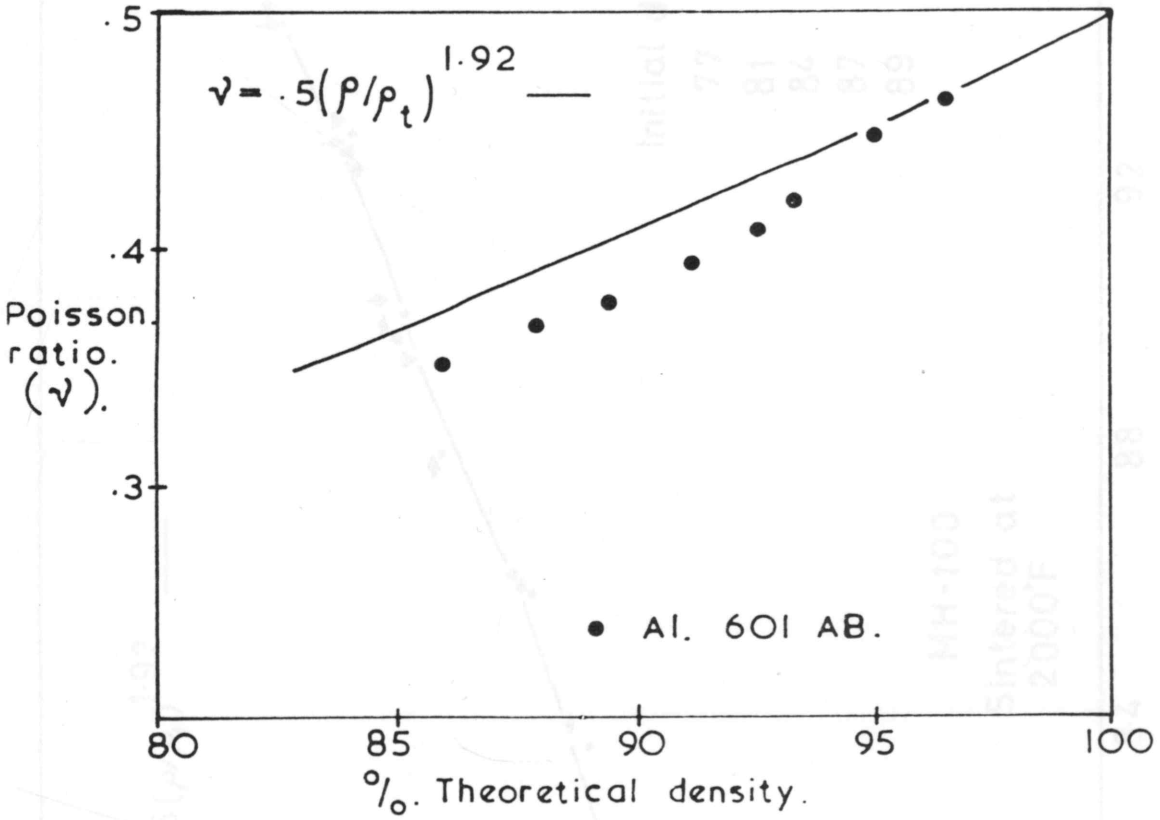


Fig. 12 Variation of poisson ratio with density for the hot forging of Al. 601 AB. preforms at 700°F(371°C.). (Ref. 93)

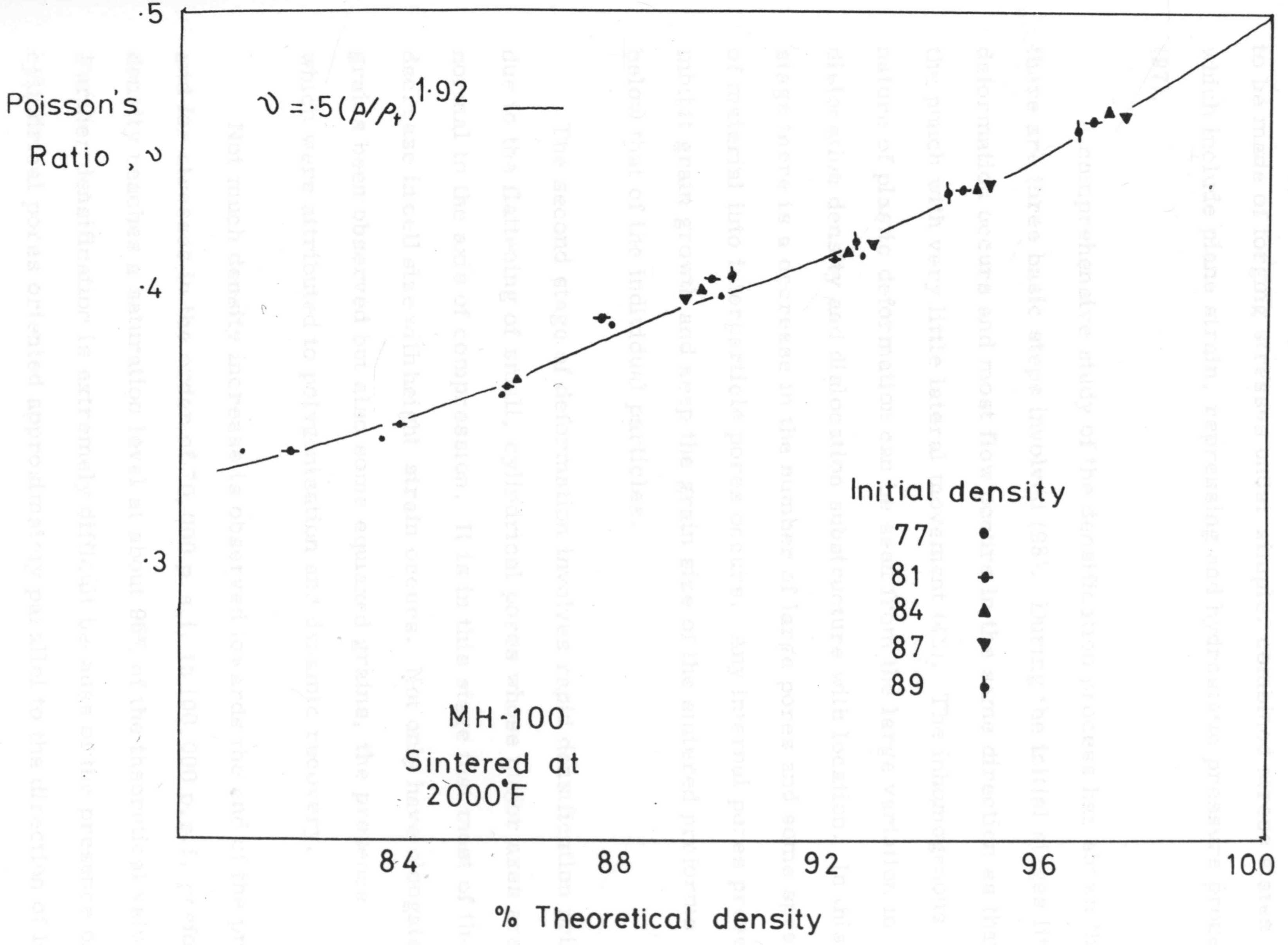


Fig. 13 Variation of Poisson's Ratio with density in iron powder compacts.

(Ref. 94)

base system in order to determine the mechanisms of densification and fracture. These observations and postulations enabled accurate predictions to be made of forging stresses under simple, combined stress states which include plane strain, repressing and hydrostatic pressure processes (97).

A comprehensive study of the densification process has shown that there are three basic steps involved (98). During the initial stages little deformation occurs and most flow occurs in the same direction as that of the punch with very little lateral movement (43). The inhomogenous nature of plastic deformation can be seen from the large variation in dislocation density and dislocation substructure with location. In this stage there is a decrease in the number of large pores and some spreading of material into interparticle pores occurs. Any internal pores present inhibit grain growth and keep the grain size of the sintered preforms below that of the individual particles.

The second stage of deformation involves rapid densification primarily due to the flattening of small, cylindrical pores whose major axes are normal to the axis of compression. It is in this stage that most of the decrease in cell size with height strain occurs. Not only have elongated grains been observed but also some equiaxed grains, the presence which were attributed to polygonisation and dynamic recovery.

Not much density increase is observed towards the end of the process and for stresses in the order of 70,000 p.s.i. to 100,000 p.s.i. preform density reaches a saturation level at about 96% of the theoretical value. Further densification is extremely difficult because of the presence of cylindrical pores oriented approximately parallel to the direction of loading, which do not flatten on compression (98).

Macroscopic observation eliminated the microscopic variations in Poisson's ratio due to local density variations, and it was seen that the Poisson's ratio increased linearily with reduction in porosity. Absence of interconnected porosity improves the rate of densification and the ultimate level achieved.

CHAPTER 7: SUMMARY OF THE LITERATURE SURVEY

It can be seen from the survey of current literature that due to its many appealing advantages, powder forging is a highly desirable operation, its greatest set back being the initial high cost of the raw materials. This is the strongest argument against the introduction and utilization of hot powder forging processes in many present day industries. The one case where it is economically advantageous is in the forging of expensive materials such as titanium and aerospace alloys. Here, the initial expense of the raw powder is balanced by the vastly improved material utilization when compared with conventional forging methods. The other main advantages of using hot powder forging include reduced machining costs, decreased die and tool wear and less noise on the shop floor.

Design of the preform is an important consideration in powder forging as it greatly influences the success of the operation. It is technically and economically better to have a simple preform shape, but it is often more successful to have a more complicated one. This is because a simple preform would need to be of low density so that sufficient flow could take place to give the desired shape. Care must be taken though as too much flow could result in planes of weakness in the finished forging. When complex components are produced, the problem of balancing forces to give uniform density arises. It is therefore clear that a compromise must be made between these two options.

Closely related to that of preform shape is the influence of preform density and weight control. These must be adequately controlled to minimise dimensional and density variation in the hot powder forging. The density, or degree of porosity of the compact is important in that it is responsible for the improved forgeability of the compact. However, as long as density fluctuations are not too great, preform density does not have a significant effect on the properties of hot forged components. As mentioned previously, density considerations are necessary when the extent of flow occurring during the operation is to be controlled.

Another advantage of hot powder forging over the conventional methods is that low forging loads can be used to give the desired shapes. It has been observed that denser preforms require greater pressures to achieve a given strain, but the density at this strain is greater than that for a similar strain given to a low density preform.

The temperature of forging dictates the extent of die fill due to the temperature dependence of the flow stress. This is one of the most critical factors and therefore it is essential that the correct forging temperature is chosen if the desired properties are to be attained. When low temperatures are used, high pressure must be used due to the reduced flowability of the material. Consequently a poor surface finish is obtained. Too high a temperature however introduces lubrication problems and the die life is reduced through thermal fatigue. It is therefore quite clear that an optimum temperature range must be found.

A further consideration that must be taken into account during the hot forging of powder preforms is that of powder characteristics, namely; powder shape, particle size and particle size distribution. The characteristics of particle shape and size are interrelated, different combinations give different final properties. The best combination for optimum sintering is to use a small and irregularly shaped powder. Spherical powders are noted for having poor compactability but good sintering properties. A greater density can be obtained by using large particles, this is because of the fewer number of pores obtained than by using fine powders. However, if the maximum density is required then a large particle size distribution must be used.

The powder purity and nature and form of any impurities present greatly affect the final properties of the forgings, usually by reducing the ductility and impact strength of the component. Unreduced oxide is the impurity that causes most problems in that it reduces the extent of interparticle bonding which results in properties which are inferior to those of the compacts made from the pure powder. This suggests that a reducing or protective atmosphere should be used during the sintering stage.

Friction between particles also affects the extent of densification obtainable by a mass of loose powder. This again is reflected from the powder particle characteristics of size, shape and purity. Efficient mixing cannot be obtained unless the friction conditions and particle characteristics are favourable, therefore they cannot be ignored.

Many currently produced components involve the use of alloys. These alloys can be produced from powder forgings by three basic methods, all three of which have their advantages and disadvantages. Elemental powders give compacts of higher green densities and are highly compressible. However, for complete homogeneity they need long, uneconomical diffusion times. This also applies when partially prealloyed powders are used. By using prealloyed powders the need for a homogenisation treatment can virtually be eliminated, the reason being that each powder particle is in fact a miniature ingot of the alloy in question. Because of the better homogenisation of this type of powder, enhanced mechanical properties can be obtained. The main disadvantage of this type of powder is the increased resistance to deformation of the individual particles which gives a reduction in compressibility and consequently a need for greater compaction pressures.

Basically the compaction method is governed by the powder characteristics and the size and shape of the final forging.

Preforms can be produced by various methods, the conventional method being in a rigid die. Choice of presses is dependent on the compact to be produced, they range from being plain to complex, multi-patterned ones. Isostatic pressing overcomes many of the problems of the previously considered method. It gives a high degree of uniformity, eliminates the need for admixed lubricants and involves the use of cheaper, and more flexible equipment. The main drawbacks of this method are the poor dimensional control achieved and the low production rates. Low density preforms can be made by slip casting. This method is normally employed when complicated shapes are to be produced from powders of low compressibility.

These preforms can then be forged by either of three main methods, namely; hydraulic pressing, mechanical pressing or by the use of a high energy rate press. Of the three it is generally agreed that the mechanical method is most favourable. The reason being that hydraulic compaction is not fast enough and that high energy rate compaction delivers the maximum load at the wrong moment. However, it must be noted that highly acceptable results have been obtained from all three compaction methods.

Heating processes during the forging operation can be varied from forging to forging. Some prefer induction heating due to the shorter times and less space consuming equipment. However, the times may be too short to allow complete diffusion, giving poorer mechanical properties. Also, control of the atmosphere is more difficult, which is why many operators prefer the conventional methods. In some cases, a separate heating stage must be incorporated to cater for the removal of lubricants and binders as they are detrimental to the final properties due to the poorer bonding obtained. Binders were mainly used with sinterforgings however, and die wall lubrication overcomes the problems incurred with admixed lubricants.

The main heating operation is the sintering stage when the individual particles are transformed to a mass of particles without identity due to recrystallisation and grain growth across the grain boundaries. Homogenisation is usually completed during this stage. Unless the sintered preform is well bonded and has a ductile structure, then uniform properties will not be obtained in the forged material. Post forging heat treatments have been found to be beneficial and deleterious to the properties of the final forging and it has been noted that prealloyed powders respond far better to post forging heat treatments than blends of elemental powders.

The success of the hot powder forging operation can be seen therefore to hinge on three main factors. These being the design, density and weight control of the preform, the temperature and pressure of forging and the powder characteristics. Other variables such as the method of compaction and heat treatment given are controlled by the factors mentioned and also the properties desired of the final forging. Once the optimum combination for these variables has been found and the initial cost problem overcome, then the process can be regarded as a superior substitute for conventional forging methods.

CHAPTER 8: EXPERIMENTAL WORK

Section 1: Aims of the Research

At the outset of this investigation, very little work had been published regarding the effect of certain variables on the structure and properties of hot forged powder alloys.

It has already been shown that there are numerous variables involved in the hot forging process; forging temperature, initial particle size, and initial particle size ratio being amongst the more important ones.

The aims of the present research therefore was to investigate the effects of these variables on the properties of hot forged alloy powder preforms and to study the processes occurring during the forging operation.

A comparison would also be made between the properties of the alloys made from a mixture of elemental powders and those mode from prealloyed powders and to determine whether similar results would be obtained by altering the variables examined.

It was anticipated that this study would yield an understanding of the significance of the variables in question to the final structure and properties of the forgings.

Section 2: Preliminary Experiments

The selection of powders to be employed in the research was based upon availability, cost, forging temperature range, oxidation characteristics and the compactability of the materials chosen.

Considering the above factors the only two materials applicable were copper and iron, both of which however have their respective drawbacks.

Both were readily available in high purity form, (copper being the most expensive) and common everyday alloys can be made from both. Copper is more susceptible to oxidation than iron but it can be forged over a much lower temperature range. Also, due to its lower strength, copper is much easier to compact than iron. Another advantage of the use of copper over iron as a base material is that there is no phase change between the forging temperature and room temperature.

Nickel was chosen as the alloying element because it also was readily available, had similar properties to copper and more important, formed a single phase alloy with copper. As Nickel has a somewhat higher melting point than copper, the ten percent Nickel alloy was chosen for the investigation in order to keep the forging temperature within a practical range.

The oxidation problem was overcome by employing a reducing atmosphere during the sintering and preheating stages.

A survey of the available literature indicated that irregular shaped powders compressed far easier than spherical ones, their flow properties also being far superior. The high purity irregular shaped copper and nickel powders were obtained from Berk Chemicals Limited and the Monde Nickel Company respectively.

The next problem was the selection of powder particle size and powder particle ratios. This was limited because the maximum particle size of nickel available was 90 microns and the smallest sieve size available was 38 microns. Subsequently, the copper and nickel powder obtained were sieved to the five possible size fractions within the range 38 - 90 microns and tests were performed to establish the most convenient usable ratios. These tests revealed that the maximum passible ratio for optimum mixing was 2 : 1. If ratios greater than this were used then the large particles merely acted as a sieve for the smaller ones which then segregated to the bottom of the mixture.

Therefore, considering the sizes of nickel available, ratios of 1.5 : 1 and 0.75 : 1 were chosen as the two extremes. Keeping these figures in mind, the powder particle size extremes selected to give these ratios were 38 - 45 microns : 45 - 53 microns for the small particle size, and 63 - 75 microns : 75 - 90 microns for the large particle size. These ratios were inverted to give the 'high' value for both large and small powder particle size.

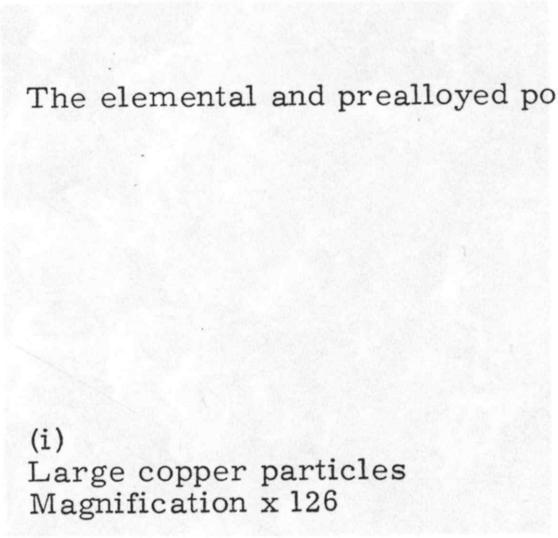
Similarly, the prealloyed powder was also separated into high and low values of particle size. However, it was not possible to study the effect of ratio for the prealloyed powder as it would have been completely impractical to form separate melts and ingots for each size ratio.

A sample of each powder size and ratio was then mounted and coated with gold for observation under the scanning electron microscope (Fig. 14) The difference in shape between the prealloyed and elemental powders is instantly obvious and results from the different manufacturing procedure.

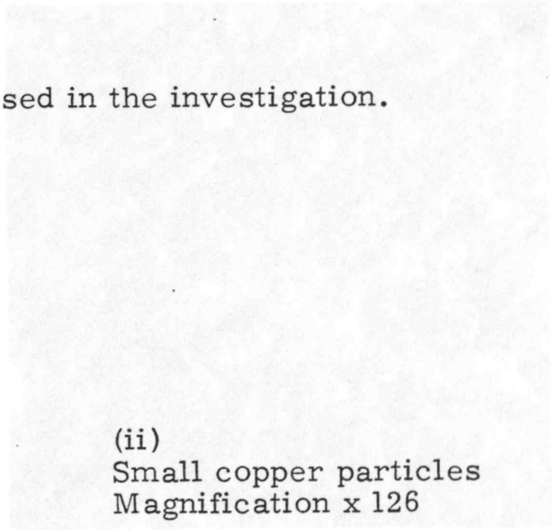
Green Compaction

The green compaction or preform dies are shown in Fig. 15 these produced a compact with base dimensions of 6.4 x 0.7 cms, the height being dependent on the mass of the powder and the pressure of compaction. A set weight of 10 gms was used for the elemental and prealloyed preforms for both large and small particle sizes. Preliminary trials revealed that slightly higher pressures were needed to achieve a

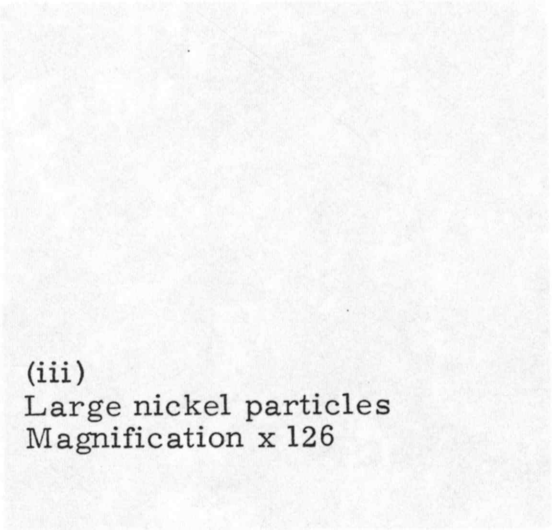
The elemental and prealloyed powders used in the investigation.



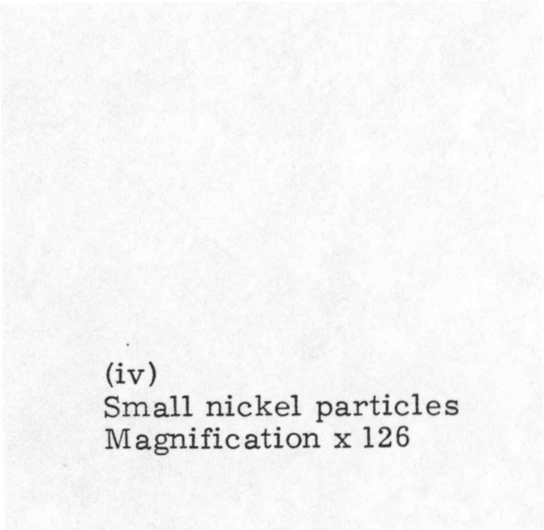
(i)
Large copper particles
Magnification x 126



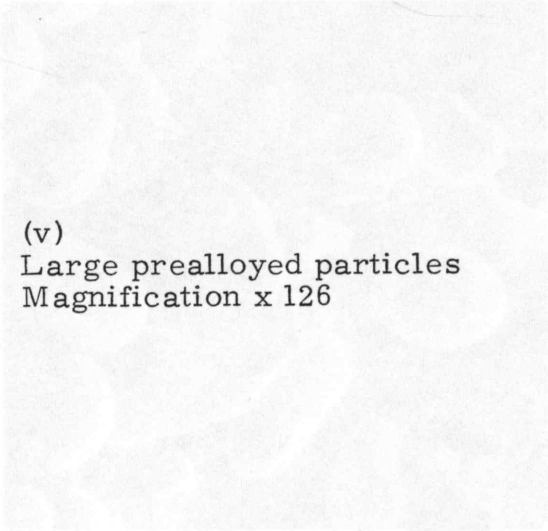
(ii)
Small copper particles
Magnification x 126



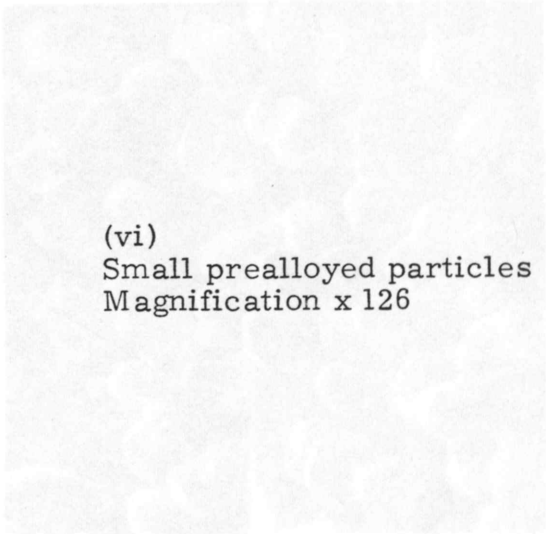
(iii)
Large nickel particles
Magnification x 126



(iv)
Small nickel particles
Magnification x 126

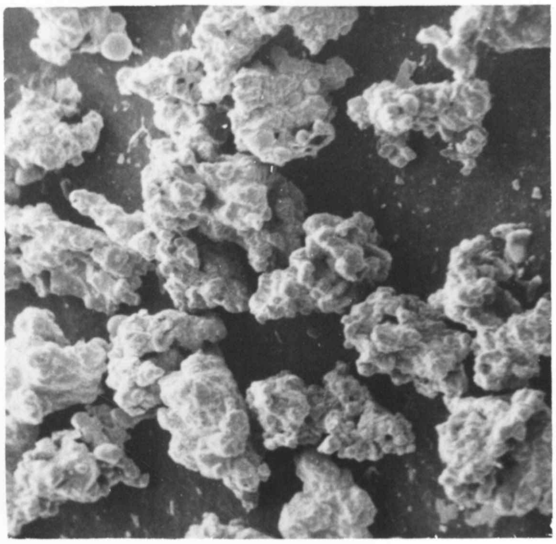


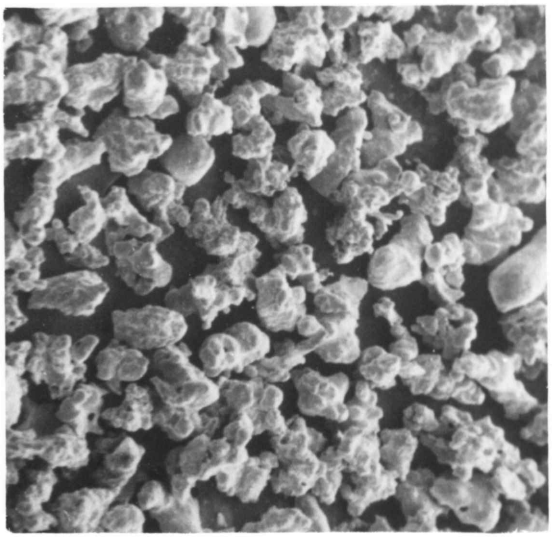
(v)
Large prealloyed particles
Magnification x 126

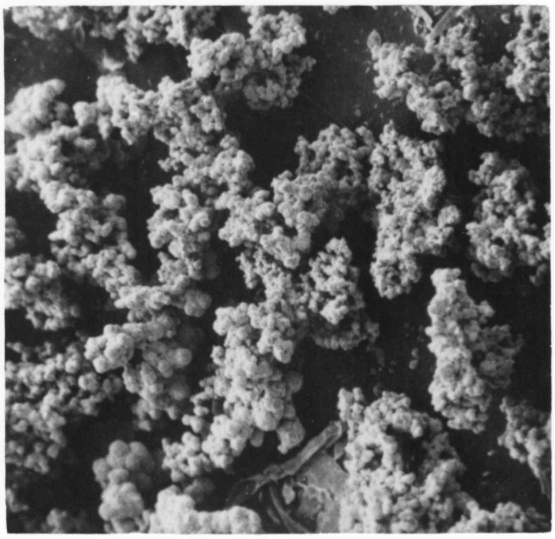


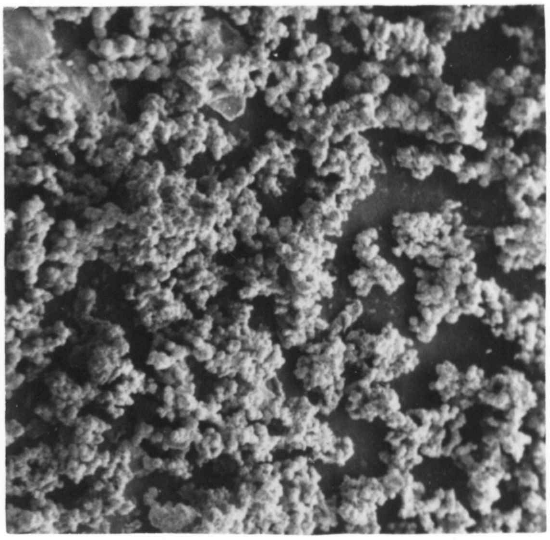
(vi)
Small prealloyed particles
Magnification x 126

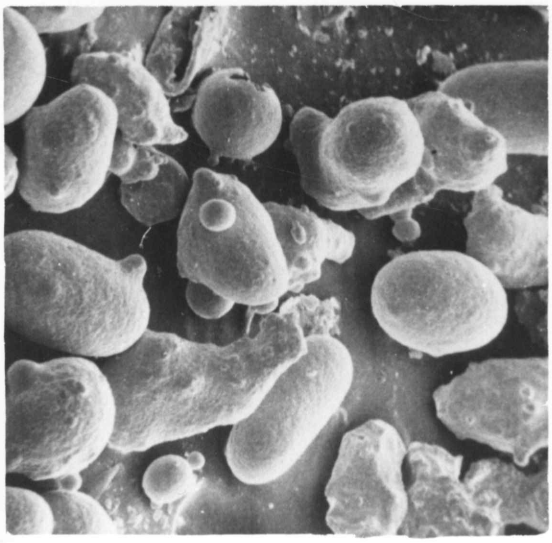
Fig. 14











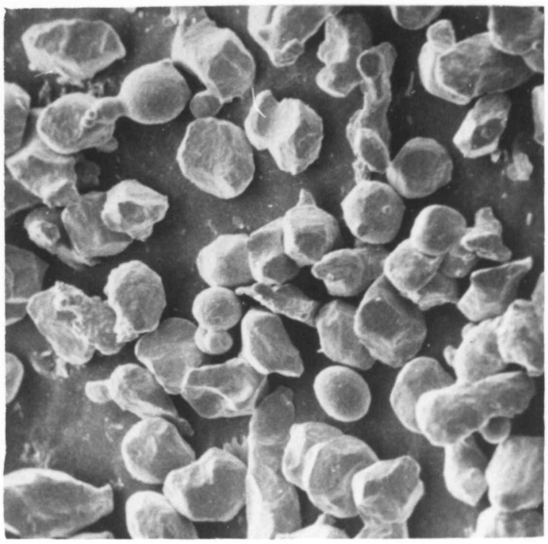
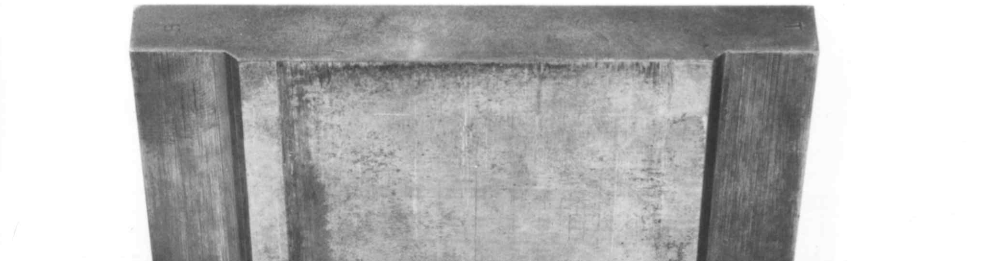


Fig. 15

The Green Compaction Dies



given density for the finer particle sizes. This was especially noticeable with the prealloyed powders and was probably due to the roundness of the particles.

Previous research work has shown that the rate of loading and dwell time has very little effect on the compaction for the range used. Therefore, considering the above factors a load of 20,454 Kg was used for the coarse powder and 21,818 Kg was used for the fine powder giving a preform density of 83.4% ± 0.8%. The rate of loading used was 0.212 mm/sec with a dwell time of 20 seconds.

The dies were thoroughly cleaned with a fine painter's brush after each compaction to eliminate any friction effects of loose powder particles.

Sintering Treatment

Fig. 16 shows the equilibrium diagram for the Cu/Ni system (99), from this diagram it can be seen that the melting point of the 90 - 10 alloy is 1400 K approximately. On this basis and on some previous work by Holly (6) a temperature of 1353 K was selected for the sintering treatment. This operation was needed to facilitate handling of the green compacts which were extremely brittle following the cold compaction. It was also needed to provide some degree of homogenisation prior to forging and to reduce any oxide formed on the particle surface during storage. Sintering tests proved that the minimum, most convenient time needed to satisfy these requirements was 24 hours. Following this period, the compact could be handled easily, a certain amount of homogenisation had been achieved (Fig. 18) and sufficient time had been given not only to reduce the surface oxidation but also to penetrate the interparticle porosity and provide extra protection in these areas within the compact.

The reducing gas used for the treatment was a mixture of 96% Nitrogen and 4% Hydrogen. The apparatus can be seen in Fig. 17.

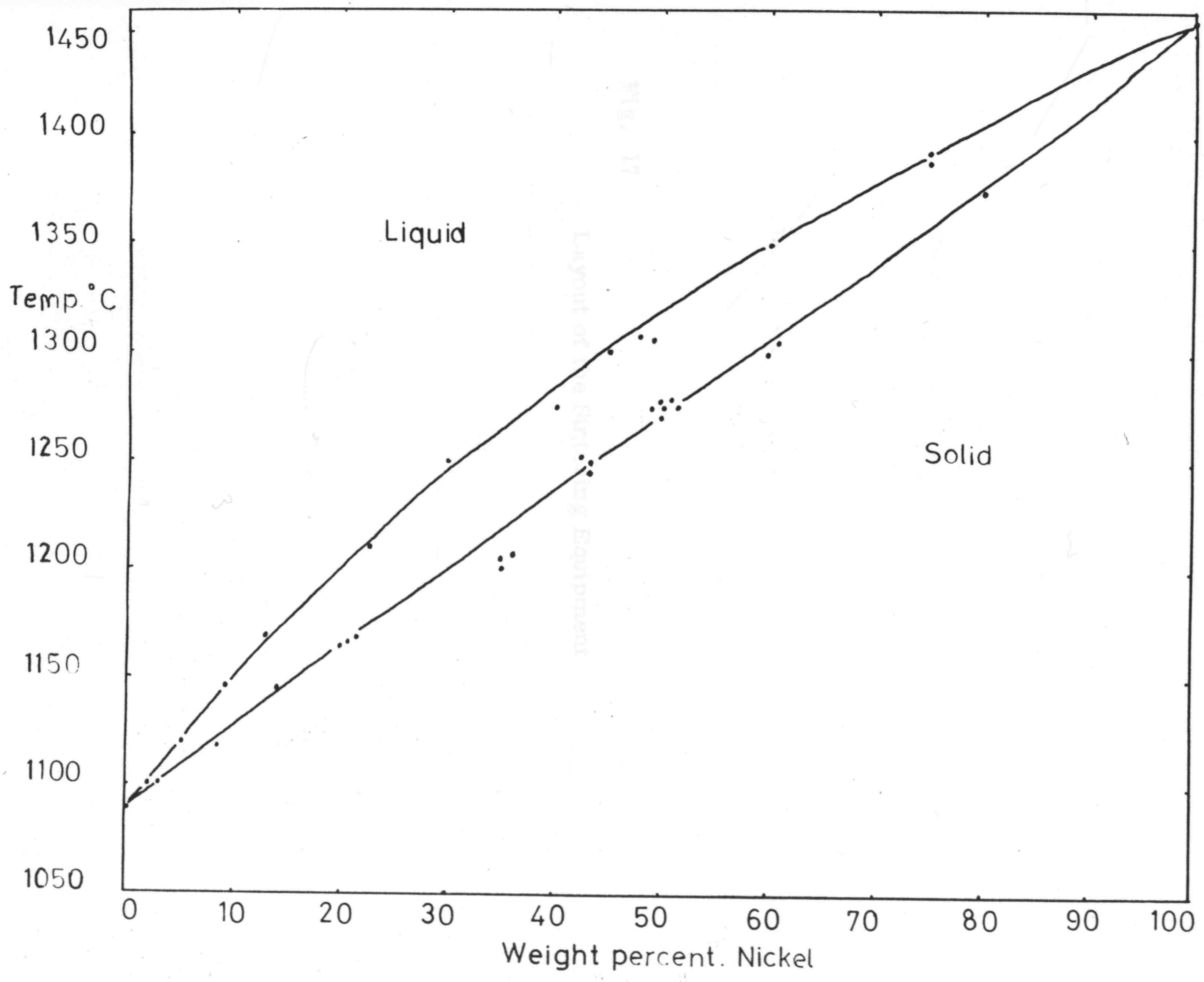
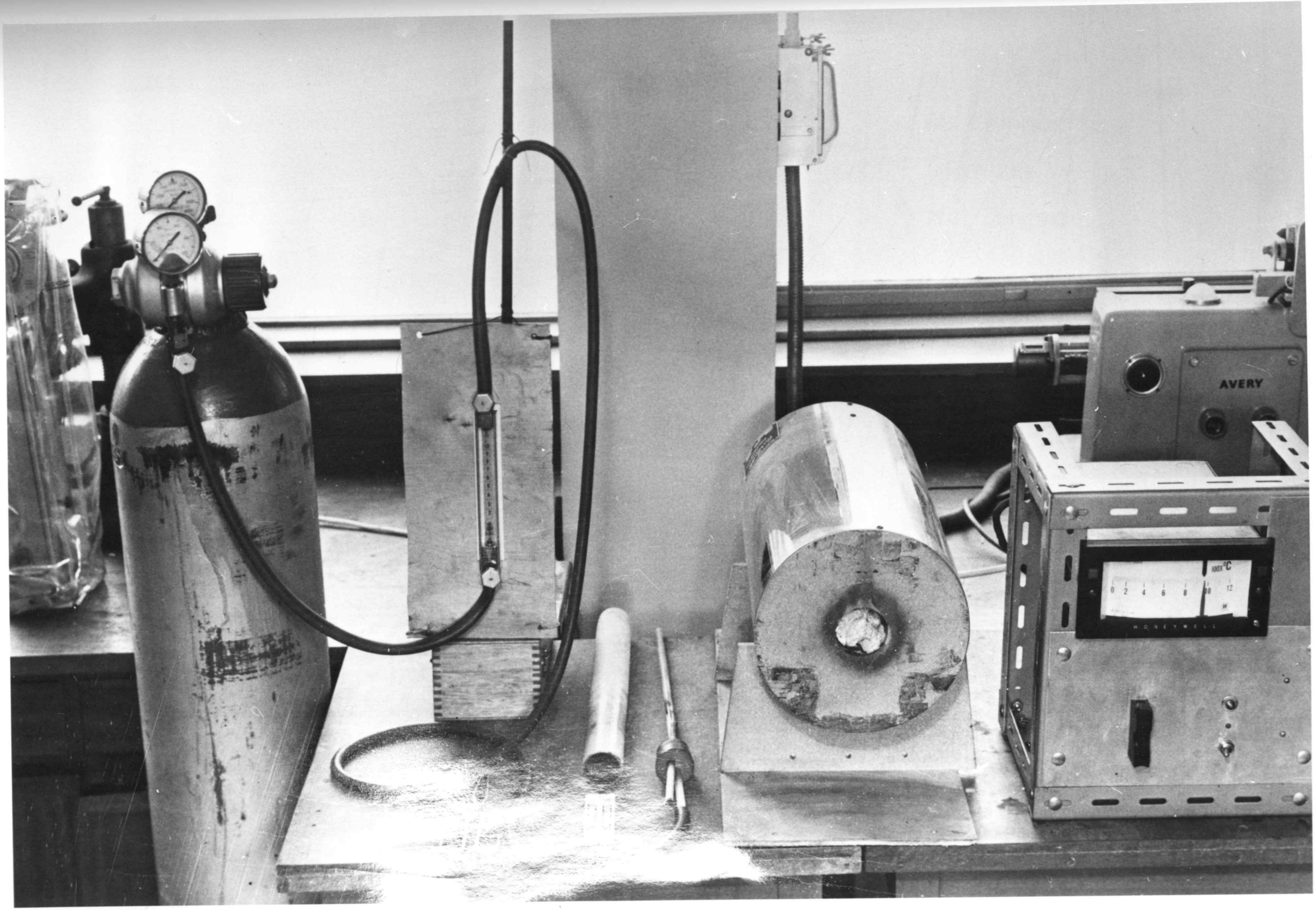


Fig. 16 The Copper - Nickel Equilibrium Phase Diagram

Fig. 17

Layout of the Sintering Equipment


AVERY
100X°C
0 2 4 6 8 10 12
HONEYWELL

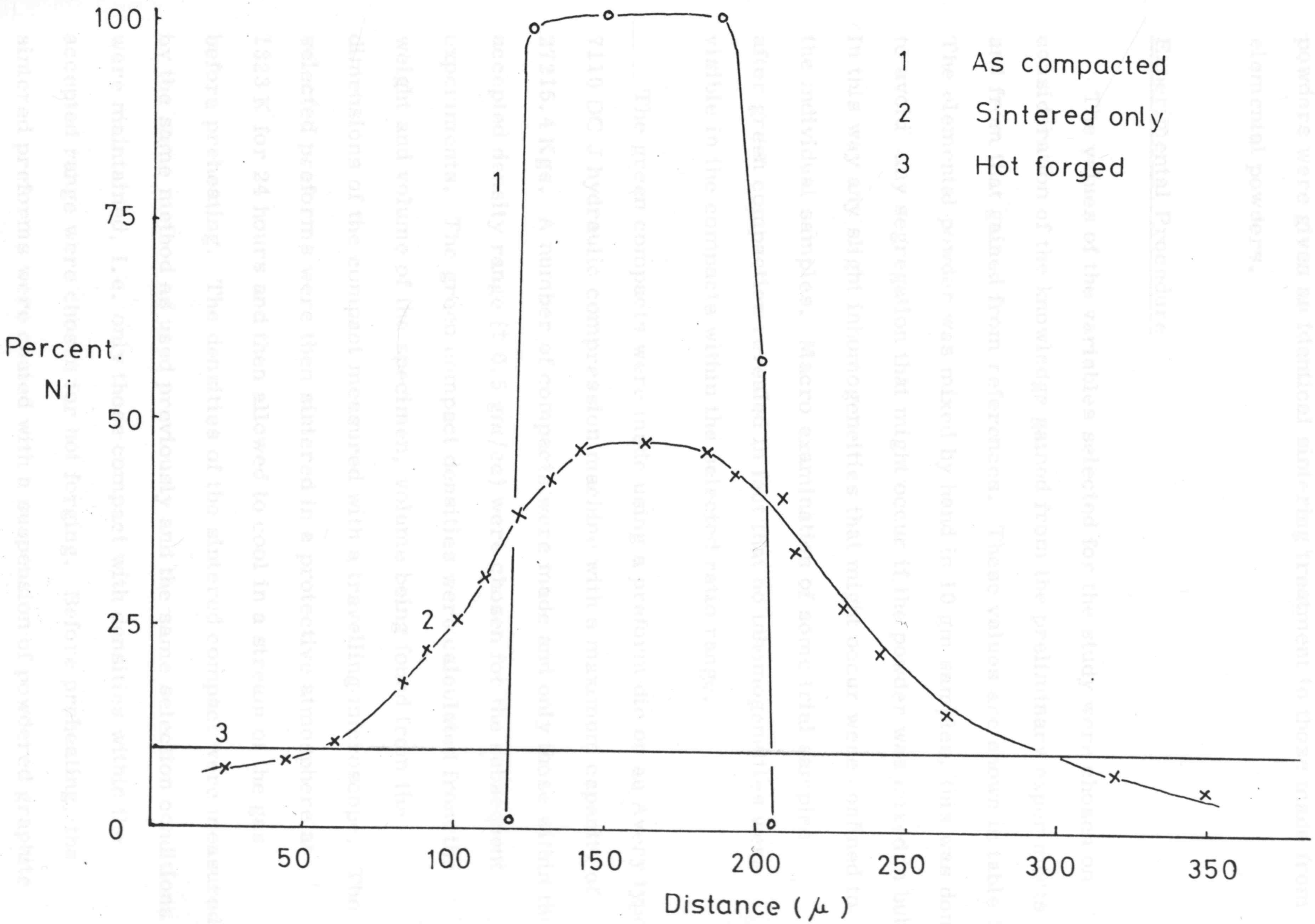


Fig. 18 Variation in Nickel concentration with distance across selected grains

To preserve uniformity the compacts prepared from prealloyed powders were given an identical sintering treatment to those made from elemental powders.

Experimental Procedure

The values of the variables selected for the study were chosen on consideration of the knowledge gained from the preliminary experiments and from that gained from references. These values are shown in table 1. The elemental powder was mixed by hand in 10 gm samples, this was done to avoid any segregation that might occur if the powder was mixed in bulk. In this way any slight inhomogeneities that might occur were confined to the individual samples. Macro examination of some trial samples after green compaction revealed in fact that no inhomogeneities were visible in the compacts within the selected ratio range.

The green compacts were made using a preform die on an Avery type 7110 DC J hydraulic compression machine with a maximum capacity of 27215.4 Kgs. A number of compacts were made and only those within the accepted density range (± 0.5 gm/cc) were chosen for the subsequent experiments. The green compact densities were calculated from the weight and volume of the specimen, volume being found from the dimensions of the compact measured with a travelling microscope. The selected preforms were then sintered in a protective atmosphere at 1323 K for 24 hours and then allowed to cool in a stream of the gas before preheating. The densities of the sintered compact were measured by the same method as used previously and the same selection conditions were maintained, i.e. only those compact with densities within the accepted range were chosen for hot forging. Before preheating, the sintered preforms were coated with a suspension of powdered graphite in ethanol.

and allowed to dry. Not only did this act as a lubricant but also protected the compact from oxidation during transfer from the furnace to the compacting die. The press for hot compaction was the same as that used for green compaction.

Tests showed that the length of time required by the preform and dies to attain forging temperature was 90 seconds, and 90 minutes respectively. However, the dies only needed approximately 20 minutes between forgings. The preform was preheated in the same apparatus as that used for sintering and the dies were preheated in a wide mouth muffle furnace placed next to the forging press. The preheating and forging layout can be seen in Fig. 19 It was desired that as little heat as possible be lost from the dies during the forging cycle, therefore they were designed in such a way that the forging could be quickly and easily removed after the operation (Fig. 20). The final forging was the same length as the original preform but was approximately twice as wide.

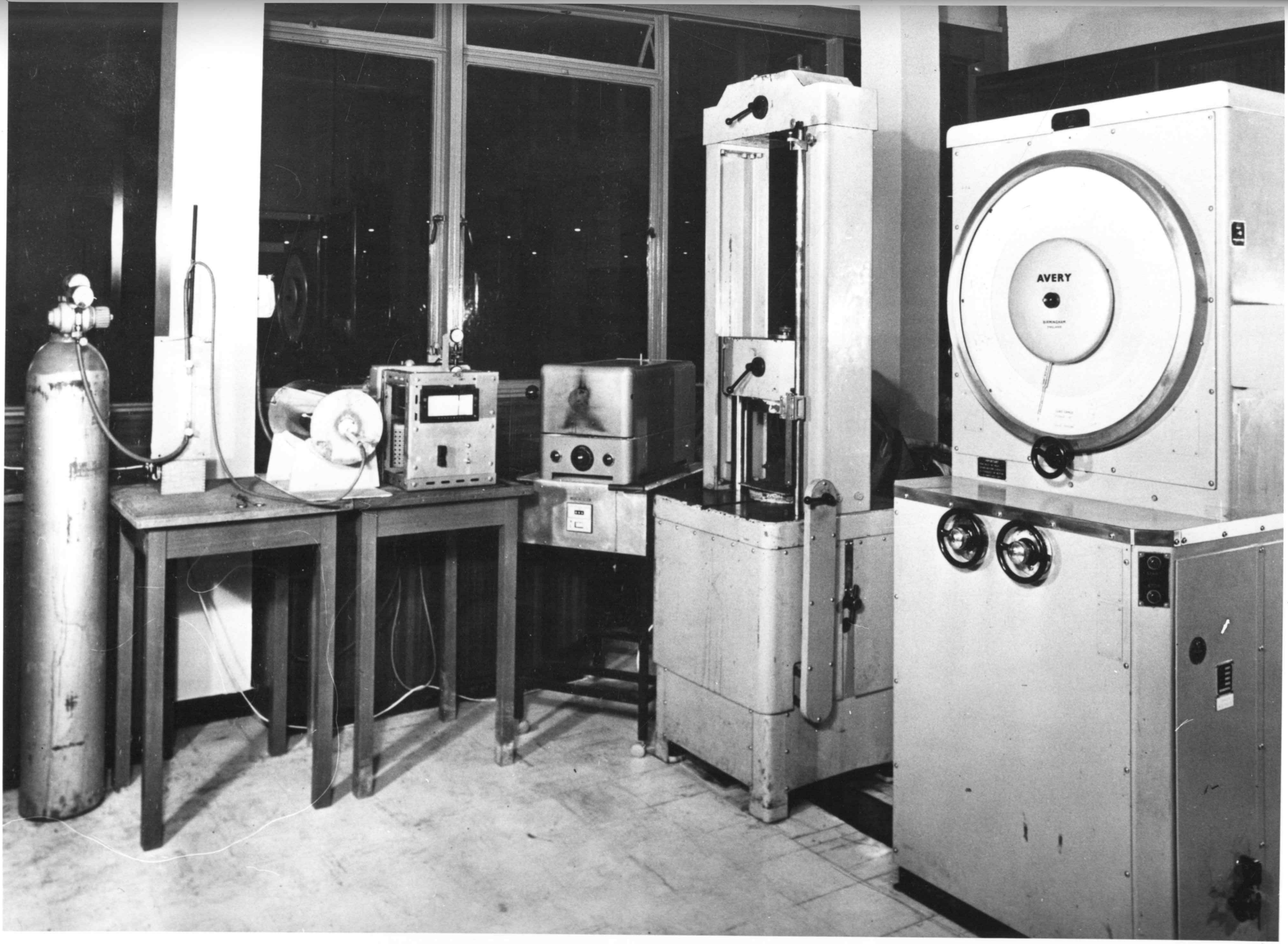
A dummy run showed that from the time of removal from the preheating furnace to the end of the forging stroke the dies lost between 15^{o} - 20^{o}, the time taken varied between 90 - 110 seconds. Therefore to compensate for this loss the dies were preheated to about 25^{o} above the forging temperature; a base plate was also preheated to the same temperature to avoid chilling of the bottom surface of the die when it was placed on the Avery compacting machine.

A 13,608 Kg load was used for each forging and the end product was air cooled or chilled on the steel surface of the forging press.

The time of transfer from the preheating furnace to the dies was 15 - 20 seconds and as stated previously the total time taken for the cycle

Fig. 19

Layout of the Forging and Pre-Heating Equipment


AVERY
BIRMINGHAM
ENGLAND

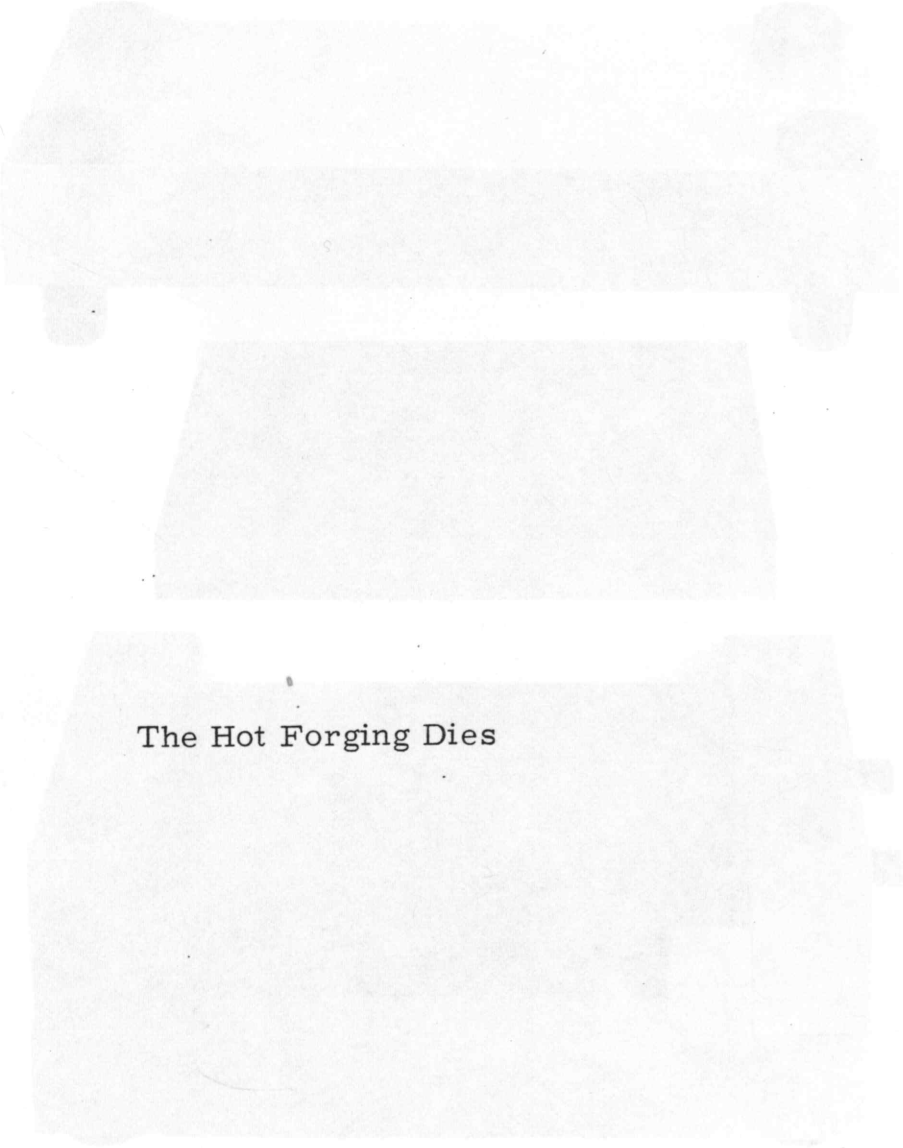
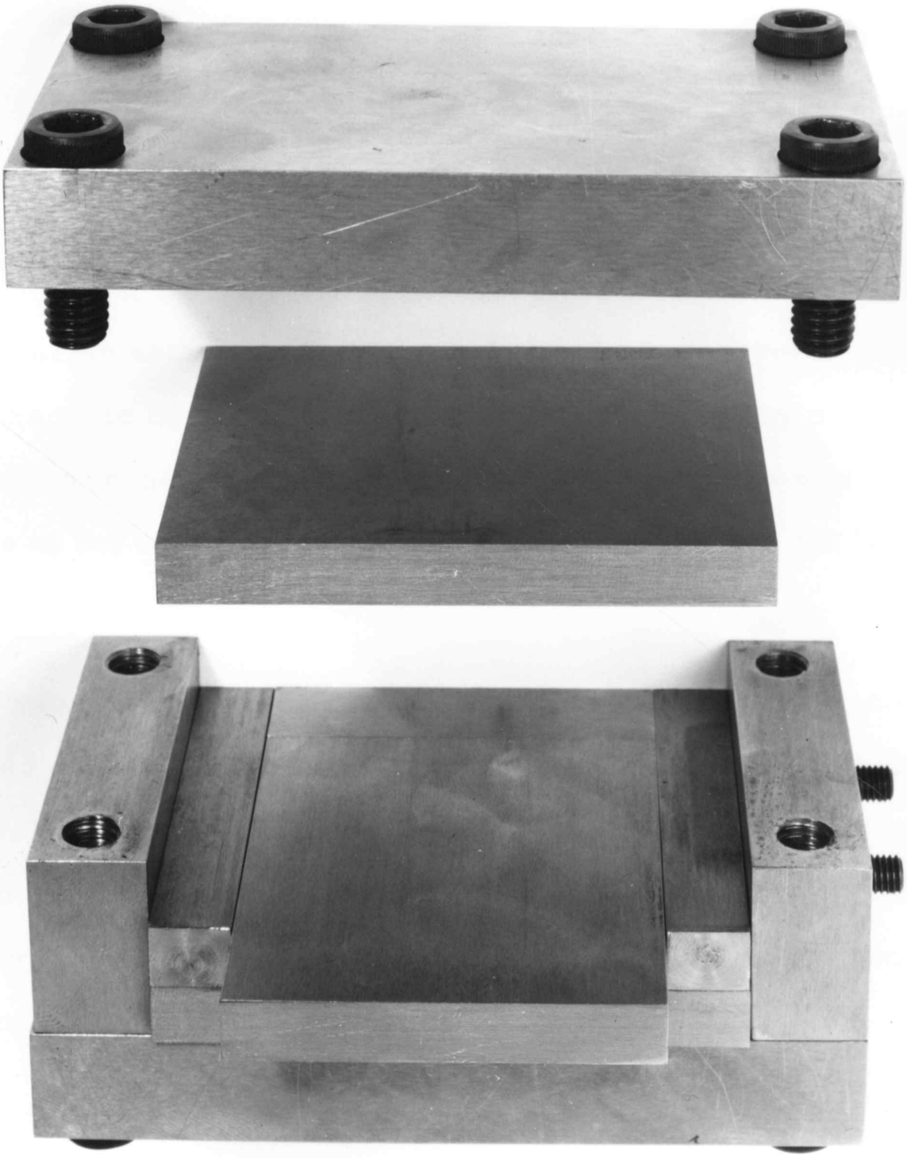


Fig. 20

The Hot Forging Dies



was about 2 minutes. To avoid excessive die wear, the lower temperature forgings were completed first and the dies were 'serviced' after every six forgings to ensure optimum performance.

Section 3: Manufacture of the Prealloyed Powder

The prealloyed powder used in the experiments described was made by the following method.

Six separate samples of copper and nickel powder were carefully mixed in the ratio of 10 : 1, and cast into 500 gm ingots by melting in a high frequency induction furnace. The size of the ingots was restricted by the size of the crucible that would fit into the coil with the largest diameter.

Care was needed to ensure that the coil did not come into contact with the crucible at any point. Stirring of the melt could only be carried out with the H.T. switched off. Oxidation of the melt was reduced by covering the surface with a suitable flux. Once the crucibles had cooled sufficiently, the ingots were removed and cleaned in preparation for the atomisation melt.

The atomising equipment can be seen in Fig. 21

The atomiser consists of a tundish (1) sealed down onto the jet block (2) which is again sealed on to the atomising vessel (3). A stream of molten metal of controlled size (regulated by nozzle apperture) falls from the tundish through the water jets which atomise and freeze the metal. The resulting slurry is allowed to settle in the collecting pot (4). Having decanted away the water the wet powder can be removed and dried.

Fig. 21

The Atomosing Equipment,

Showing 1. Tundish

2. Jet Block

3. Atomising Vessel

4. Collecting Pot

The nozzle size is restricted in that it must not be so large that the metal flowing through it vapourises the water. Neither must it be so small that it restricts the flow of molten metal. If larger melt sizes are being processed, then the minimum nozzle size becomes that which allows the required quantity of metal to flow before the water level in the atomiser rises too far.

Theoretical calculations of nozzle sizes are not usually successful because the very small nozzles have low and varying discharge coefficients. In addition, as the fluidity of molten metals varies so much, it has been proved necessary in some cases to double the nozzle diameter when pouring alloys of similar composition.

For the alloy in question, previous tests had shown that the optimum nozzle size was 4 mm.

The mean particle size obtained is roughly proportional to the atomising pressure. Tests had shown that the mean particle size obtained for pure copper at 11.37 MN in^2 was 63 microns, which is that required. However, small alloying additions of 1% can change the particle size obtained by as much as 50%, it was therefore decided to operate at 12.41 MN in^2 which gave a mean particle size of 70 microns. Nevertheless, enough of the small particle size was obtained to complete the necessary tests.

The metal should be poured into the tundish with a superheat of at least 50° K for metals melting from 873 K to 1473 K and at least 100 K for metals melting above 1573 K. Higher superheats give finer and rounder powder. Ideally the metal should be thoroughly molten and fluxed or deoxidised for good fluidity. Care must be taken to remove all slag, dross or flux before pouring into the tundish. This can either contaminate the powder or block the tundish nozzle or both.

Three kilogrammes of alloy was used for each run as it was found to be the most convenient for melting and handling.

Before commencing the operation the vessel is purged with Nitrogen. Purging time depends on the required oxygen content. Twenty to thirty minutes at 10 litres a minute is adequate for general use. For the lowest possible oxygen content, a displacement purge should be performed. This consists of filling the atomiser up to the vent with water before starting the purge. After sealing, the remaining space is purged for ten minutes and then the water is run out through the drain line, taking care that gas is still bubbling through the vent. The tundish is preheated for about 15 minutes before pouring the molten metal and operating the water jets. Only when the tundish is empty or when the metal has stopped flowing should the water pump be stopped. The powder is allowed to settle in the pot for a couple of hours before it is removed, washed in ethanol and then dried. To reduce the risk of caking, the powder should be constantly turned and the drying temperature kept at about 333 K. Samples of each size fractions were then taken for quantitative analysis.

Analysis Procedure

Each separate batch of alloy powder made was tested by the following method.

0.5 gms of the clean alloy powder was accurately weighed and placed in a beaker sufficiently large to be used an an electrolytic vessel. A mixture of 10 ml water, 1 ml concentrated sulphuric acid and 2 ml concentrated nitric acid was used to dissolve the alloy, and before starting the electrolysis, the oxides of nitrogen were boiled away and the solution diluted to 100 ml. The Platinum electrodes must be thoroughly cleaned in nitric acid, washed with distilled water and dried by burning off acetone before the eletrolysis for highest accuracy.

The weight of this electrode was then accurately measured and noted.

A current of 0.15 amp was passed through the solution, which was continually stirred, for two hours. At the end of this time, the electrode was slightly lowered and if no more copper was deposited then the electrolysis was complete. The difference in weight of the electrode before and after the operation indicated how much copper had been deposited and hence the composition of the alloy in question.

Evaluation of the readings obtained showed that two of the three alloys made were acceptable within the range of experimental error for an alloy of 90 : 10 copper/nickel. These two alloys were then mixed and sieved to the required size fractions in preparation for the hot forging experiments.

Section 4: Mechanical Testing and Examination of the Hot Forged Samples

Every forging was duplicated for each temperature, powder particle ratio and powder particle size used. Half of the samples were used for metallographic, density and hardness measurements and the other half for tensile testing. The metallographic samples were mounted by the usual methods and alumina (0.5 microns) was used for the final polish. Hardness measurements were made using a standard Vickers Hardness Testing Machine and the density of the compacts was measured by immersion in air free distilled water and corrected for temperature fluctuations. Pore size and distribution measurements were made on the specimens in the unetched conditions using a Quantimet 720 image analyser. The structure and grain size of the samples was examined after etching the samples in a solution of 4 gms Potassium Dichromate, 3 gms Sodium Chloride, 16 ml Conc. Sulphuric acid and 200 ml distilled water.

The tensile specimens, which were machined from the other forgings, were designed on the basis of G.L. = $5.65 \sqrt{A}$ with a set gauge length of 1.26 cms.

Hardness measurements and metallographic observations were also made on the shoulders of the tensile specimens to serve as a check for the observations made on the other specimens.

Tensile testing was carried out on an Instron machine at the strain rate of 1.67×10^{-3} sec^{-1} mm/sec and a chart speed of 5 ins per minute. Values of U.T.S., elongation and reduction in the area to fracture were measured from these tests.

Section 5: Experimental Procedure for Concentration Gradient Measurement

Analyses were carried out on a Cambridge Stereoscan Microscope using an energy dispersive analyser. The analyser spot size used was less than one micron. All the specimens were prepared by careful metallographic polishing and by using a standard electropole electropolishing agent. Analyses were carried out at a beam incidence angle of 45^{o} and take off angle of 45^{o} also.

Alloy specimens and standards of pure Nickel and pure Copper were analysed and in each case the ratios of the X-ray intensities of alloys and standards were obtained. Measurements were made on Copper K_{α} and Nickel K_{α} radiation. The ratios obtained were corrected for atomic number, absorption and fluorescence by a standard Z.A.F. technique and the results were presented as percentage Nickel observed for distance travelled across the grain.

X-ray Analyses of the Compacts to determine the extent of Homogenisation occurring during the Hot Forging Operation for both Elemental and Prealloyed Powder Compacts

X-ray analysis was performed on all the different combinations of forgings made to examine the extent of homogenisation occurring during the hot forging operation for each combination of factors. The process of homogenisation can be traced by the change in X-ray diffraction peak profiles. As the homogenisation increases, the peaks of the relative constituents approach each other, merge and finally produce a single peak as complete homogeneity is achieved. Therefore the degree of penetration of the curves gives an indication of the extent of homogenisation of the alloy.

This broadening of X-ray traces consists of three factors:

1. Instrumental inaccuracy causing broadening.
2. Cell size due to work hardening.
3. Inhomogeneity of the alloy.

To measure the broadening due to instrumental error a trace is made of a completely homogenised, fully annealed alloy so that the effects of inhomogeneity and cell size due to work hardening can be eliminated. This means that the broadening of this profile is due entirely to the instrumental error. The line braodening due to lack of homogeneity and work hardening can therefore be determined by separating the broadening due to instrumental error from subsequent traces made from the hot forged samples. As the broadening due to inhomogeneity in the alloy is much greater than that due to work hardening no further separation of the peaks was considered necessary.

A numerical method has been derived using Fourier analysis (109) for the correction of widths and shapes of lines on X-ray powder photographs, i.e., a method for finding the corrected distribution across an X-ray diffraction line. A computer programme based on this numerical method was devised and utilised for the current series of experiments. Details of this programme and examples of the traces obtained are included in the appendix

The experimental procedure followed for performing these analyses was as below:

The diffraction measurements were carried out using a Siemens diffractometer. Copper K_{α} radiation was used with the β component and background radiation reduced by the use of a carbon monochromator. Analysis was performed on the $\{311\}$ peaks and each sample was scanned over the range of 20 from 89° to 95°. The specimens were prepared by careful grinding of the surface under water until approximately 0.5 mm of the surface had been removed. Chart recorder plots of the diffraction peaks were analysed by the Fourier unfolding technique referred to earlier, also (107), (108).

CHAPTER 9: RESULTS AND DISCUSSION OF RESULTS

The results obtained from the research work carried out are presented in four main sections. The first deals with the statistical analysis of the results obtained from mechanical testing of the forgings made from elemental and prealloyed powder compacts. Then follows a section on the X-ray analysis work performed to examine the homogeneity of the alloys formed and to relate this where possible to the mechanical properties observed. Similar comparisons and contrasting observations are made in the third section in which the metallographic findings are discussed. Finally a summary of the complete results is given in the fourth section.

Section 1: Statistical Analysis of the Results of Mechanical Testing

Preface

When dealing with scientific investigations problems frequently arise if the effects of a number of different factors on some property or process require to be evaluated. Such problems can usually be most economically investigated by arranging the experiments according to an ordered plan in which all the factors are varied in a regular sequence. As long as this plan is correctly chosen, it is possible to determine not only the effects of each individual factor, but also the way in which each effect depends on the other factors (i.e. the interactions). In this way it is possible to obtain a more complete picture of what is happening than would be obtained by varying each of the factors one at a time while keeping the other constant. The advantage of using factorial design in scientific/industrial experiments can be summed up in the following way:

(a) When there are no interactions the factorial design gives the maximum efficiency in the estimation of the effects.

(b) When interactions exist, their nature being unknown, a factorial design is necessary to avoid misleading conclusions.

(c) In the factorial design, the effect of a factor is estimated at several levels of the other factor, and the conclusions hold over a wide range of conditions.

These conclusions have been arrived at for two factors only, they hold with even greater emphasis when more than two factors are involved.

In factorial experiments in which there is no replication and for which no prior estimate of the error is available, it is customary to employ an estimate of the error variance based on the higher order interactions. This practice may be justified on the grounds that often higher order effects of this kind are non-existent or at least negligible. On this assumption the sums of squares corresponding to the higher order interactions may be added together and divided by their total degrees of freedom to give an estimate of error variance based on several degrees of freedom.

The method of analysis used in the current series of experiments is the systematic tabular method developed by Yates (105). Further analysis and interpretation was as described by O. L. Davies - ref 'The Design and Analysis of Industrial Experiments' (106).

These methods were used to interpret the results of mechanical testing obtained for both elemental and prealloyed powder forgings. Only the most significant results were investigated, i.e. those factors and interactions of factors whose effect on the result of property being examined had a significance of greater than 90%. As explained earlier, the three factor interactions were regarded as being suitable for use as an estimate of the error involved in the experimentation. The results obtained will be discussed for each property in turn for both elemental and prealloyed powders and a comparison made of the findings for both types of powder and related where possible to the observations made from the metallographic examination of the forged specimens.

Details of the relevant proability points of the variance ratio (F distribution) for each significance level are given in table 11 and the values of Error Mean Square for each property examined are given in table 12.

The full statistical analyses performed for both elemental and prealloyed powder forgings are detailed in Appendix 1 and Appendix 2 respectively. The relevant details have been extracted from the analyses and are detailed in the tables referred to in the text.

Results obtained for the testing of compacts made from elemental powders

1. Ultimate Tensile Strength

It is clearly evident from the results shown in Table 13 that the largest and most significant effect on the U.T.S. of the forged specimens is that of forging temperature. An increase in the forging temperature results in a dramatic decrease in the U.T.S. of the forgings with the advent of greater ductility and recrystallisation causing a decrease in internal energy at the higher temperatures. Other less significant results include the quadratic effect of particle size ratio and the interaction of temperature with both particle size ratio and particle size, all of which occur at the 10% level. Considering first the interaction between temperature and particle size ratio we can see from table 13.1 that there is no significant effect on the U.T.S. of the forging when the particle size ratio is varies at any of the forging temperatures used. Whereas when the effect of the interaction between particle size and temperature is examined (table 13.2) it appears that changing the particle size from Co to C_1(from low to high) at the lower levels of temperature produces a drop in the measured U.T.S. this reduction decreases as the forging temperature increases until there is effectively no change in the U.T.S. when the particle size is changed at the highest temperature. A possible explanation for this is that at the lower temperatures the effect of forging temperature does not dominate as much as it does at the highest temperature, thus revealing the less dominant effect of particle size. The drop in U.T.S. observed by increasing the particle size is probably brought about by the increased area of contact between the larger particles facilitating more efficient diffusion, the ensuing increase in homogeneity giving a greater extent of recrystallisation with a subsequent decrease in the U.T.S. Another interesting effect is the rapid drop in U.T.S. between the two higher levels of temperature for both the particle size/temperature interaction and the particle size ratio/temperature interaction when compared with the change in U.T.S. between the two lower levels of temperature. It is possible that the proximity of the recrystallisation temperature for the alloy in this region contributes to this effect. In fact, in the temperature/particle size interaction there is no difference between the U.T.S. at b_o and b1 for the higher particle size, and for the same levels of temperature with the particle size ratio interaction an increase in U.T.S. is observed at a_2. Actually, it can be observed for both interactions that the difference in U.T.S. when the temperature changes from b_o to b_1 and from b_1 to b_2 decreases as the levels of the other factors increase.

2. Proof Stress

Once again it is apparent from table 14 that the most significant effect is that of temperature. The effect is not as great however as that noted for the U.T.S. Other significant effects include the interaction between the particle size ratio and the particle size, and that between the temperature of forging and the particle size at the 10% and 5% level respectively. Closer examination of the interaction between temperature of forging and the powder particle size (table 14.2) shows once again that the temperature plays a major role in deciding the properties of the allow produced. Increasing the temperature for both particle sizes gives a decrease in the proof stress. Further examination reveals another interesting effect in that at the lower level of temperature, changing from low particle size to the high particle produces a drop in proof stress. However, on increasing the temperature this effect is apparently reversed. It seems that there is a change from a negative effect at the lowest level of temperature to an increasing positive effect as the temperature is increased over the two levels of particle sizes i.e., C_0-C_1 at b is equal to -26.24, C_0-C_1 at $b_1 = 13.74$ and C_0-C_1 at $b_2 = 24.77$. No apparent trend could be detected in the interaction between powder particle size and power particle size ratio (table 14.1).

3. Pore Size

The results of the statistical analysis on pore size readings can be seen in table 15. It is immediately apparent that the most significant factor affecting this property is that of forging temperature. Both linear and quadratic components of the effect display significance levels of between 0.1% and 1%. The only interaction and in fact the only other factor which has a significant effect on the pore size results is that of the particle size/temperature interaction (table 15.1). From the analysis it is apparent that on the mean there is a tendency for the percentage of small pores to increase as the temperature of forging rises and decreases as the particle size increases. This again is as would be expected since a rise in temperature would enhance grain growth and accelerate vacancy migration thus eliminating the larger pores, whereas increasing the particle size would tend to cause large pores due to geometric reasons. It might be considered surprising to note that there is no significant effect of particle size ratio on the pore size. The probable reason for this is because of the small concentration of nickel particles in the alloy, their influence in filling or creating cavities is negligible. One results that is inconsistent with the above discussion is the increase in percentage of small pores at the highest particle size and temperature. It is suggested that this could be due to the fact that increasing particle size provides a greater surface area of contact between the particles thus enhancing diffusion. At the highest temperature it is possible that the diffusion mechanisms operate at a greater efficiency hence reducing the number of large pores faster than at the lower temperatures.

4. Recrystallisation

The results of statistical analysis of the recrystallisation observations detailed in table 16 show that all three factors contribute at different levels of significance. Once again it is evident that the most significant is that of temperature followed by the effect of particle size at the 1% level. Then follows the temperature/particle size linear interaction and the quadratic effect of temperature at the 2.5% level and finally the linear effect of particle size ratio at the 5% level of significance.

The highly significant effect of temperature on this property was certainly expected as it plays such an important role in the mechanisms of recrystallisation. However, the analysis also indicates the high significance of the effect of interaction between the temperature of forging and the initial particle size. This in fact, is as significant as the quadratic effect of temperature but not as significant as the particle size effect alone. Closer examination of the interaction between temperature and particle size effects (table 16.1) shows that for both small and large initial particle sizes, an increase in forging temperature yields an increase in the extent of recrystallisation. The rate of increase in the extent of recrystallisation can be seen to be much greater at the higher particle size than it is at the small particle size. At the lowest temperature the extent of recrystallisation for both large and small particle sizes is virtually the same, however, at the higher temperatures an increase in the extent of recrystallisation is noted between the small and large particle sizes. This effect is seen to increase with further rise in temperature, suggesting once again, the proximity of the recrystallisation temperature for the alloy. One explanation for the particle size effect could be that as the initial particle size is increased diffusion between the particles is enhanced due to the greater area of contact between the particles as previously explained. The diffusion mechanisms would be enhanced further as the temperature of forging is increased. This greater degree of diffusion will mean that the rate of homogenisation of the alloy will be accelerated, reducing the number of obstacles to recrystallisation resulting in a greater amount of recrystallisation at the end of the forging stroke.

5. Grain Size

All three factors are again seen to be involved with having a significant effect when the result of the grain size analysis is examined (table 17). However, the significance of these effects is much higher than those examined for the previous property. The effects of both temperature of forging and initial particle size are significant at the 0.1% level. The quadratic effect of particle size ratio has a reaction with both the linear component of the temperature effect and the linear component of the particle size effect, both of which are significant at the 1% level. Also significant at this level is the quadratic component of the temperature effect. It is interesting to note that these interactions of quadratic particle size ratio effects are much more significant than both the linear and quadratic components of the main effect of particle size ratio which have 5% and 2.5% levels of significance respectively. Other significant interactions include the one between the quadratic component of temperature and the linear components of particle size at the 2.5% level and those between linear temperature and linear particle size, and linear particle size ratio and quadratic temperature - both of which occur at the 5% level of significance.

Closer examination of the three interactions show that in the case of the particle size ratio and temperature interaction (table 17.1), the grain size decreases for all particle size ratios as the temperature of forging increases. An interesting feature of this interaction is the fact that for both low and high particle size ratios the change in grain size over the range of forging temperatures is much greater than that for the intermediate ratio; i.e., when the initial sizes of the copper and nickel particles are equal the change in grain size observed when the compacts are forged at 773K and 973K is much less than if either the initial copper or nickel particle size was greater than the other for the same forging temperatures. This could be due to the greater number of points in contact with each other resulting in fewer cavities and hence greater opportunity for grain growth and diffusion mechanisms to proceed. At the lower levels of temperature it is evident that not a great deal of change in grain size occurs, however, at the higher temperatures there is quite a drop in grain size indicating the commencement of recrystallisation. This drop is much greater when the initial copper and nickel particle sizes are unequal, possibly due to the reasons given above.

Considering the interaction between the initial particle size ratio and the initial particle size (table 17.2), one can see that for all particle size ratios the grain size is decreased as the initial particle size is increased. Again this is probably due, as suggested previously, to the greater area of contact between the large particles, giving fewer grain boundaries hence improved diffusion, faster homogenisation and greater extent of recrystallisation. It seems that this effect is greatest, i.e., a greater drop in grain size, when the particle sizes are equal (the intermediate ratio) which would seem to contradict the findings of the previously considered interaction. This discrepancy can be explained however, by the fact that the presence of temperature in the former interaction has the greatest effect and has a greater bearing on the diffusion processes than the two other factors.

Examination of the third interaction between the initial particle size and temperature of forging (table 17.3) reveals the now familiar trend of decreasing grain size for both original particle sizes as the temperature of forging is increased. Also evident is the decrease in grain size with increasing particle size at all temperatures and that this effect is greatest at the higher temperatures for reasons given previously. It is also noticeable that there is a much greater reduction in grain size between the two higher temperatures than between the lowest and intermediate temperatures indicating once again, that the recrystallisation temperature must be in this vicinity.

In the remaining four properties examined the most significant factor in the hot forging operation is again seen to be that of forging temperature. For the properties of reduction in area and elongation at fracture, the original particle size is seen to be significant at the 1% and 10% levels respectively along with the highly significant effect of forging temperature (tables 18, 19). It would appear that in all cases an increase in the original particle size leads to a decrease in the elongation and in the reduction in area hence a loss in ductility whereas increasing the temperature gives the anticipated increase in ductility. Examination of the analyses for the final two properties (tables 20, 21), reveals that for the density measurements the only significant factor is that of temperature at the 0.1% level. For the hardness results, the particle size and temperature interaction and the temperature effects were significant at the 10% level. No other factor of higher significance could be seen. Examination of the temperature/particle size interaction did not reveal any consistent trends.

Summary of observations made from Statistical Analysis

From the statistical analysis made on the results of mechanical testing the alloys made from elemental powder mixes, it is instantly apparent that the most significant factor in the hot forging operation is that of temperature of forging. The analysis also revealed the important role played, in a number of properties, by the effect of initial particle size and the interaction between initial particle size and temperature of forging. Increasing both the temperature and initial particle size tended to improve the property of the forging by enhancing the diffusion mechanisms hence accelerating the rate of homogenisation and increasing the extent of recrystallisaion. The effect of particle size ratio could only be seen to be significant in the grain size results where it seems that the extent of the effect varies on departure from equal grain sizes. This lack of significance of initial particle size ratio effects can probably be attributed to the fact that the effective distance between the nickel particles, those causing the inhomogeneity, will remain the same whatever the particle size ratio. This fact along with the low concentration of nickel would seem to diminish the significance of this factor on the hot forging operation.

Therefore, when specific properties are desired from hot forged compacts made from elemental powders, not only must the temperature of forging be considered but also the original particle size should be taken into account. Table 22 lists the important factors affecting each property examined with details of the relevant levels of significance.

Results obtained following the mechanical testing of compacts made from prealloyed powders.

Statistical analysis was also performed on the results obtained for each property when the compacts had been made from prealloyed powders. In this case, however, only two variables were utilised, those of temperature of forging and the original particle size. The estimate of error involved in the results was taken as being the same as that calculated for the results obtained from elemental powders because of the greater number of variables and hence more accurate estimation of error possible in this latter case. However, due to the lowest number of results and combinations of interactions, these analyses were not as valuable or as informative as those obtained from the elemental powder compacts. Nevertheless, it is evident from the results obtained that this series of experiments did serve to confirm statistically the observation made in the case of elemental powders that not only is the temperature of forging critical, but also the initial particle size of the powders used is an important consideration when specific properties are required from the final forging.

Considering initially the statistical analysis made on the results obtained for the recrystallisation measurements (table 23), it is apparent that the temperature of forging is the dominant factor with the linear component of temperature being significant at the highest level and the quadratic component at the 1% level. Also significant for this property is the effect of initial particle size (95% significant). As noted for the elemental powders, varying the original particle size causes a change in the extent of recrystallisation. In this case however, an increase in original particle size causes a decrease in the extent of recrystallisation. A possible explanation for this discrepancy can be given by the fact that in the case of the compacts prepared from elemental powder, homogenisation must take place before recrystallisation can satisfactorily occur. To assist in this homogenisation the most efficient diffusion processes must be operating and this occurs when the area of contact between the particles is as large as possible, i.e., when large initial particle sizes are used. With the smaller particles, the extra number of grain boundaries will act as obstacles to the homogenisation processes, whereas in the case of the prealloyed powder compacts, due to the much higher initial homogeneity, this need for homogenisation is not as great. The extra grain boundaries present due to the smaller particle size, then act as nucleii for recrystallisation as the homogeneity is sufficient for the recrystallisation processes to commence. The difference in extent of recrystallisation can be attributed to the fact that in the case of compacts made from elemental powder a fraction of the forging time is taken up in the homogenisation prior to recrystallisation whereas in the case of prealloyed compacts the greater homogeneity means that more time is available for recrystallisation processes to occur resulting in more extensive recrystallisation. This observation is discussed in greater detail in Section II of the results which deals with the x-ray analysis of the forged compacts.

Once again, however, a discrepancy is noted in that for large initial particle sizes and low forging temperatures, the extent of recrystallisation in compacts prepared from elemental powder is greater than those made from prealloyed powders. When small particle sizes are used, a greater extent of recrystallisation is observed for prealloyed compacts for all three temperatures. This is possibly due to the difference in initial homogeneity, in the case of the prealloyed powders, the extra grain boundaries will act as nucleii for recrystallisation. However, for elemental powders, because of the reduced homogeneity, these grain boundaries act as obstacles to diffusion, hence delaying homogenisation and hence resulting in less recrystallisation. When large initial particle sizes are used however, this observation is reversed at the lower temperatures, possibly due to the fact that improved diffusion occurs in the elemental powders because of the greater area of contact between the particles, thus facilitating similar amounts of recrystallisation at these lower temperatures where recrystallisation activity is minimal. It could also be argued that for such small extents of recrystallisation, the observed differences could be considered negligible. However, at the higher temperatures, where recrystallisation activity is at a maximum and where homogenisation effects would be emphasised, it is possibly this difference in homogeneity between the powders that results in the enhanced recrystallisation observed in the case of forgings made from prealloyed powders.

The ductility results of reduction in area (table 24) and elongation at fracture (table 25) also echo what was found previously in the case of elemental powder forgings that the temperature of forging is the controlling factor with the initial particle size also having a significant effect especially in the case of reduction in area where a 95% significance level is observed. It would appear that greater ductility is obtained as the initial particle size is increased and the temperature of forging increased. Examination of the ductility and grain size results for prealloyed powders (table 24, 25, 26) reveals two apparently conflicting tendancies. On the one hand increasing the grain size by increasing the initial particle size gives an increase in ductility, whereas on the other, decreasing the grain size by increasing the forging temperature also increases the resulting ductility. This can be explained with reference to some work performed by Jaoul (104) on the stress/strain curves of aluminium. It is established that

$$\sigma = A \varepsilon^{n}$$

where σ = stress

ε = strain

n = work hardening coefficient

A = constant

From this it is clear that the ultimate tensile strength is proportional to the work hardening coefficient. Jaoul observed that this work hardening coefficient was sensitive to the grain size of the material being tested. A large grain size gave 'n' values of around 0.7, whereas small grain sizes gave a value of 0.5 for the work hardening coefficient. These results indicate that a small grain size gives a material lower ductility than one with a larger grain size. This explains the increase in ductility noted as the grain size is increased by using larger initial particle sizes. The increase in ductility observed when the grain size is reduced by elevating the forging temperature is probably due to the fact that at these higher temperatures recrystallisation phenomena eliminate the effects of work hardening causing the noted increase in ductility. This observation is contradicted however, when the case is considered for the results obtained from elemental powders. Here it can be seen that increasing the original particle size results in a decrease in grain size due to more extensive recrystallisation and a subsequent drop in ductility. This would seem to suggest that other factors apart from the effects of work hardening, influence the complex relationship between ductility and grain size. Examination of tables 27, 13 show that for both prealloyed and elemental powders the initial particle size has a significant effect on the U.T.S. of the forging. Compacts made from powders with small initial particle size give forgings of higher U.T.S. than those made from large initial particles, at the lowest temperatures for both elemental and prealloyed powders. This difference however can be seen to diminish as the forging temperature is increased for compacts made from both elemental and prealloyed powders. In fact at the highest temperature the U.T.S. of compacts made from large particle size is marginally the greater of the two in the case of the elemental powder compacts, and in the case of prealloyed powder compacts the strengths are almost equal. This again goes to show that the temperature is the dominant effect in the hot forging operation in that it eliminates the cold working or particle size effects discussed previously, which give the higher strength, by encouraging recrystallisation and uses up the stored internal energy available from the cold working.

The strength results obtained can be related to the extent of recrystallisation present in the forgings as can be seen from tables 27, 28. A large increase in the extent of recrystallisation at the higher temperatures corresponds with an equally large drop in the value of the overall strength of the forgings. Similarly the small difference in strength noted between the forgings made at the lower temperatures reflect the small change in the extent of recrystallisation observed at these temperatures. These observations can be made for both the elemental and prealloyed powder forgings. There was an exception however, at the largest initial particle size for prealloyed powder. No significant change could be detected in the U.T.S. of the forging over the range of temperatures employed.

As was observed in the elemental powder forgings, only slight variations in pore size measurements were seen between the two initial particle sizes used (table 29). These slight differences did seem to suggest that an increase in initial particle size resulted in a finer pore size. A possible explanation for this slight variation in pore size noticed by increasing the particle size could be the fact that large particles result in larger but fewer interparticle pores due to the different packing characteristics. It is known that large pores are removed faster than small ones, therefore pore removal to a certain size would occur faster in compacts made from powders of large initial size. On reaching this critical size it becomes increasingly more difficult to remove the final traces of porosity. Hence, because of the few number of pores present originally in compacts made from large initial particle sizes, the number of pores at this critical size would be less than those made from small initial particle sizes. However, the statistical analysis performed proved this result to be insignificant at the levels considered. The highly significant effect once again proved to be that of forging temperature. This was as expected due to the critical effect an increase in this property has on the rate of recrystallisation and grain growth which are related to the efficiency of pore removal.

Due to the high densities obtained on forging the prealloyed compacts only small increases could be detected on raising the forging temperature (table 30).

As in the case of compacts prepared from elemental powders the only factor to have any visible effect on the hardness of the forgings was that of temperature. Increasing forging temperature resulted in the anticipated drop in hardness values as can be seen from table 31.

Section 2: Results of X-Ray Analyses

An investigation into the extent of homogenisation in the alloy formed was made on compacts forged from elemental and prealloyed powders. It was also hoped that this investigation would give an insight into the importance of powder selection in achieving the desired hot forged properties.

The first series of experiments was performed to investigate the homogeneity locally within the grains and across the grain boundaries for both sintered and hot forged compacts made from elemental powders. This investigation was carried out by taking line traces across a random selection of grains and grain boundaries as described in the section on experimental techniques. Two samples of sintered compacts were examined initially, the first had been cold compacted and sintered in a protective atmosphere for five minutes. The second had also been cold compacted but had been sintered for 20 hours. Examination of the traces obtained from the x-ray measurements revealed a steep concentration gradient in the case of the compact that had received a five minute sinter. The traces made for the compact sintered for 20 hours displayed a more gradual gradient indicating a higher degree of homogeneity. When similar investigations were carried out on the hot forged compacts which had received similar preliminary treatments no change in concentration was detected in either the grains or across the grain boundaries. This suggests that any inhomogeneities or segregation present had been completely removed by the hot forging operation even without complete recrystallisation. The traces obtained can be seen in figure 18.

As with the compacts prepared from elemental powders a test was performed on those made from prealloyed powders to examine for any inhomogeneities present within the grains or across the grain boundaries. This was again done by making line traces across suspected grains to check for any evidence of concentration gradients. It was thought that variations in the concentration gradients might alter the mechanisms of the recrystallisation processes occurring during the hot forging operation. However, examination of the x-ray results obtained eliminated this possibility, as once again, no variation in concentration gradient could be detected either within the grain or across the grain boundary. A typical area examined is shown in figure 28. These results suggest that the ultimate extent of homogenisation is similar for both compacts prepared from elemental powders and those prepared from prealloyed powders when concentration gradients are measured across grain boundaries.

The second series of experiments was aimed at examining the extent of homogenisation present in the compact formed on a larger scale, i.e. to determine the homogeneity of the alloy formed by the hot forging operation. Once again the experimental procedure employed was detailed in the section on experimental work. An evaluation of the copper/nickel system has previously been carried out by Kuhn and Lawley (107). They suggest that the initial stages of homogenisation proceed faster than predicted indicating that diffusion occurs via the extensive interconnected interparticle surfaces and possibly the grain boundaries. The later stages proceed slower than predicted probably due to the effects of imperfect mixing and/or small changes of copper particle sizes about the mean. They also observed that the copper rich peaks broadened faster than the nickel rich peaks and that non-uniform particle sizes can result in decreased homogenisation kinetics.

The results of the x-ray analysis obtained for the current series of experiments are presented in table 32 giving details of the forging treatment, half peak breadth and the corresponding extent of recrystallisation measured. Examples of the traces obtained from the x-ray analysis are given in Appendix 3.

The main observation from these results is that the extent of homogenisation in the prealloyed and elemental powder prepared compacts is much closer than expected following the hot forging operation, and no apparent relationship could be seen between the extent of recrystallisation and the broadening of the x-ray trace. The extent of homogenisation is high when compared with that observed in a compact that has merely received a sintering treatment. This effect is emphasised even further when compared to the amount of homogenisation present in the cast material where steep concentration gradients can be measured as a result of the coring and segregation present. Diffusion rates are much lower in the latter two cases hence a much longer period of treatment is required for complete homogenisation. The use of powdered metals is advantageous due to the much smaller diffusional distances because of the close contact of the particles. Whereas in the cast material large distances can occur between areas of different concentration, i.e., the interdendritic distances can be quite appreciable. This indicates that the mechanical element of the hot forging operation is a key factor in producing the dramatic increase in the rate of homogenisation by means of the additional energy provided. This energy is absent in the case of sintered or cast alloys.

These results indicate therefore the similarities between compacts prepared from elemental powders and those prepared from prealloyed powders. However, these similarities are merely chemical, there are many mechanical differences based on the difference in extent of recrystallisation between the compacts made by the two methods. It is the difference in amount of energy available that gives rise to the difference in extent of recrystalliation. The most important factors which affect recrystallisation processes are temperature and amount of deformation, another factor that also affect recrystallisation is purity or homogenisation of the material. As both the temperature and amount of deformation are the same for compacts prepared from elemental and prealloyed powder, the governing factor must be the difference in homogeneity. It is known that for a homogenous alloy the stacking fault energy is at a minimum hence the stored energy is at its maximum. In the case of an inhomogenous alloy as in the case of compacts prepared from elemental powders areas of high stacking fault energy exist which tend to lower the total stored energy available. As homogenisation is approached the amount of stacking fault energy decreases giving a corresponding increase in stored energy. Therefore it can be seen that compacts prepared from prealloyed powders have a greater amount of energy available for recrystallisation due to their higher initial homogeneity. Compacts made from elemental powders on the other hand have less energy available and subsequently only achieve recovery of the worked material. Another factor that delays recrystallisation in the case of the elemental powders is that the inhomogeneities also act as obstacles to grain boundary movement hence reducing the rate of recrystallisation. This indicates that complete homogeneity is essential if the maximum amount of recrystallisation is to occur. Again in the case of the elemental powder compacts part of the time taken for the forging stroke is needed for this homogenisation resulting in less time being available for recrystallisation. It is considered that a combination of these factors gives rise to the difference in extent of recrystallisation observed between alloyws formed from elemental powders and those formed from prealloyed compact. However, as is often the case, an exception does occur, the one observed in the current series of experiments was discussed in an earlier section and a possible explanation given. It is considered that further work is required in this area to provide a more comprehensive explanation for the phenomenon observed.

The mechanical properties observed reflect the importance of the homogenisation and recrystallisation processes in attaining the desired forging properties.

Section 3: Metallographic Observations

Elemental Powders

The specimens for metallographic examination were prepared by the standard methods, 0.05 alumina powder being used for the final polish. A solution of:

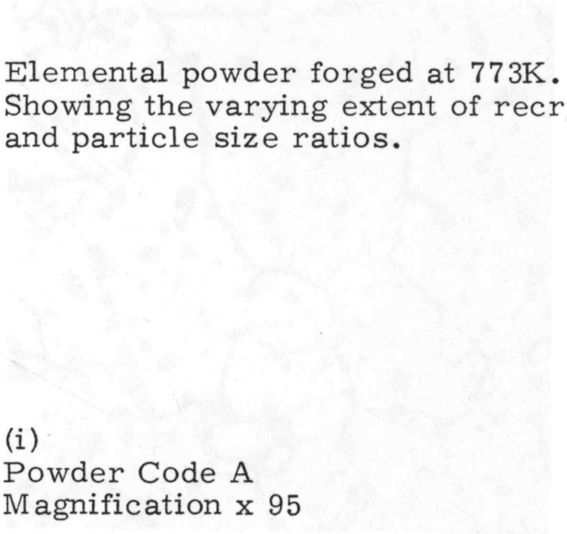
4 gms	Potassium dichromate
3 gms	Sodium chloride
16 mls	canc. sulphuric acid
200 mls	Distilled water

was found to be the best etchant.

Macroscopic examination of the forged samples revealed a pore envelope around the edges of the forgings indicating the presence of dead metal zones. These are formed by the effects of die wall friction which reduces the metal flow, hence inhibiting pore reduction thus resulting in a higher degree of propsity in this region.

Microscopic examination of the prepared samples revealed a changing microstructure with every forging temperature used. Examples of the microstructures obtained can be seen in Figs. 22, 23, 24. The forging carried out at 773 K and 873 K are very similar in both grain size and porosity content, the difference being a slight increase in the extent of recrystallisation observed in the higher temperature forging. Neither of the lower temperature forgings showed much change in the grain size; some of the annealing twins from the singering stage still remained, whereas others appeared bent and deformed as if by 'cold working'. However, it can be seen that the structure obtained by forging at 973 K is completely different - The original grain size can be seen to be slightly larger than that observed for the lower temperature forgings, but within these large grains are numerous

Elemental powder forged at 773K.
Showing the varying extent of recrystallisation with different particle size and particle size ratios.



(i)
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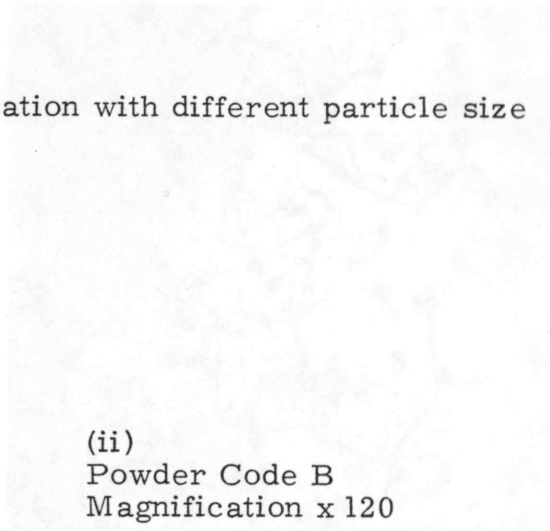
Magnification x 95



(ii)
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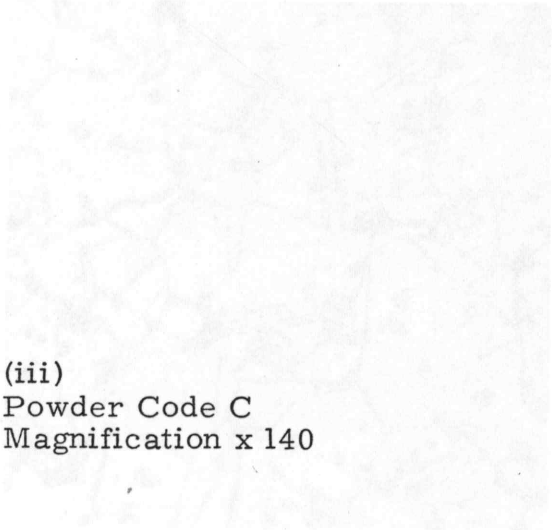

(iii)
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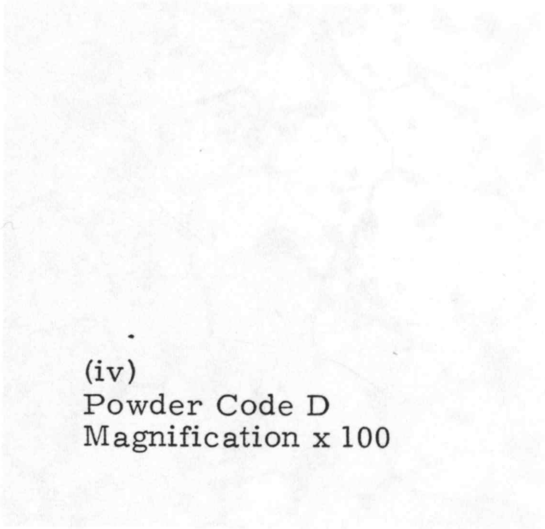
Magnification x 140



(iv)
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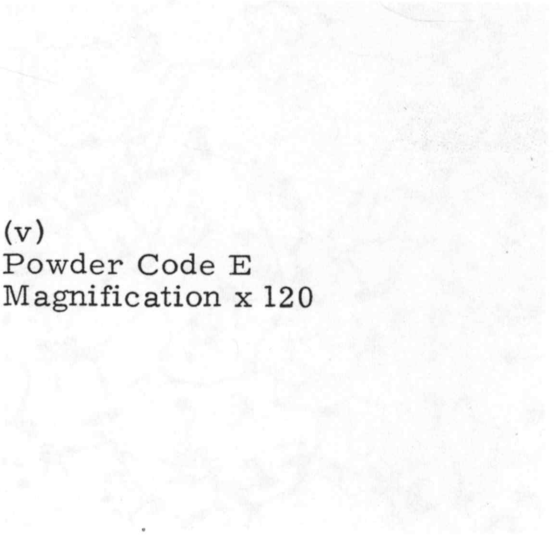

(v)
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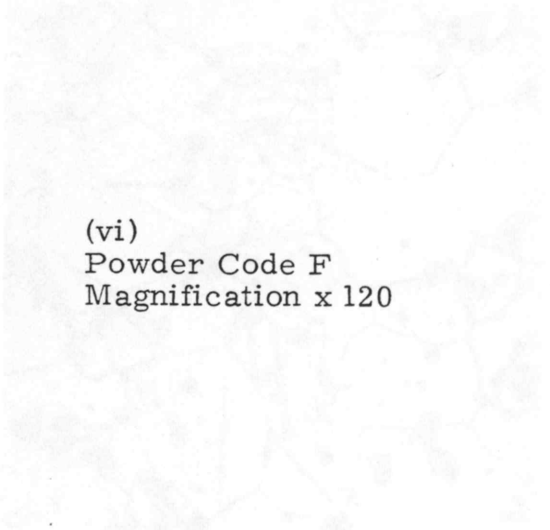
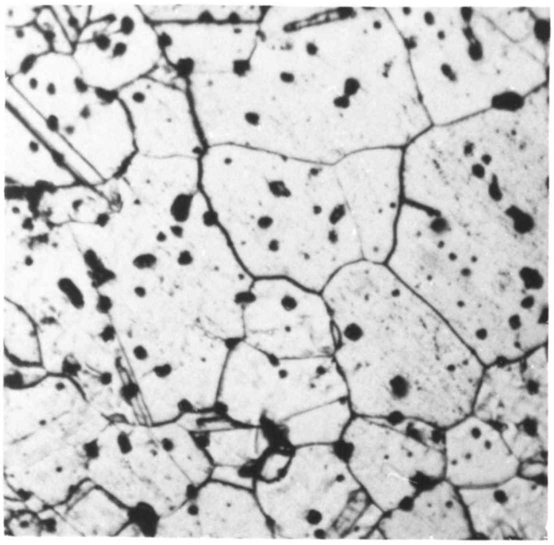
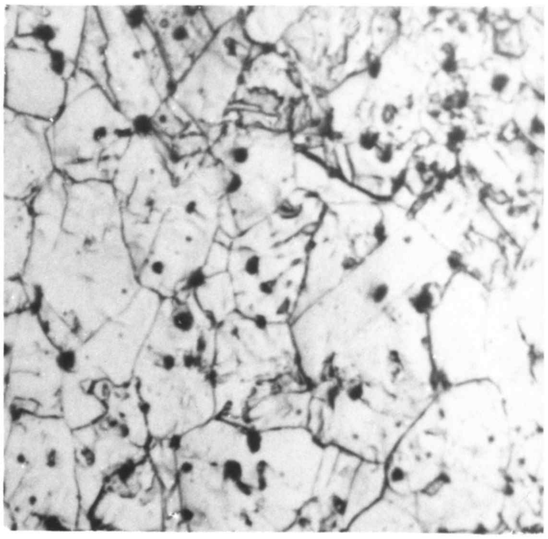
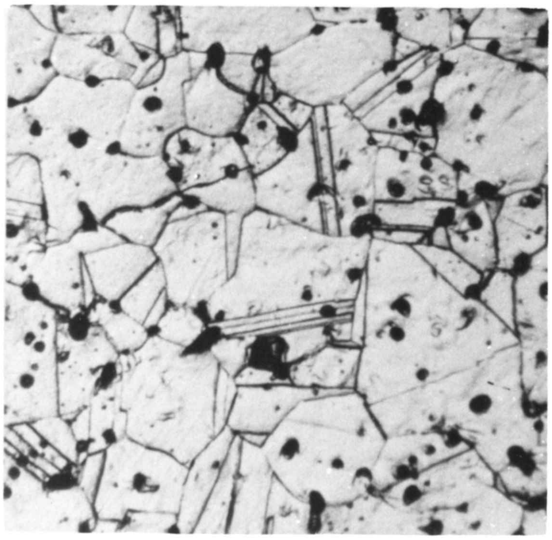


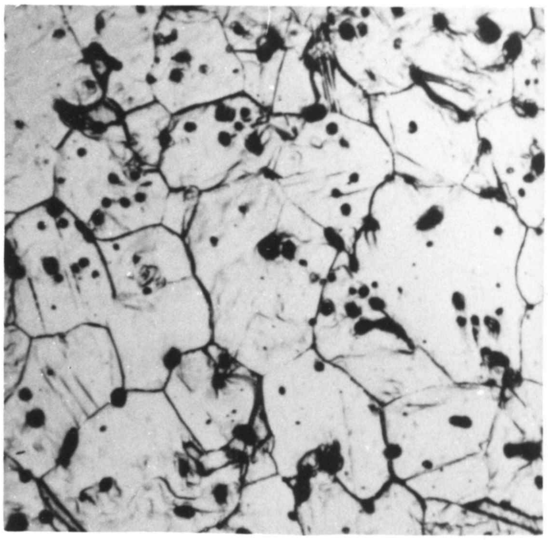
(vi)
Powder Code F
Magnification x 120

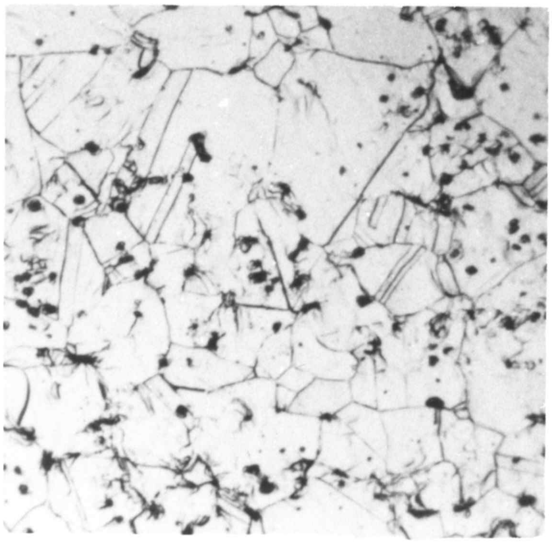
Fig. 22

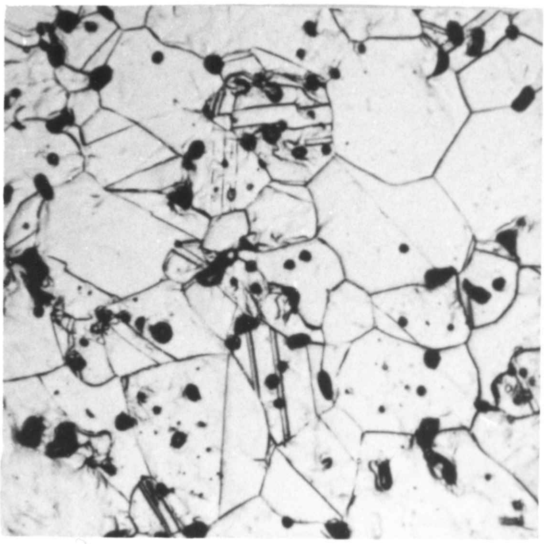




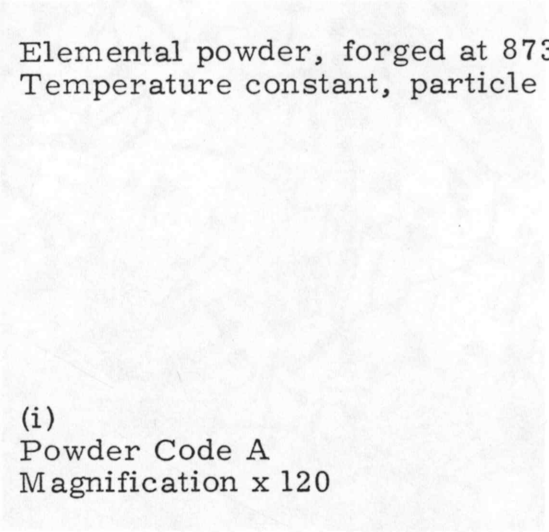








Elemental powder, forged at 873K.
Temperature constant, particle size and size ratio varying.



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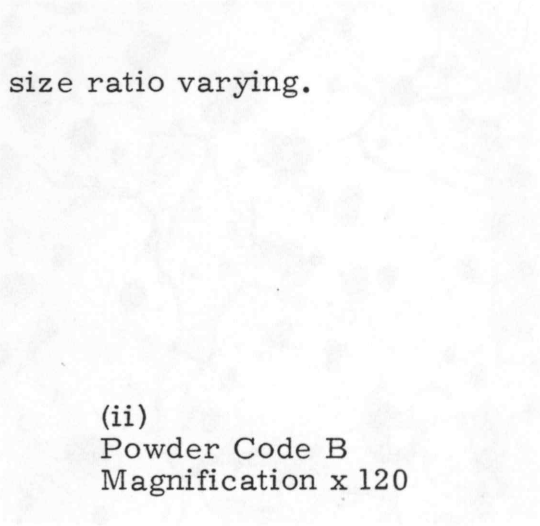

(ii)
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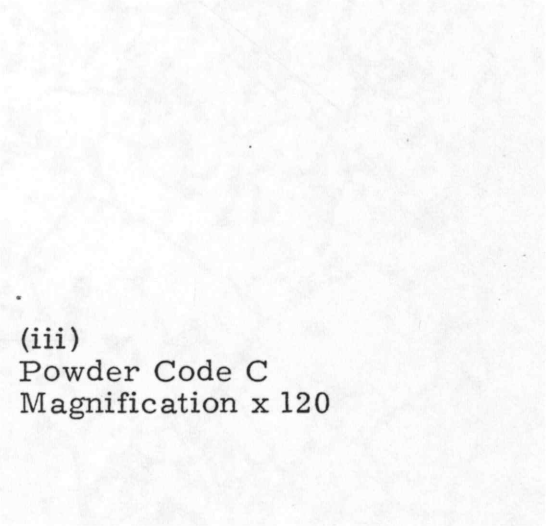

(iii)
Powder Code C
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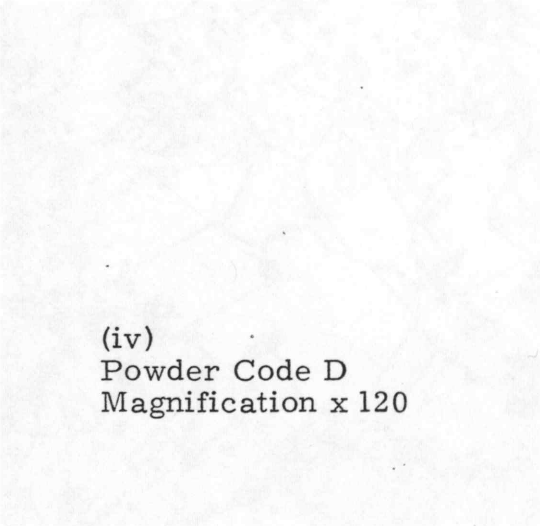
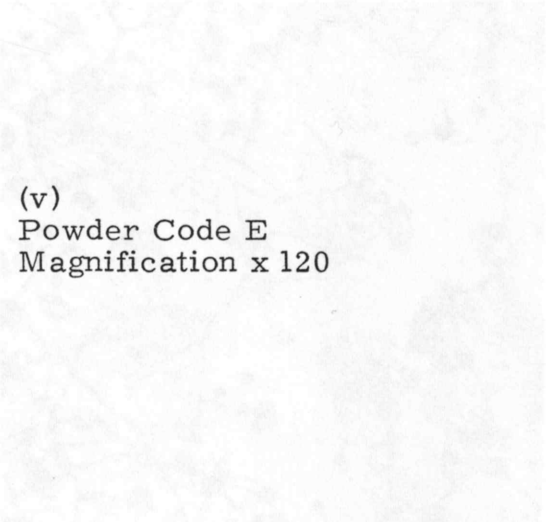


(iv)
Powder Code D
Magnification x 120



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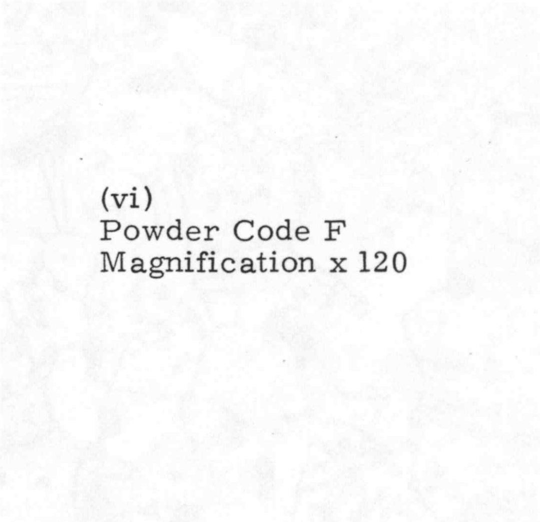
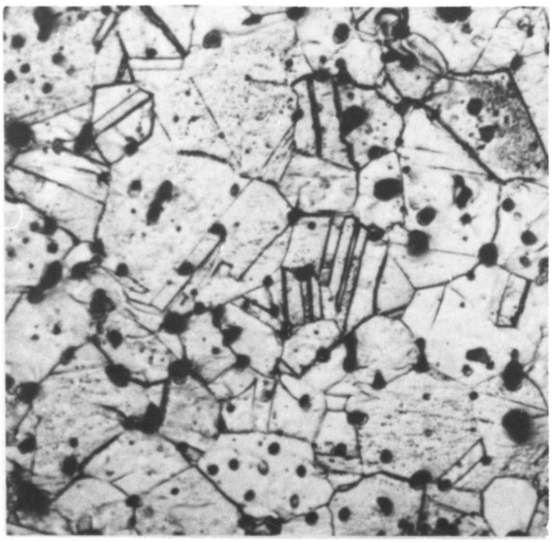
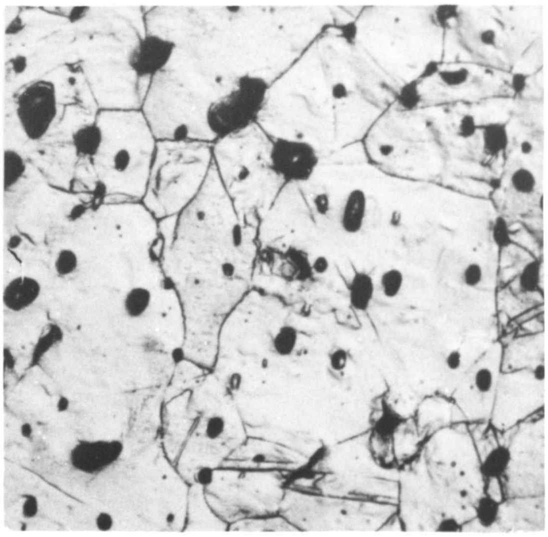
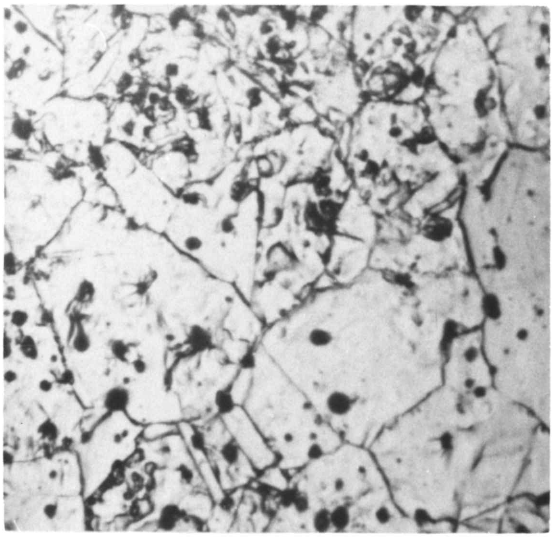


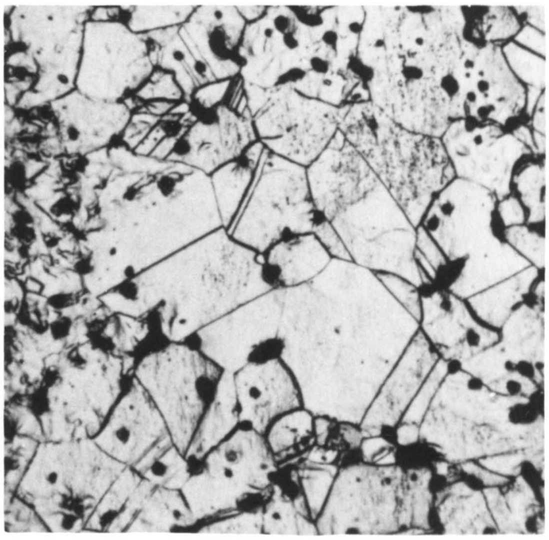
(vi)
Powder Code F
Magnification x 120

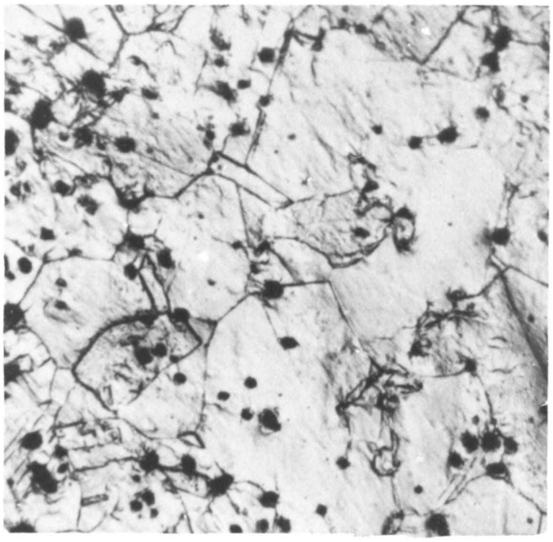
Fig. 23

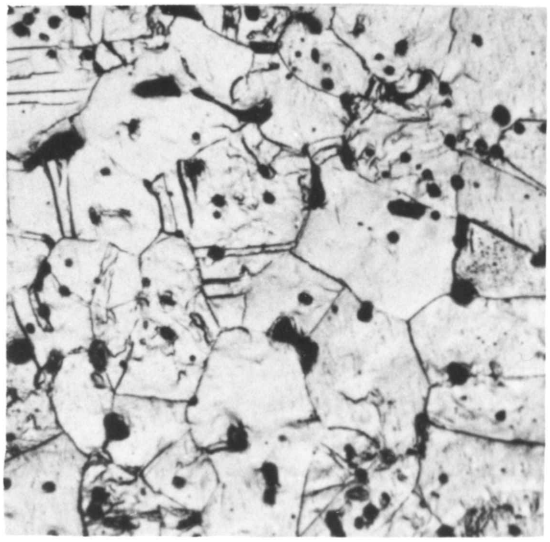




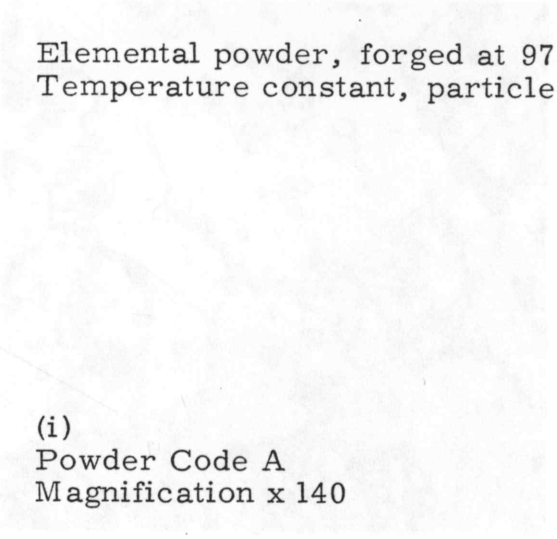




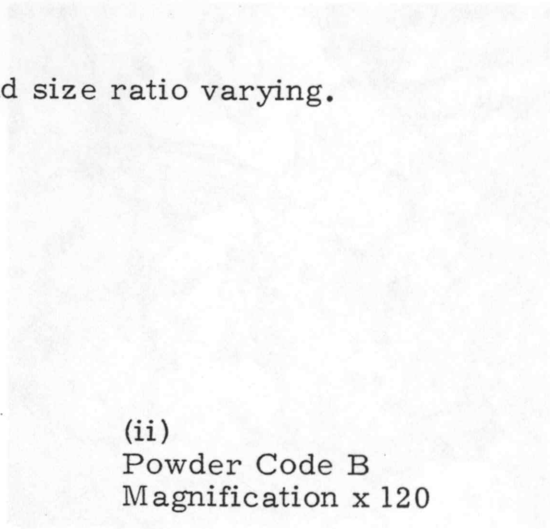




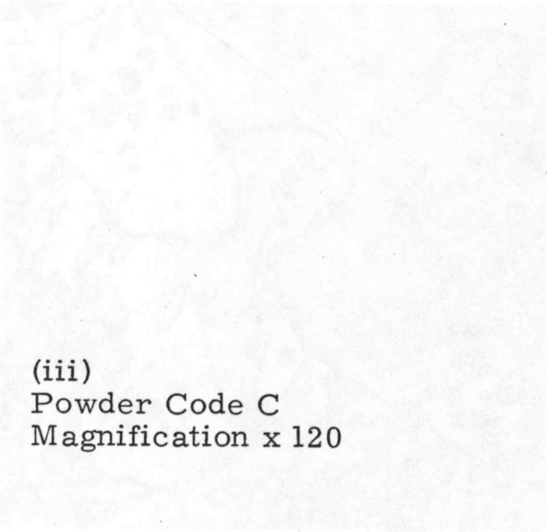
Elemental powder, forged at 973K.
Temperature constant, particle size and size ratio varying.



(i)
Powder Code A
Magnification x 140



(ii)
Powder Code B
Magnification x 120



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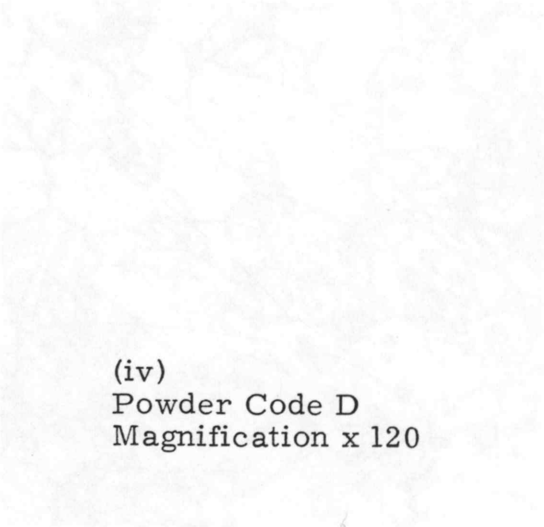

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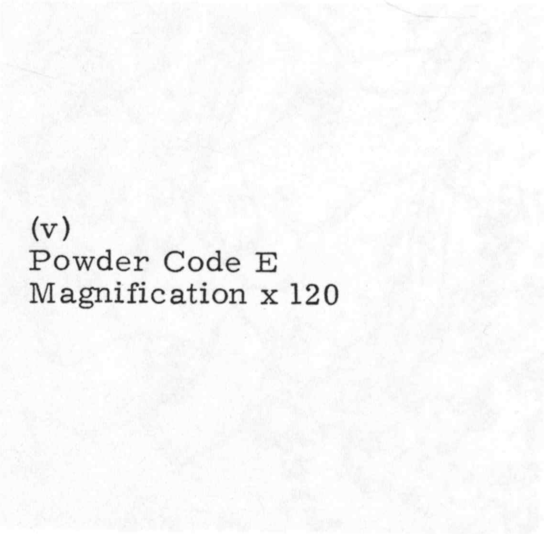

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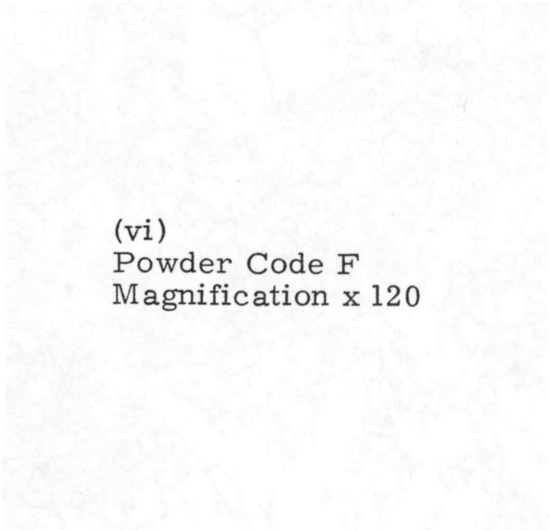
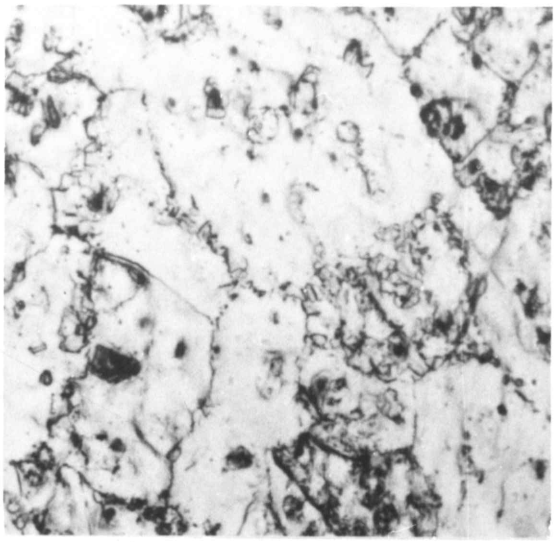
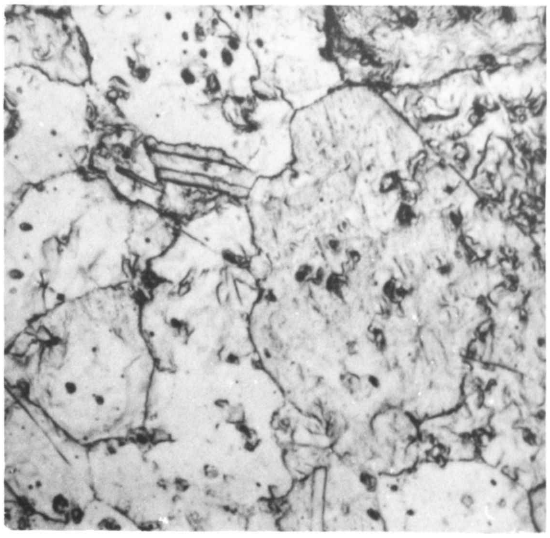
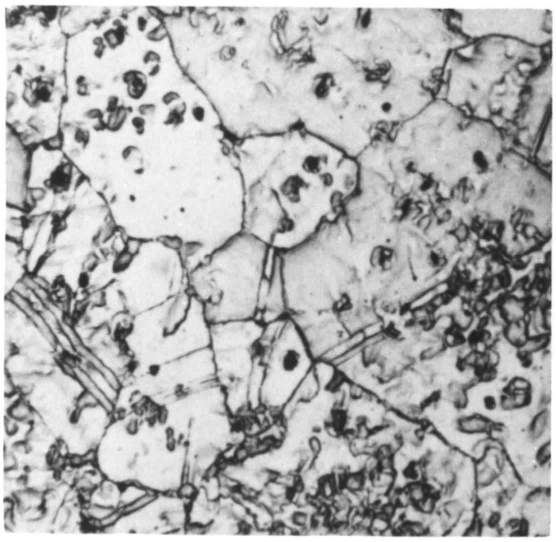


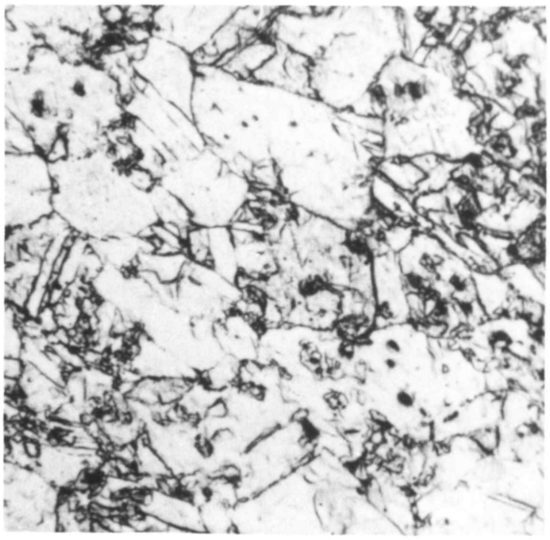
(vi)
Powder Code F
Magnification x 120

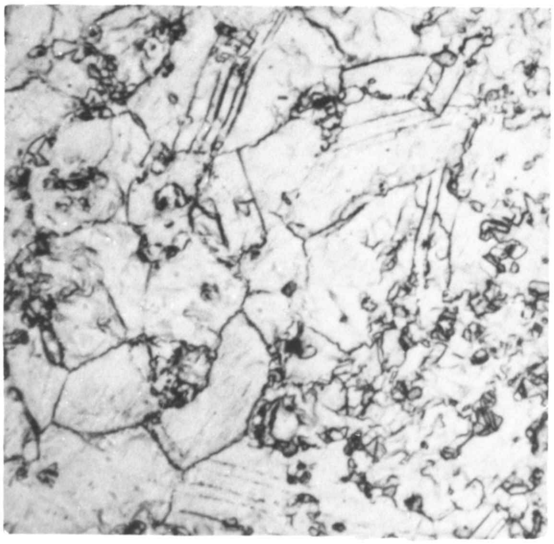
Fig. 24

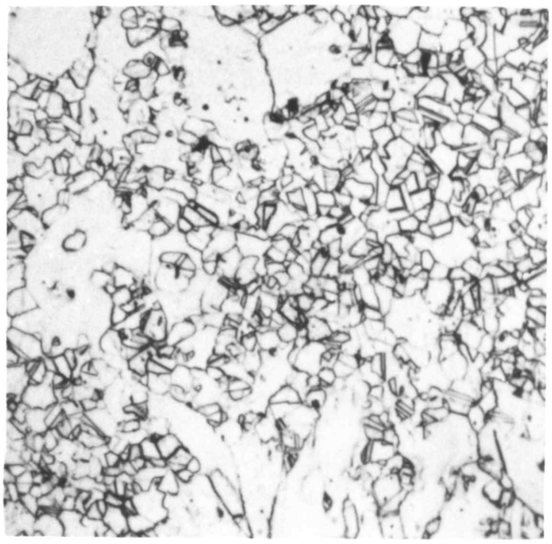












fine recrystallised grains. These recrystallised grains appear to nucleate at the high energy points of the grain boundaries, twin intersections and boundaries and also in the grain interiors. It is these fine grains within the original grains that give an overall reduction in grain size for the forgings completed at higher temperatures. Grain size measurements were made by the linear intercept method, fifty counts being taken for each reading. Distortion of the grain boundaries and twins is clearly evident in the low temperature forgings which reflects the severe working of the compact brought about by the forging operation. Distortion is also evident within the grains giving them a wavy appearance as can be seen in the 873K forgings (Fig. 23).

The temperatures used in the forging operation are well below those normally used for sintering, therefore when no recrystallisation occurs the densification is small and arises mainly from the mechanical deformation of the pores. However, once recrystallisation begins, the rate of densification is very rapid, as the forging is over in only a few seconds, and occurs by removal of the pores along the grain boundaries and by grain growth. It is therefore clear that recrystallisation is highly desirable for the production of greater densification as it enhances the spherodisation of voids due to the increased number of grain boundaries. This spherodisation of the pores increases the rate of grain growth which assists in further pore removal and hence greater densification. The amount of cold work given is also an important factor in controlling the extent of recrystallisation at it increases the number of points of high lattice strain energy which then act as nucleii for recrystallisation. From Fig. 24 it can be seen that recrystallisation occurs preferentially at these high energy points in the structure, i.e. at distorted grain boundaries and twin intersections. The appearance of recrystallised grains within the original grains is probably due to the fact that these areas are again twins running in different directions and have not been highlighted by the metallographic preparation.

Various stages of the recrystallisation sequences can be seen for the elemental powder forging in Figs 25, 26, they show the nucleation points for the recrystallised grains at the grain boundaries and other areas of high lattice strain. The advancement of the recrystallisation process and grain growth can also be seen for the higher forging temperatures.

The extent of recrystallisation was measured by means of a spot chart, 500 counts being made for each reading obtained. In practice it is both inconvenient and difficult to project a random array of points onto a microstructure, consequently a regular or systematic array of points is used on the assumption that the dispersion of phases in the microstructure is random. A square grid was used in the current case and superimposed onto the microstructure using a projection microscope screen. The total number of points on the grid falling on the areas to be measured were counted and compared with the total number of points laid down (101). From the results obtained it can be seen that the percentage recrystallisation for each forging temperature falls on the sigmoidal curve produced when these two parameters are plotted against each other. The microstructures show that a greater amount of recystallisation occurs when the largest initial particle size is used. This fact was discussed in the section on mechanical testing.

Prealloyed Powders

Prealloyed powder compacts were prepared in exactly the same way as those made from elemental powders. Macroscopic examination again revealed the presence of dead metal zones indicated by the pore envelope around the forgings. The microstructure was similar to that observed for forgings made from elemental powders in that it was continually changing with increase in forging temperature.

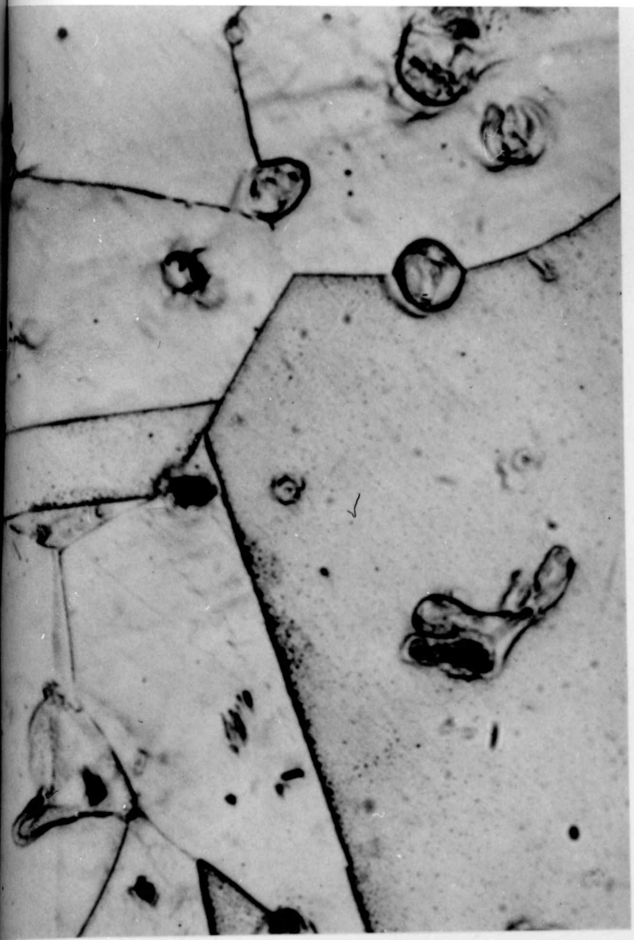
Fig. 25 Photographs of the recrystallisation sequence for elemental powder

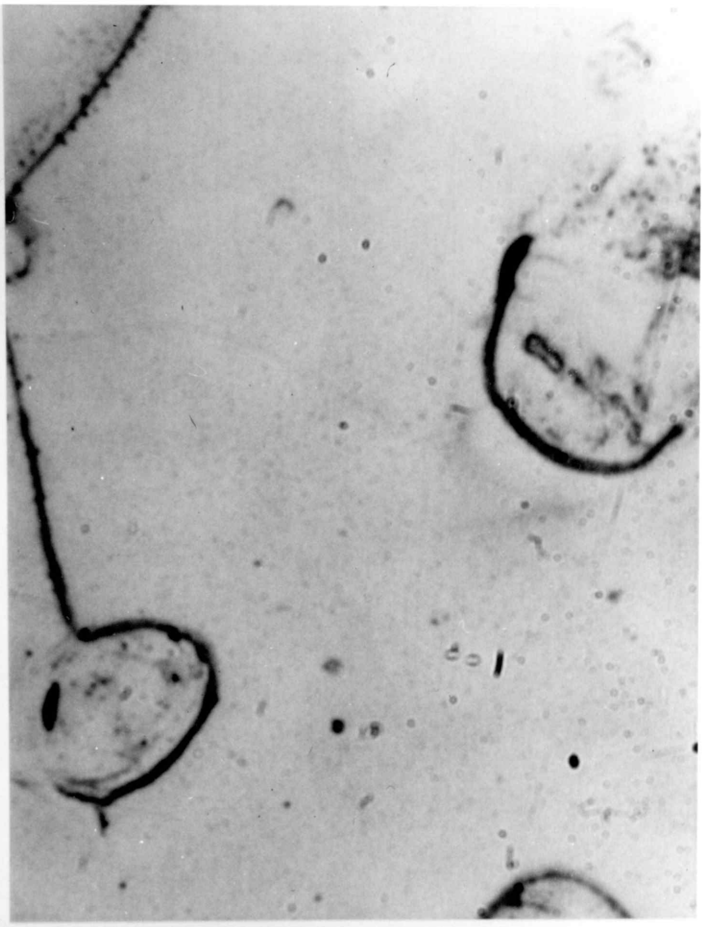
(i) Forging Temperature 773K
Magnification x 600

(ii) Forging Temperature 773K
Magnification x 1200

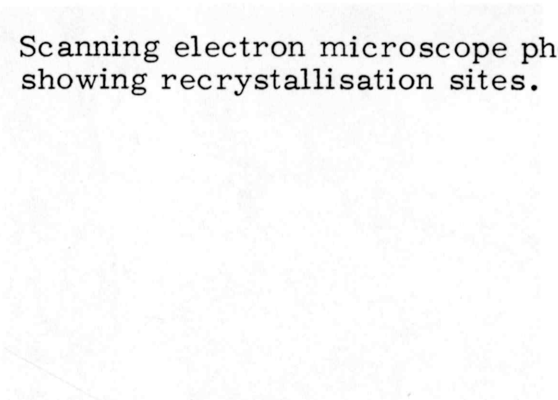
(iii) Forging Temperature 973K
Magnification x 400

(iv) Forging Temperature 973K
Magnification x 1000

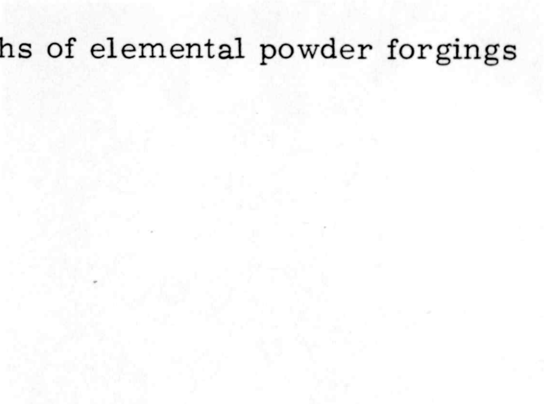




Scanning electron microscope photographs of elemental powder forgings showing recrystallisation sites.



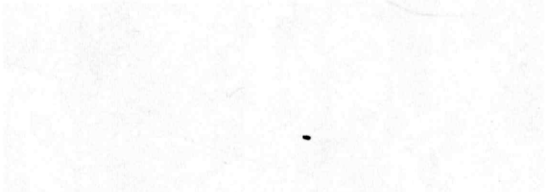
(i)
Powder Code E
Forging temperature 973K
Magnification x 1K



(ii)
Powder Code D
Forging temperature 973K
Magnification x 1K

(iii)
Powder Code C
Forging temperature 973K
Magnification x 1K

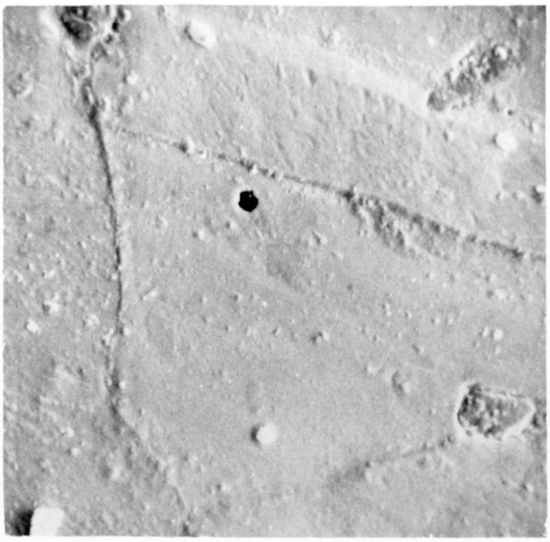
(iv)
Powder Code A
Forging temperature 973K
Magnification x 1K

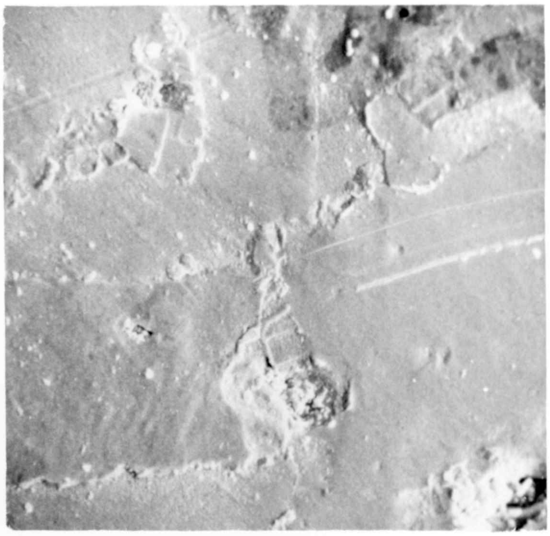


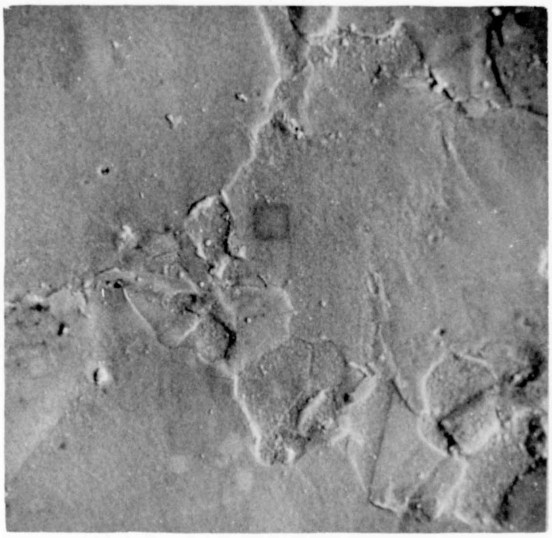
(v)
Powder Code F
Forging temperature 973K
Magnification x 2K

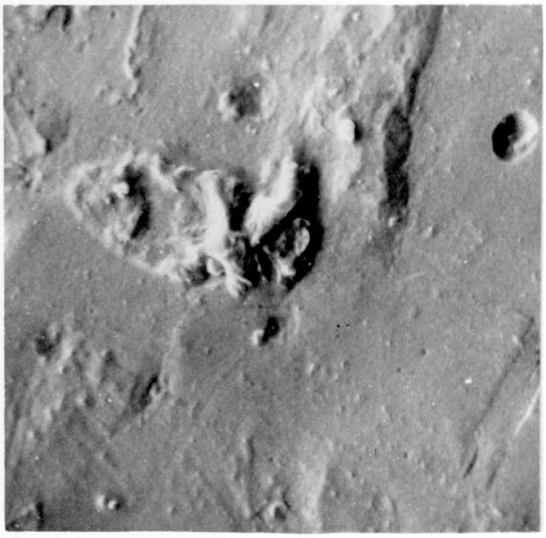
(vi)
Powder Code C
Forging temperature 973K
Magnification x 1K

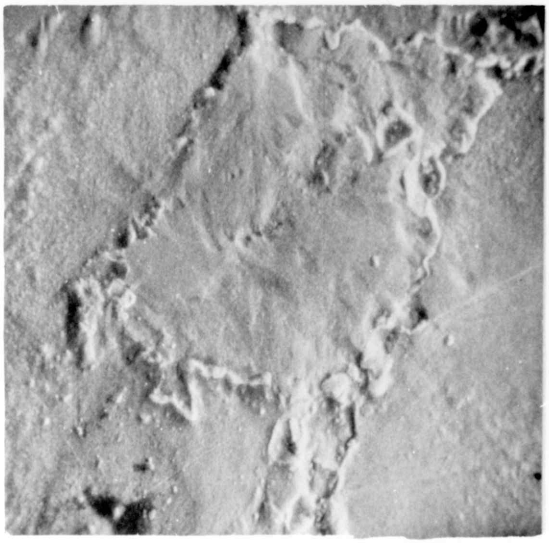
Fig. 26











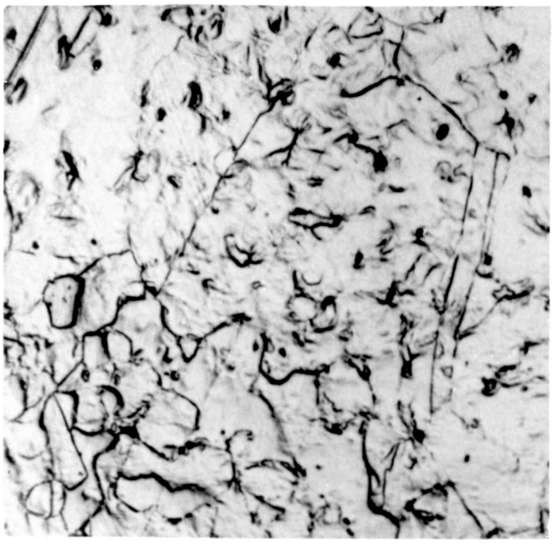
However, it appeared that the extent of recrystallisation was slightly more advanced at each forging temperature than was observed in the case of the elemental powder forgings, especially for the small initial particle size. The difference was much greater at the highest temperature. Examples of the microstructures observed for prealloyed powder forgings can be seen in Fig. 27 – 31.

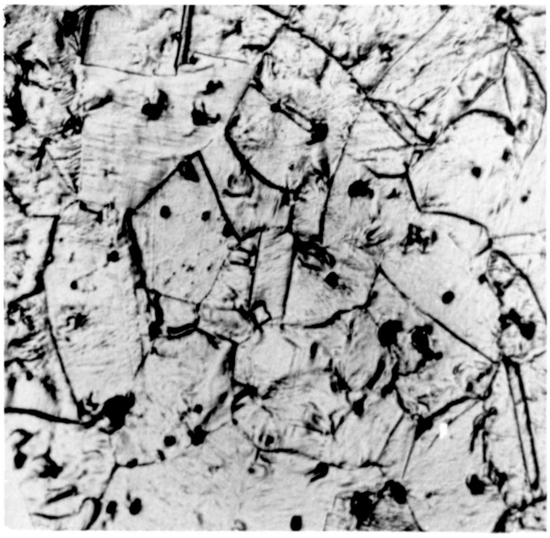
For the lower temperature forging not much grain boundary distortion can be seen in the case of large initial particle sizes, the twins are mainly undistorted however small recrystallisation sites can be seen at the distortions present. The original grain size can also be seen to be slightly larger than when small initial particale sizes are used. When the small particle sizes are used there is a definitive difference in the microstructure. Grain boundary migration is evident, twins are more distorted, grain interiors have a wavy appearance as was observed in the case of elemental powders and a more advanced state of recrystallisation is witnessed as can be seen by examining Fig. 27. Examination of the microstructures available for the 873 K and 973 K forgings shows a continuation of the trend of more extensive recrystallisation in the case of small initial particle size. This suggests that decreasing the initial particle size is beneficial in increasing the extent of recrystallisation. A possible reason for this is because of the extra number of grain boundaries and high energy sites available for nucleation, which in turn enhances the properties of the material. This observation has in fact been made by other researchers (55) but is the opposite to that observed for compacts made from elemental powder. It is thought that the degree of homogeneity in the compact prior to forging controls the extent of recrystallisation. This suggestion was dealt with in greater detail in the section on mechanical testing.

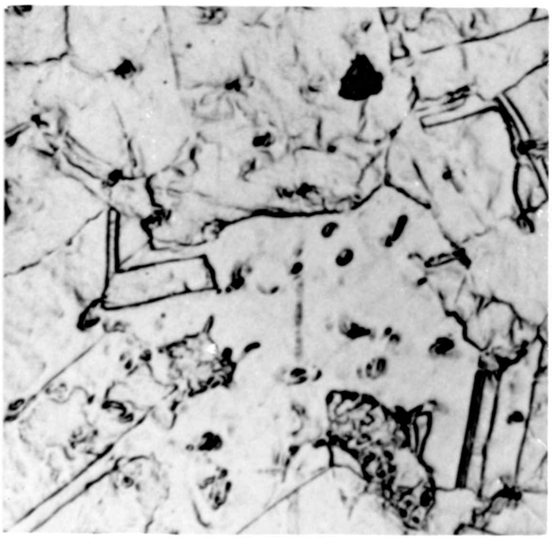
Comparison between small and large prealloyed powder forgings.

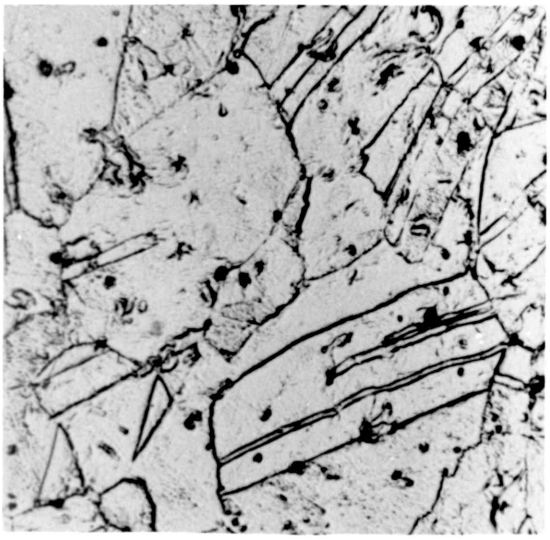
Small Particles	Large Particles
(i) Forging temperature 773K Magnification x 100	(ii) Forging temperature 773K Magnification x 70
(iii) Forging temperature 873K Magnification x 120	(iv) Forging temperature 873K Magnification x 70
(v) Forging temperature 973K Magnification x 140	(vi) Forging temperature 973K Magnification x 120

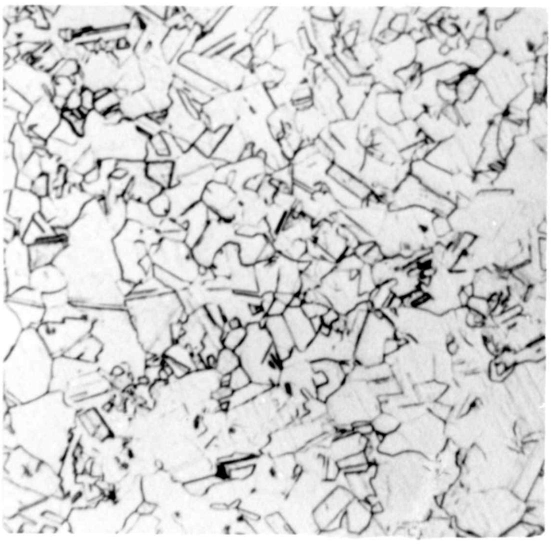
Fig. 27











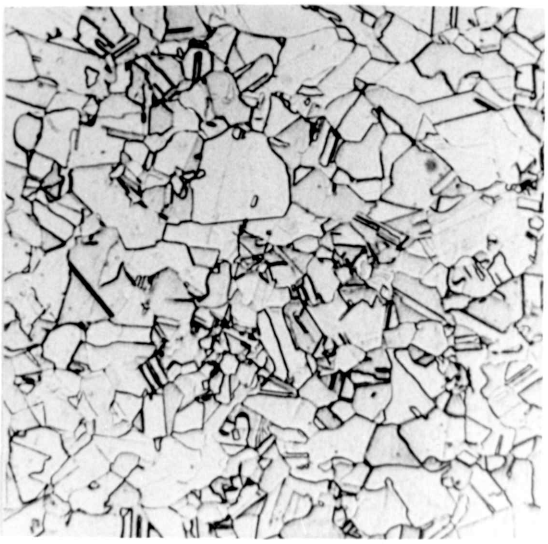
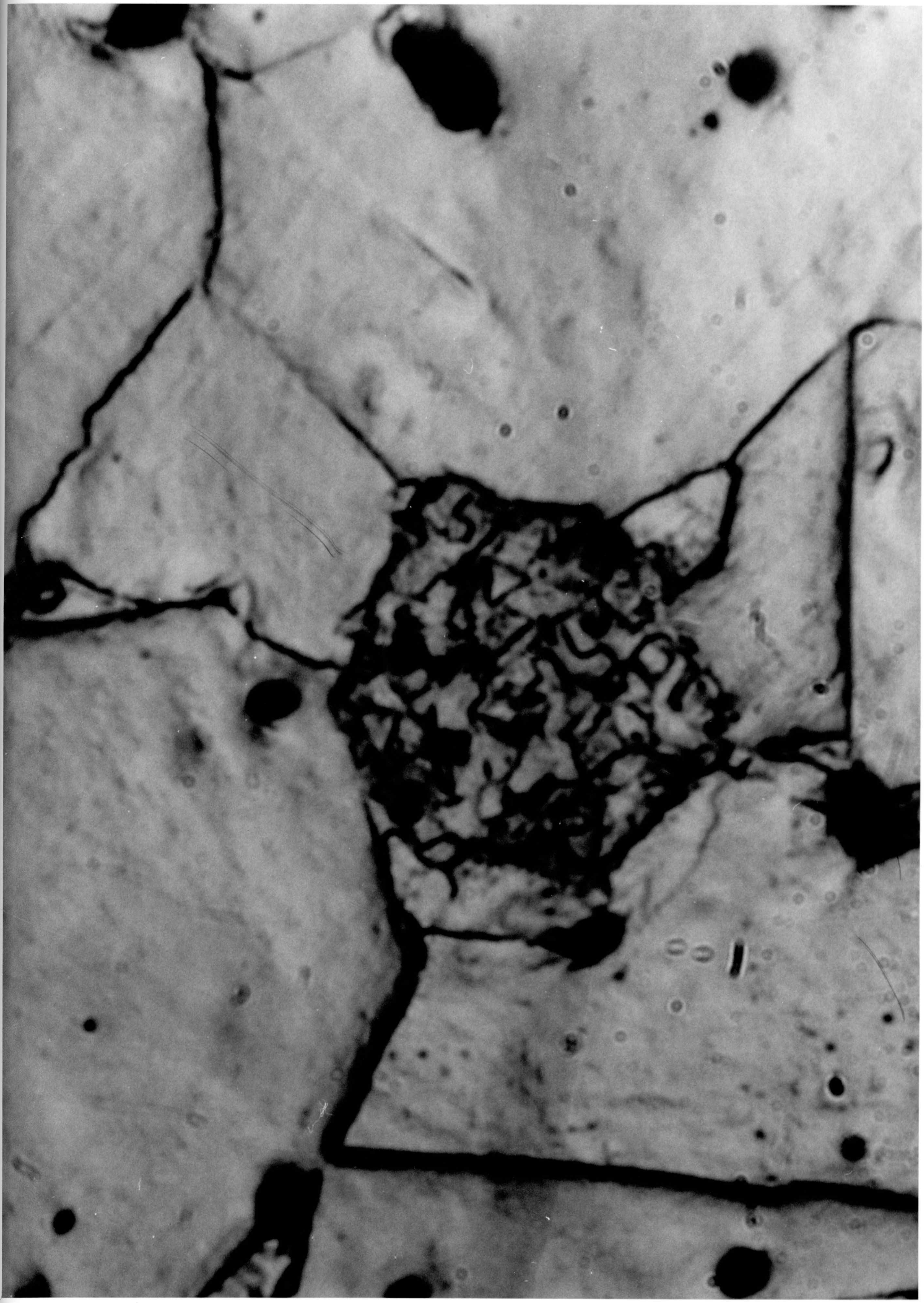


Fig. 28

Sample used for Concentration Gradient Measurement



Small prealloyed powder, recrystallisation sequence showing grain boundary migration and development of nuclei on both grain boundaries and grain interiors.

(i)
Forging temperature 773K
Magnification x 150

(ii)
Forging temperature 773K
Magnification x 200

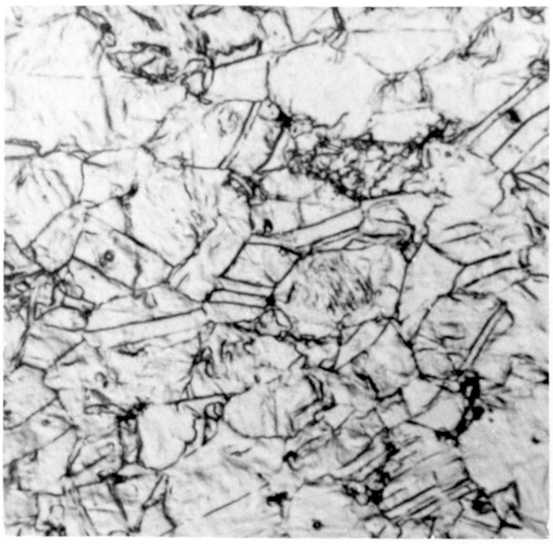
(iii)
Forging temperature 773K
Magnification x 350

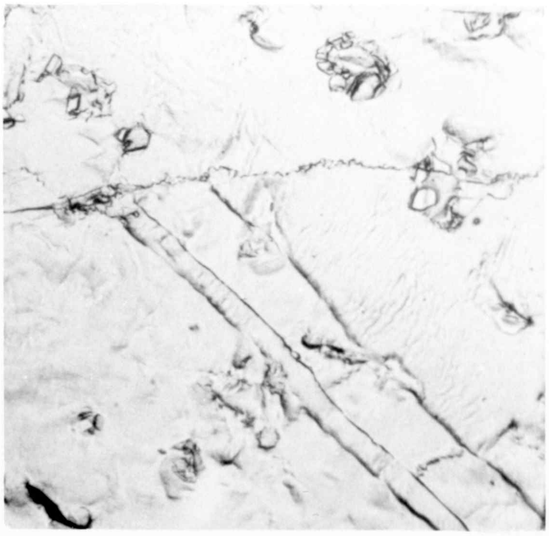
(iv)
Forging temperature 873K
Magnification x 95

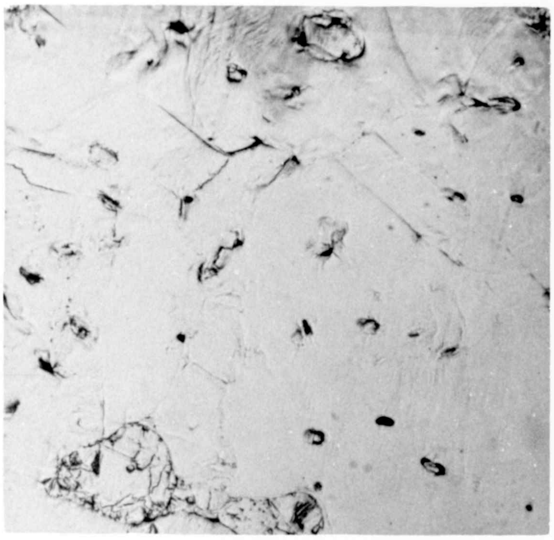
(v)
Forging temperature 873K
Magnification x 300

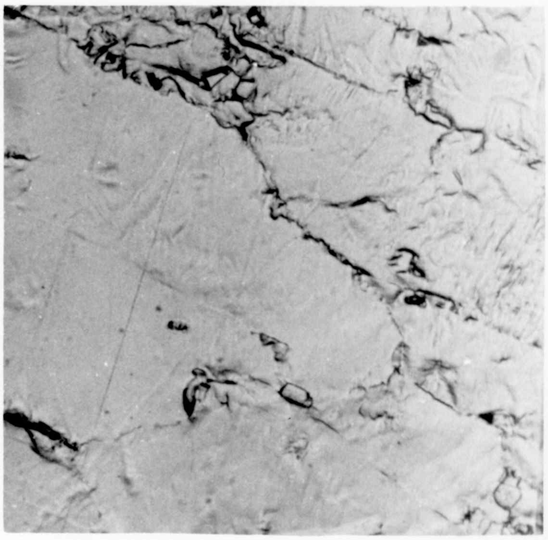
(vi)
Forging temperature 973K
Magnification x 300

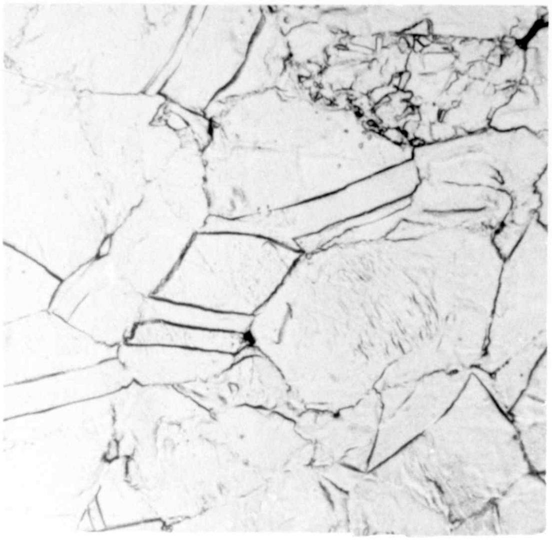
Fig. 29

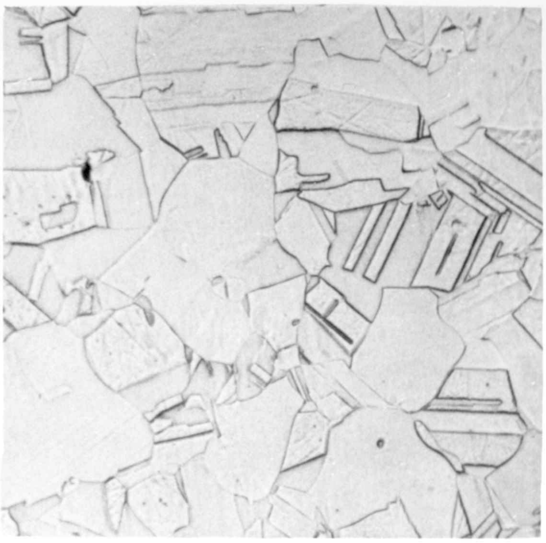












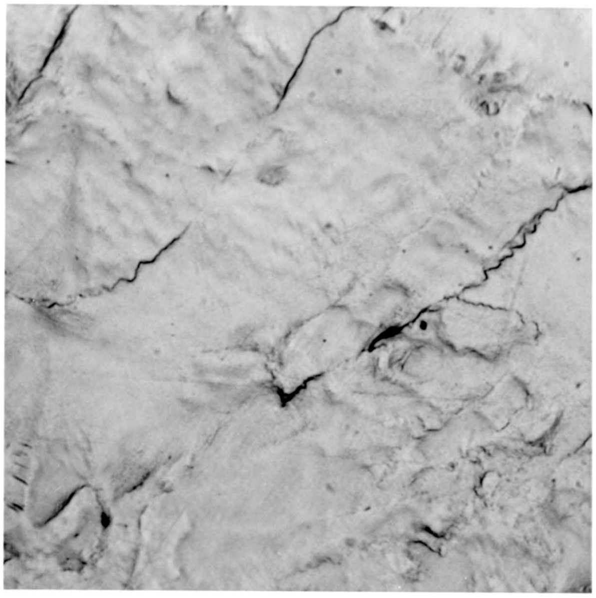
The photographs show the recrystallisation sequence for small prealloyed powders forged at 873K.

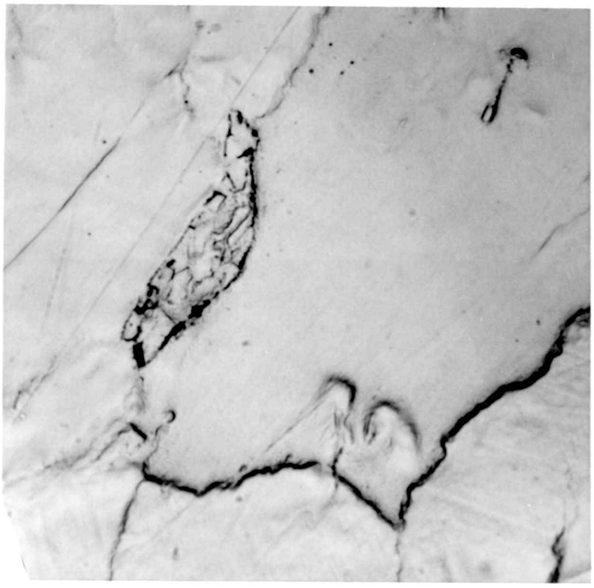
(i)
Magnification x 400
Onset of grain boundary migration

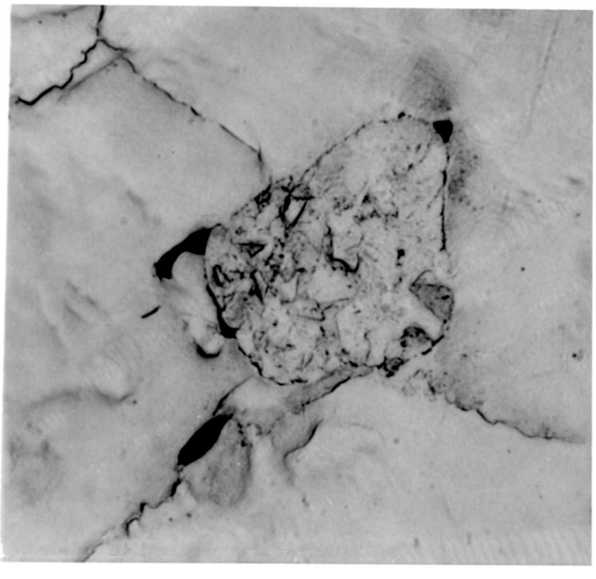
(ii)
Magnification x 400
Appearance of small grain on the boundary

(iii)
Magnification x 400
Development of the grain with recrystallisation occurring in its interior

Fig. 30







The photographs show the recrystallisation sequence observed for larg
prealloyed powder forgings.

(i)
Forging temperature 773K
Magnification x 200

(ii)
Forging temperature 973
Magnification x 100

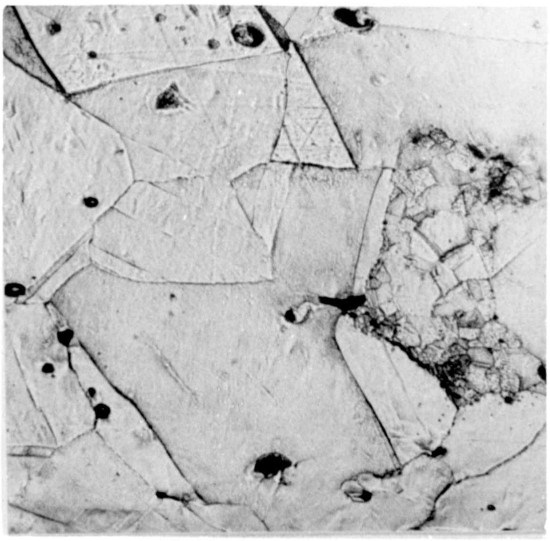
(iii)
Forging temperature 873K
Magnification x 200

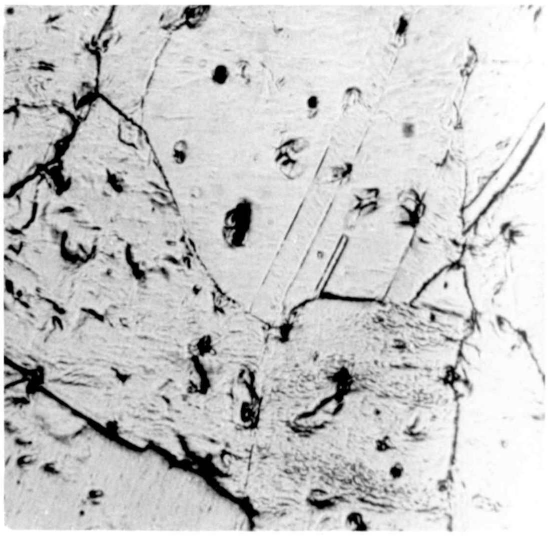
(iv)
Forging temperature 973
Magnification x 550

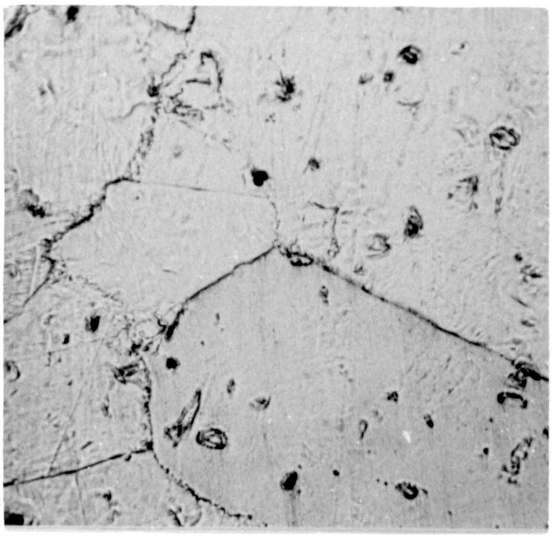
(v)
Forging temperature 873K
Magnification x 350

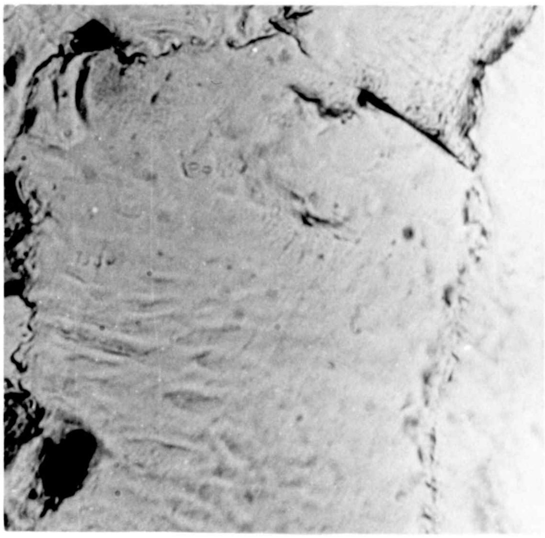
(vi)
Forging temperature 973
Magnification x 120

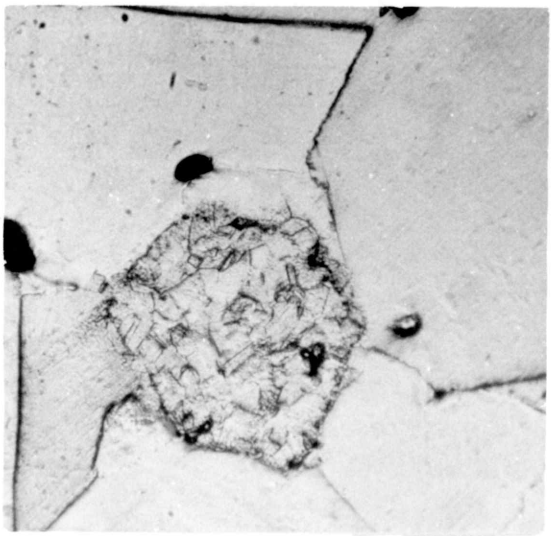
Fig. 31

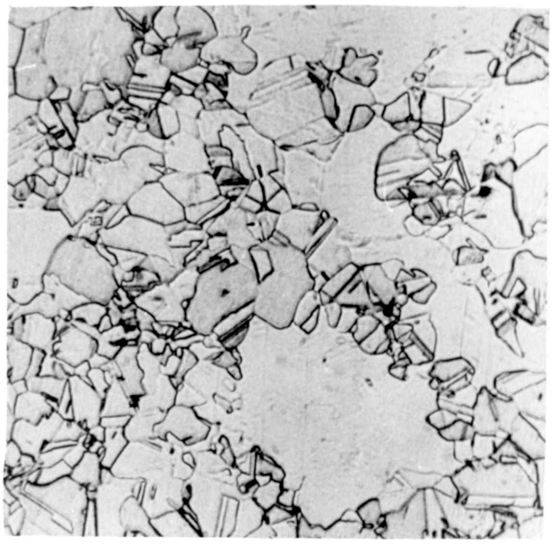












A model for recrystallisation behaviour has been proposed by Evans and McColvin (102) in which they state that the forged material consists of a mixture of material with intrinsically different mechanical properties. These differing mechanical properties can be explained on the basis that the material is in fact a mixture of phases, i.e. cold worked copper, recrystallised copper and pores. Assuming that at yield the strain the individual components is the same, the proof stress is then given by:

$$\sigma = \sum \sigma_i \nu_i \qquad \ldots\ldots (1)$$

where ν_i = volume fraction of the phase with proof stress σ_i

i = 1 for cold worked copper

i = 2 for recrystallised copper

i = 3 for pores

Variation in ν_2 with temperature is sigmoidal and can be represented by:

$$\nu_2 = 1 - e\left[-(CT)^n\right] \qquad \ldots\ldots (2)$$

C, n are constants

The variation of ν_3 with temperature is also sigmoidal and can be represented by:

$$\nu_3 = \alpha \exp\left[-(dT)^m\right] \qquad \ldots\ldots (3)$$

d, m are constants and α is the value of ν_3 for the lowest forging temperature.

It is clear that $\nu_1 = 1 - \nu_2 - \nu_3 \qquad \ldots\ldots (4)$

Combining 1, 2, 3, 4 and assuming $\nu_3 = 0$, we have

$$\sigma = \sigma_2 + \exp\left[-(CT)^n\right]\left[\sigma_1 - \sigma_2\right] - \sigma\,\alpha \exp\left[-(dt)^m\right] \ldots\ldots (5)$$

It was found that the experimental and predicted values agreed well both in previous work carried out (103) and in the present work. However, the analysis cannot be extended to U.T.S. as the flow stress of recrystallised grains increases during testing towards that of the cold worked material.

A similar analysis was made for the ductility of the material based on an approximation that

$$\frac{1}{\varepsilon} = a + b\upsilon \qquad \ldots\ldots (6)$$

ε = ductility at fracture

a, b = constants

υ = total volume fraction of hard phases and pores $(\upsilon_1 + \upsilon_3)$

then $\frac{1}{\varepsilon} = a + b\left\{\exp\left[-(CT)^n\right]\right\} \qquad \ldots\ldots (7)$

Poor agreement was found with observed values of proof stress, however this can be attributed to the fact that the effect of pore size distribution was ignored in equation (6).

A slightly higher compaction pressure was needed with the prealloyed powders to give green compacts with densities equivalent to those obtained for elemental powders. This is probably due to the difference in hardness between the two types of powder. However the increase is negligible when compared to the compaction pressures used and on considering the sintering treatment given. On hot forging however it can be seen that higher densities resulted from the prealloyed powder compacts. This again can be attributed to the difference in homogeneity and the increase in recrystallisation that subsequently occurs results in more efficient densification.

Section 4: Summary of Results

The results obtained from mechanical testing, X-ray analyses and metallographic observation show that of the factors considered, those of forging temperature and initial particle size had the greatest effect on the properties of the final forging. It was also apparent that the combinations of the interactions between the variables examined must also be considered. Differing trends and properties were revealed by comparing the results obtained from testing the forgings made from elemental powders and those from prealloyed powders. The trends occurring in one type of powder did not necessarily occur in the other type, in some cases the converse effect took place. This suggests that the use of powders prepared by different methods affected the influence of certain factors on the final properties of the forgings. Amongst the possible reasons for this occurring could be the effect of varying homogeneity and the effect of changing particle shape. It was noted that some of the resulting properties compared quite favourably with those obtained for alloys made by conventional methods.

Mechanical testing of the forgings highlighted the differences and similarities between compacts prepared from the two types of powder. The U.T.S. results indicated that both types reacted similarly when the particle size and temperature of forging were increased; a drop in strength was noted for forgings prepared from elemental powder and those prepared from prealloyed powder. Similar effects were observed when the results of proof stress measurements were examined for both properties and both types of compact the change in strength between the two higher temperatures was much greater than that between the lower temperatures.

Examination of the results obtained for pore size measurement revealed again that the effect of the most significant factor - that of temperature - was similar for both types of powder. Increasing the forging temperature gave a decrease in the effective pore size found in the forgings.

Different effects and properties were noted by using the different types of powder when the results of grain size and recrystallisation measurements were taken however. The effect of forging temperature again proved to be highly significant in both cases for both properties in that it decreased the grain size and increased the extent of recrystallisation as it was raised. In fact it tended to dominate some of the interactions. Initial particle size effects varied however. For prealloyed powder forgings, increasing the initial particle size tended to give an overall increase in grain size and a reduction in the extent of recrystallisation. Whereas forgings made from elemental powders showed the opposite effect, i.e. the grain size decreased and the extent of recrystallisation increased with the increase in original particle size. In general it was considered that the use of prealloyed powder resulted in a greater extent of recrystallisation especially at temperatures near to the recrystallisation temperature.

Contrasting results were again evident on examination of the ductility results. Once more the variable giving the different trends was that or original particle size. The ductility of forgings made from prealloyed powders were significantly improved by increasing the original particle size. On testing compacts made from elemental powder it was found that an improvement in ductility was obtained by reducing the original particle size. Both types of powder displayed the expected improvement in ductility with increase in forging temperature.

Similar effects were also observed on examination of the density of hardness results. An increase in forging temperature tended to give higher densities but lower hardnesses.

Examination of the compacts formed following the hot forging operation revealed that chemically there is effectively no difference between the final forgings made from prealloyed powders and those made from elemental powders. Completely homogenous alloys were formed in both cases whereas sintered compacts showed only partial homogeneity. It was suggested that the mechanical contribution of the hot forging operation has a significant effect on the homogenisation processes. The only differences between the compacts prepared from the different types of powder were mechanical differences and it is considered that these are due to the varying degrees of recrystallisation observed in the final forgings. Differences in homogeneity between the compacts prepared from the different types of powder are probably responsible for the differences in extent of recrystallisation, this was especially so at the highest forging temperatures.

The metallographic observations served to confirm the findings of the statistical analyses and X-ray experimentation carried out on compacts prepared from both elemental and prealloyed powders.

CHAPTER 10: CONCLUSIONS

1. The temperature of forging has the most significant effect on the properties of the final forgings for compacts prepared from both types of powder.

2. Significant effects were also observed by altering the initial particle size of the powder used.

3. The trends observed were not necessarily identical for compacts made from both types of powder.

4. The final forgings were chemically similar in that they were both completely homogenous when compared to alloys prepared from other methods. They were however different mechanically by virtue of the difference in the extent of recrystallisation resulting from the varying degrees of homogeneity prior to forging.

5. The mechanical effect of the hot forging had a significant effect on the homogenisation process.

6. The alloys formed are comparable in properties to those made by conventional methods.

7. No significant effect was noted on the final forging properties by varying the particle size ratio.

Table 1

Coding	Ratio	Elemental Powder Size (μ)	
		Cu	Ni
A	1.5:1	45-53	38-45
B	1:1	38-45	38-45
C	0.75:1	38-45	45-53
D	1.5:1	75-90	63-75
E	1:1	75-90	75-90
F	0.75:1	63-75	75-90

Table 2

Size	Prealloyed Powder Particle Size (μ)
Small	38 - 53
Large	63 - 90

Elemental Powder 773K Forgings

Table 3

	Density %	Grain Size (cm x 10^{-3})	%**Rex** %	Pure Size % $<16 \times 10^{-4}$ mm
A	94.96	12.45	4.4	78.8
B	93.7	10.48	5.6	78.3
C	93.8	11.58	4.8	78.6
D	93.1	12.95	5.0	78.5
E	93.7	8.85	5.0	74.4
F	93.4	11.25	4.6	75.1

Table 4

	Comp Press MNm $^{-2}$	Hardness Hv	Elongation %	0.1 Proof St MNm $^{-2}$	U.T.S MNm $^{-2}$	Red in Area %
A	288.0	109.7	18.2	136.0	309.6	39.12
B	292.4	110.1	15.0	180.3	294.5	31.61
C	288.5	97.1	14.2	127.1	287.5	30.87
D	286.8	102.9	14.5	124.9	293.4	27.1
E	282.3	90.5	13.7	118.5	268.2	26.73
F	289.1	91.6	13.4	120.9	278.1	21.47

Elemental Powders 873K Forgings

Table 5

	Density %	Grain Size (cm x10 $^{-3}$)	rex %	Pore Size % < 16 x 10^{-4} mm
A	94.7	10.69	8.2	78.7
B	95.5	11.19	7.8	77.3
C	96.2	10.38	14.0	80.2
D	95.2	8.18	11.2	74.7
E	96.1	6.81	17.6	76.6
F	95.2	9.09	14.0	75.2

Table 6

	Comp Press MNm $^{-2}$	Hardness Hv	Elongation %	0.1% P Stress MNm^{-2}	U.T.S MNm^{-2}	Red in Area %
A	288.0	111.8	20.90	82.4	305.4	35.93
B	287.6	111.6	19.74	92.4	277.3	35.47
C	290.5	117.0	17.55	104.4	300.5	35.16
D	287.3	111.8	18.01	128.3	283.5	31.46
E	289.5	102.7	17.20	94.6	275.7	32.01
F	277.6	118.5	15.24	97.5	280.6	26.38

Elemental Powder 973K Forgings

Table 7

	Density %	Grain Size (cm x 10^{-3})	% rex %	Pore Size % < 16 x 10^{-4} mm
A	98.4	5.44	18.2	84.9
B	98.6	6.38	19.6	82.8
C	98.4	4.33	24.2	83.3
D	98.4	3.92	28.2	86.1
E	98.3	3.33	30.4	90.1
F	98.1	2.93	33.6	89.1

Table 8

	Comp Press MNm $^{-2}$	Hardness Hv	Elongation %	0.1% P Stress MNm $^{-2}$	U.T.S MNm $^{-2}$	Red in Area %
A	289.8	101.2	27.6	73.1	213.3	65.6
B	292.4	88.8	28.5	82.7	216.3	67.7
C	299.4	91.8	32.8	79.2	213.3	62.6
D	284.0	96.5	26.9	114.4	213.9	60.8
E	290.4	101.6	30.0	89.8	211.8	61.4
F	280.2	88.8	26.7	105.1	221.0	60.9

Prealloyed Powder Forgings

Table 9

Particle Size	Temp K	Density %	Grain Size (cm x10^{-3})	rex %	Pore Size % $<16\text{x}10^{-4}$mm	Comp Press MNm $^{-2}$
Small	773	98.5	10.8	6.9	59.9	302.2
Small	873	99.4	8.5	12.3	82.4	303.4
Small	973	99.9	2.9	84.0	88.8	301.9
Large	773	98.47	12.1	2.2	62.1	312.3
Large	873	99.16	12.4	5.4	84.8	314.7
Large	973	99.47	3.6	71.1	90.3	313.3

Table 10

Particle Size	Temp K	Hardness Hv	Elongation %	Red in Area %	0.1% P Stress MNm $^{-2}$	U.T.S MNm^{-2}
Small	773	103	9.4	31.9	148.5	270.6
Small	873	94.4	16.6	46.9	126.7	271.7
Small	973	72.2	34.4	71.5	89.7	248.4
Large	773	100.3	14.2	50.2	110.0	237.2
Large	873	89.3	17.4	55.2	105.9	242.1
Large	973	79.5	35.2	74.2	69.7	241.3

TABLE 11

Significance Levels %	90	90	95	97.5	99	99.9
Prealloyed Powder $Q_1 = 1, Q_2 = 2$		8.53	18.5	38.5	98.5	998.5
Elemental Powder $Q_1 = 1, Q_2 = 4$		4.54	7.71	12.22	21.2	74.14
Code	6	5	4	3	2	1

TABLE 12

Property	E.M.S. Value
Grain size	16.91
Recrystallisation	4.06
U.T.S.	53.26
Proof stress	164.33
Pore size	3.89
Elongation	3.36
Red in area	5.60
Density	0.36
Hardness	82.02

TABLE 13

U.T.S.

Source	Result	Mean SS	Ratio	Sig Level
1	309.6	-	-	-
a_1	294.5	120.97	2.27	6
a_2	287.5	351.56	6.60	5
b_1	305.4	16258.24	305.25	1
b_2	213.3	5019.72	94.25	1
c_1	293.4	75.24	0	6
a_1 b_1	277.3	247.53	4.65	5
a_1 b_2	216.3	9.0	0	6
a_2 b_1	300.5	59.85	1.12	6
a_2 b_2	213.3	87.78	1.65	6
a_1 c_1	268.2	21.07	0	6
a_2 c_1	278.1	0.9	0	6
b_1 c_1	283.5	258.54	4.85	5
b_2 c_1	213.9	41.6	0	6

TABLE 13.1

A B				
	b_o	b_1	b_2	Mean
a_o	301.5	294.45	213.6	269.85
a_1	281.35	276.5	214.05	257.3
a_2	282.8	290.55	217.15	263.5
Mean	288.55	287.17	214.93	

TABLE 13.2

B C			
	c_o	c_1	Mean
b_o	297.2	279.9	288.6
b_1	294.4	279.9	287.15
b_2	214.3	215.6	216.95
Mean	268.6	258.5	

TABLE 14

PROOF STRESS

Source	Result	Mean SS	Ratio	Sig Level
1	136.0	-	-	-
a_1	180.0	52.08	0	6
a_2	127.0	14.44	0	6
b_1	82.4	5764.08	35.08	2
b_2	73.1	645.16	3.93	6
c_1	124.9	75.24	0	6
a_1 b_1	92.4	12	0	6
a_1 b_2	82.7	0.08	0	6
a_2 b_1	104.4	551.04	3.35	6
a_2 b_2	79.2	266.81	1.62	6
a_1 c_1	118.5	332.85	2.03	6
a_2 c_1	120.9	1038.99	6.32	5
b_1 c_1	128.3	1950.75	11.87	4
b_2 c_1	114.4	209.28	1.27	6

TABLE 14.1

A C			
	c_o	c_1	Mean
a_o	97.16	122.53	109.85
a_1	118.37	100.8	109.59
a_2	103.53	107.83	105.68
Mean	106.35	110.39	

TABLE 14.2

B C			
	c_o	c_1	Mean
b_o	147.67	121.43	134.55
b_1	93.06	106.8	99.93
b_2	78.33	103.1	90.72
Mean	106.35	110.44	

TABLE 15

PORE SIZE

Source	Result	Mean SS	Ratio	Sig Level
1	78.8	-	-	-
a_1	78.3	0	0	6
a_2	78.6	0.49	0	6
b_1	78.7	230.56	59.26	2
b_2	84.9	82.81	21.28	2
c_1	78.5	0.53	0	6
a_1 b_1	77.3	3.12	0	6
a_1 b_2	82.8	1.6	0	6
a_2 b_1	80.2	2.67	0	6
a_2 b_2	83.3	0.02	0	6
a_1 c_1	74.4	0.01	0	6
a_2 c_1	75.1	3.48	0	6
b_1 c_1	74.7	40.33	10.37	4
b_2 c_1	86.1	18.78	4.83	5

TABLE 15.1

BC			
	c_0	c_1	mean
b_0	77.9	76.0	76.95
b_1	78.7	75.5	77.1
b_2	83.7	88.4	86.05
mean	80.1	79.98	

TABLE 16

RECRYSTALLISATION

Source	Result	Mean SS	Ratio	Sig Level
1	4.4	-	-	-
a_1	5.6	52.08	11.94	4
a_2	4.8	1.21	0.2	6
b_1	8.2	1297.92	319.51	1
b_2	18.2	64.0	14.68	3
c_1	5.0	126.93	29.1	2
a_1 b_1	7.8	16.24	3.72	6
a_1 b_2	19.6	1.4	0.32	6
a_2 b_1	14.0	1.82	0.417	6
a_2 b_2	24.2	1.03	0	6
a_1 c_1	5.0	7.36	1.67	6
a_2 c_1	4.6	4.13	0.94	6
b_1 c_1	11.2	77.01	17.67	3
b_2 c_1	28.2	0.87	0	6

TABLE 16.1

BC			
	c_0	c_1	mean
b_0	4.93	4.87	4.9
b_1	8.3	14.27	11.285
b_2	20.67	30.73	25.7
mean	11.3	16.62	

TABLE 17

GRAIN SIZE

Source	Result	Mean SS	Ratio	Sig Level
1	124.5	-	-	-
a_1	104.8	138.04	8.16	4
a_2	115.8	232.56	13.75	3
b_1	106.9	14179.69	838.29	1
b_2	54.4	982.82	58.1	2
c_1	129.5	1357.2	80.24	1
a_1 b_1	111.9	2.76	0	6
a_1 b_2	63.8	143.57	8.49	4
a_2 b_1	103.8	633.45	37.45	2
a_2 b_2	43.3	6.3	0	6
a_1 c_1	88.5	2.17	0	6
a_2 c_1	112.5	374.42	22.14	2
b_1 c_1	81.8	171.01	10.11	4
b_2 c_1	39.2	220.52	13.04	3

TABLE 17.1

A B				
	b_o	b_1	b_2	Mean
a_o	127.0	94.35	46.8	83.38
a_1	96.65	90.0	65.95	84.2
a_2	114.15	97.35	36.3	82.6
Mean	112.6	93.9	49.68	

TABLE 17.2

A C			
	c_o	c_1	Mean
a_o	95.27	83.5	89.38
a_1	93.5	63.23	78.36
a_2	87.6	77.56	82.5
Mean	92.12	74.76	

TABLE 17.3

B C			
	c_o	c_1	Mean
b_o	115.03	110.17	112.60
b_1	107.53	80.27	93.90
b_2	53.83	33.86	43.85
Mean	92.13	74.77	

TABLE 18

ELONGATION

Source	Result	Mean SS	Ratio	Sig Level
1	18.2	-	-	-
a_1	15.0	3.1	0	6
a_2	14.2	0.15	0	6
b_1	20.8	581.02	172.94	1
b_2	27.6	55.01	16.37	3
c_1	14.5	19.64	5.84	5
a_1 b_1	19.7	12.75	3.8	6
a_1 b_2	28.5	5.9	1.76	6
a_2 b_1	17.6	1.45	0	6
a_2 b_2	32.8	0.26	0	6
a_1 c_1	13.7	0.37	0	6
a_2 c_1	13.4	3.93	0	6
b_1 c_1	18.0	0.02	0	6
b_2 c_1	26.9	0.51	0	6

TABLE 19

REDUCTION IN AREA

Source	Result	Mean SS	Ratio	Sig Level
1	39.1	-	-	-
a_1	31.6	43.32	7.74	6
a_2	30.8	4.41	0	6
b_1	35.9	3410.44	609.37	1
b_2	65.6	742.56	132.68	1
c_1	27.1	172.98	30.91	2
a_1 b_1	35.4	15.40	2.75	6
a_1 b_2	67.7	1.08	0	6
a_2 b_1	35.1	4.25	0	6
a_2 b_2	62.1	0.45	0	6
a_1 c_1	26.7	0.16	0	6
a_2 c_1	21.4	4.00	0	6
b_1 c_1	31.4	15.19	2.71	6
b_2 c_1	60.8	0.90	0	6

TABLE 20

DENSITY

Source	Result	Mean SS	Ratio	Sig Level
1	95.0	-	-	-
a_1	93.7	0.01	0	6
a_2	93.8	0.10	0	6
b_1	94.7	63.02	173.98	1
b_2	98.4	1.4	3.87	6
c_1	93.1	0.1	1.2	6
a_1 b_1	95.5	0.04	0	6
a_1 b_2	98.6	0.74	2.03	6
a_2 b_1	96.2	0.04	0	6
a_2 b_2	98.4	0.2	0.2	6
a_1 c_2	93.7	0.01	0	6
a_2 c_1	93.4	0.38	1.05	6
b_1 c_1	95.2	0.24	0	6
b_2 c_1	98.4	0.27	0	6

TABLE 21

HARDNESS

Source	Result	Mean SS	Ratio	Sig Level
1	109.7	-	-	-
a_1	110.1	63.48	0	6
a_2	97.1	1.78	0	6
b_1	111.8	0.44	0	6
b_2	101.2	586.45	7.15	5
c_1	102.9	0.61	0	6
$a_1\ b_1$	111.6	8.61	0	6
$a_1\ b_2$	88.8	166.95	2.04	6
$a_2\ b_1$	117.0	21.47	0	6
$a_2\ b_2$	91.8	96.84	1.18	6
$a_1\ c_1$	90.5	3.0	0	6
$a_2\ c_1$	91.6	1.44	0	6
$b_1\ c_1$	111.8	384.2	4.68	5
$b_2\ c_1$	102.7	9.92	0	6

TABLE 21.1

	BC		
	c_0	c_1	mean
b_0	105.6	95	100.3
b_1	113.5	111	112.25
b_2	93.9	105.9	99.9
mean	104.3	104	

TABLE 22

Significance Code Property	1	2	3	4	5
Grain Size	$b_1\ c_1$	$b_2.a_2\ b_1.a_2\ c_1$	$a_2.b_2\ c_1$	$a_1.a_1\ b_2.b_1\ c_1$	
Recrystallisation	b_1	c_1	$b_2.b_1\ c_1$	a_1	
U.T.S.	$b_1.\ b_2$				$a_2.a_1\ b_1.b_1\ c_1$
Proof Stress		b_1		$b_1\ c_1$	$a_2\ c_1$
Pore Size		$b_1.b_2$		$b_1\ c_1$	$b_2\ c_1$
Elongation	b_1		b_2		c_1
Red in Area	$b_1\ b_2$	c_1			
Density	b_1				
Hardness					$b_2.b_1\ c_1$

TABLE 23

RECRYSTALLISATION

Source	Result	Mean SS	Ratio	Sig Level
1	7.4	-	-	-
b_1	12.0	5270.76	1409.1	1
b_2	84.0	1365.33	336.3	2
c_1	2.4	109.23	26.9	4
$b_1 c_1$	5.4	16.0	3.9	6
$b_2 c_1$	71.0	0.65	0.2	6

TABLE 24

REDUCTION IN AREA

Source	Result	Mean SS	Ratio	Sig Level
1	31.9	-	-	-
b_1	46.9	1011.24	180.6	2
b_2	71.5	46.41	8.3	6
c_1	50.2	143.08	25.6	4
$b_1\ c_1$	55.2	60.8	10.9	5
$b_2\ c_1$	74.2	1.61	0.29	6

TABLE 25

ELONGATION

Source	Result	Mean SS	Ratio	Sig Level
1	9.4	-	-	-
b_1	16.6	529	157.4	2
b_2	34.4	52.97	15.74	5
c_1	14.2	6.83	2.03	6
$b_1\ c_1$	17.4	4.0	1.19	6
$b_2\ c_1$	35.2	1.33	0.40	6

TABLE 26

GRAIN SIZE

Source	Result	Mean SS	Ratio	Sig Level
1	108.0	-	-	-
b_1	85.0	6724.0	397.6	2
b_2	29.0	1282.0	75.8	3
c_1	121.0	580.0	34.3	4
$b_1\ c_1$	124.0	9.0	0.53	6
$b_2\ c_1$	36.0	280.0	16.6	5

TABLE 27

U.T.S.

Source	Result	Mean SS	Ratio	Sig Level
1	270	-	-	-
b_1	271.7	76.56	1.44	6
b_2	248.4	78.54	1.47	6
c_1	237.2	805.04	15.12	5
$b_1\ c_1$	242.1	165.12	3.10	6
$b_2\ c_1$	241.3	31.04	0.58	6

TABLE 28

PROOF STRESS

Source	Result	Mean SS	Ratio	Sig Level
1	148.5	-	-	-
b_1	126.7	2455.20	14.9	5
b_2	89.7	186.44	1.13	6
c_1	110.0	1048.08	6.38	6
$b_1\ c_1$	105.9	4.63	0.03	6
$b_2\ c_1$	69.7	23.8	0.14	6

TABLE 29

PORE SIZE

Source	Result	Mean SS	Ratio	Sig Level
1	59.9	-	-	-
b_1	82.4	815.1	209.5	2
b_2	88.8	92.4	23.8	4
c_1	62.1	6.2	1.6	6
b_1 c_1	84.8	0.12	0.03	6
b_2 c_1	90.3	0.1	0.03	6

TABLE 30

DENSITY

Source	Result	Mean SS	Ratio	Sig Level
1	98.5	-	-	-
b_1	99.4	1.44	4	6
b_2	99.9	0.03	0.08	6
c_1	98.47	0.06	0.16	6
b_1 c_1	99.16	0.04	0.11	6
b_2 c_1	99.47	0.003	0.008	6

TABLE 31

HARDNESS

Source	Result	Mean SS	Ratio	Sig Level
1	103	-	-	-
b_1	94.4	665.6	8.12	6
b_2	72.2	12.8	0.16	6
c_1	100.3	0.04	0.00	6
$b_1\ c_1$	89.3	25.0	0.30	6
$b_2\ c_1$	79.5	12.8	0.15	6

TABLE 32

Treatment	Measured Breadth	% Recrystallisation
*$b_2\ c_1$	13.0	71
b_2	14.5	18.2
$a_1\ b_1$	15.0	19.6
$a_2\ b_2\ c_1$	15.0	30.4
$a_2\ b_2$	15.0	24.2
c_1	15.5	5.0
*c_2	15.5	4.4
*1	15.5	7.4
a_1	15.5	5.6
$b_2\ c_1$	15.5	28.2
$a_2\ b_1$	16.0	14.0
$a_1\ b_1$	16.5	7.8
$b_1\ c_1$	18.0	11.2
1	18.0	4.4
$a_2\ b_1\ c_1$	18.0	14.0
b_1	19.0	8.2
$a_2\ b_2\ c_1$	20.5	33.6
$a_1\ b_1\ c_1$	24.5	17.6
a_1	25.0	4.8
+GPD_1	34.0	-

* Alloys made from prealloyed powders

\+ Sintered only specimen

APPENDIX I

Statistical analysis of results obtained from the testing of compacts made from elemental powders

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U.T.S.



ANALYSIS OF A 3X3X2 FACTORIAL

TREATMT  RESULT     COL.1     COL.2    EFFECT  DIV  MEAN S.S.    RATIO

(1)      309.60    891.60   2417.70   4743.90  18. 1250254.84     0.00
A1       294.50    883.20   2326.20    -38.10  12.     120.97     2.27
A2       287.50    642.90    -27.00    112.50  36.     351.56     6.60
B1       305.40    839.70    -11.10   -441.70  12.   16258.24   305.25
B1A1     277.30    839.80     53.40     44.50   8.     247.53     4.65
B1A2     300.50    646.70     59.10    -37.90  24.      59.85     1.12
B2       213.30    -22.10   -248.70   -425.10  36.    5019.72    94.25
B2A1     216.30     -4.90   -193.00    -14.70  24.       9.00     0.00
B2A2     213.30      0.00     22.10    -79.50  72.      87.78     1.65
C1       293.40    -15.30     22.40    -91.50  18.     465.13     8.73
C1A1     268.20     -2.90    -14.10     15.90  12.      21.07     0.00
C1A2     278.10      7.10    -23.80      5.70  36.       0.90     0.00
C1B1     283.50      8.10   -231.90     55.70  12.     258.54     4.85
C1B1A1   275.70     51.30   -193.20      0.30   8.       0.01     0.00
C1B1A2   280.60     -6.00    -12.30     -9.70  24.       3.92     0.00
C1B2     213.90     35.10     -2.40     38.70  36.      41.60     0.00
C1B2A1   211.80     12.70   -100.50      9.90  24.       4.08     0.00
C1B2A2   221.00     11.30     21.00    121.50  72.     205.03     0.00

    ERROR MEAN SQUARE =          53.26167
16.09.12 FREE *CPO ,37 TRANSFERS

16.09.20 FREE *LPO ,54 TRANSFERS
0.29 :DELETED : 00
16.09.24 0.29 DELETED,CLOCKED 0.00
DISPLAY: EXECUTION ENDED
END OF MACRO
16.09.27-
```

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PROOF STRESS


ANALYSIS OF A 3X3X2 FACTORIAL

TREATMT  RESULT     COL.1      COL.2     EFFECT   DIV  MEAN S.S.    RATIO

(1)      136.00    443.00     957.20    1951.20   18.  211510.08     0.00
A1       180.00    279.20     994.00     -25.00   12.      52.08     0.00
A2       127.00    235.00      19.10     -22.80   36.      14.44     0.00
B1        82.40    364.30     -44.10    -263.00   12.    5764.08    35.08
B1A1      92.40    320.40    -108.10       9.80    8.      12.00     0.00
B1A2     104.40    309.30      85.30     115.00   24.     551.04     3.35
B2        73.10     -9.00    -208.00     152.40   36.     645.16     3.93
B2A1      82.70     22.00     -55.00       1.40   24.       0.08     0.00
B2A2      79.20      6.10      15.10    -138.60   72.     266.81     1.62
C1       124.90     -4.00      -5.30      36.80   18.      75.24     0.00
C1A1     118.50    -30.80      83.90     -63.20   12.     332.85     2.03
C1A2     120.90     -9.30      31.10     193.40   36.    1038.99     6.32
C1B1     128.30    -97.00     119.60     153.00   12.    1950.75    11.87
C1B1A1    94.60      2.00      32.80     -20.40    8.      52.02     0.00
C1B1A2    97.50    -13.10     -46.90     -52.80   24.     116.16     0.00
C1B2     114.40      8.80      48.30     -86.80   36.     209.28     1.27
C1B2A1    89.80     36.60    -114.10      95.20   24.     377.63     0.00
C1B2A2   105.10     39.90     -24.50      89.60   72.     111.50     0.00


       ERROR MEAN SQUARE =        164.32722
12.23.58 FREE *CR0 ,37 TRANSFERS

12.24.09 FREE *LP0 ,54 TRANSFERS
0.25 :DELETED : 00
12.24.10 0.25 DELETED,CLOCKED 0.00
DISPLAY: EXECUTION ENDED
END OF MACRO
12.24.15-
```

```
PORE SIZE


ANALYSIS OF A 3X3X2 FACTORIAL

TREATMT  RESULT     COL.1     COL.2    EFFECT  DIV  MEAN S.S.   RATIO

(1)       78.80    235.70    722.90   1442.70  18.  115632.40    0.00
A1        78.30    236.20    719.80     -0.20  12.       0.00    0.00
A2        78.60    251.00     -0.30      4.20  36.       0.49    0.00
B1        78.70    228.00      0.10     52.60  12.     230.56   59.26
B1A1      77.30    226.50      7.70      5.00   8.       3.12    0.00
B1A2      80.20    265.30     -3.50     -8.00  24.       2.67    0.00
B2        84.90     -0.20     15.30     54.60  36.      82.81   21.28
B2A1      82.80      1.50     37.30     -6.20  24.       1.60    0.00
B2A2      83.30     -1.60     -1.40      1.20  72.       0.02    0.00
C1        78.50     -3.40      6.40     -3.10  18.       0.53    0.00
C1A1      74.40      0.50      1.80      0.40  12.       0.01    0.00
C1A2      75.10      3.00     -9.80    -11.20  36.       3.48    0.00
C1B1      74.70      0.80     14.30     22.00  12.      40.33   10.37
C1B1A1    76.60      4.30     40.30      7.80   8.       7.61    0.00
C1B1A2    75.20      2.60     -4.80    -11.60  24.       5.61    0.00
C1B2      86.10      4.80     -1.40     26.00  36.      18.78    4.83
C1B2A1    90.10     -3.30     -5.20      3.40  24.       0.48    0.00
C1B2A2    89.10     -5.00      6.40     11.60  72.       1.87    0.00

     ERROR MEAN SQUARE =             3.89056
11.26.44 FREE *CR0 ,37 TRANSFERS

11.26.45 FREE *LP0 ,54 TRANSFERS
0.22 :DELETED : 00
11.26.50 0.22 DELETED,CLOCKED 0.00
DISPLAY: EXECUTION ENDED
END OF MACRO
11.27.01←
```

```
RECRYSTALIZATION


ANALYSIS OF A 3X3X2 FACTORIAL

TREATMT  RESULT     COL.1      COL.2     EFFECT  DIV  MEAN S.S.   RATIO

(1)        4.40     14.80     106.80     256.40  18.    3652.28    0.00

A1         5.60     30.00     149.60      20.00  12.      33.33    8.21

A2         4.80     62.00      12.20      -1.60  36.       0.07    0.00

B1         8.20     14.60       7.80     124.80  12.    1297.92  319.51

B1A1       7.80     42.80       7.80      11.40   8.      16.24    4.00

B1A2      14.00     92.20      -9.40       6.60  24.       1.82    0.00

B2        18.20      0.40      47.20      38.00  36.      40.11    9.87

B2A1      19.60      5.80      77.60      -5.80  24.       1.40    0.00

B2A2      24.20      6.00       5.60       8.60  72.       1.03    0.00

C1         5.00     -0.40       5.80      42.80  18.     101.77   25.05

C1A1       5.00      2.80       5.20      -4.40  12.       1.61    0.00

C1A2       4.60      5.40       1.40     -17.20  36.       8.22    2.02

C1B1      11.20     -2.00      16.80      30.40  12.      77.01   18.96

C1B1A1    17.60      6.60      21.20       0.20   8.       0.00    0.00

C1B1A2    14.00      3.20      -5.20      -3.80  24.       0.60    0.00

C1B2      28.20     -0.40      -0.60       4.40  36.       0.54    0.00

C1B2A1    30.40    -10.00     -12.00       4.60  24.       0.88    0.00

C1B2A2    33.60      1.00      20.60      32.60  72.      14.76    0.00


     ERROR MEAN SQUARE =           4.06222
11.16.45 FREE *CR0 ,37 TRANSFERS

11.16.46 FREE *LP0 ,54 TRANSFERS
0.11 :DELETED : 00
11.16.51 0.11 DELETED,CLOCKED 0.00
DISPLAY: EXECUTION ENDED
END OF MACRO
11.17.03-
```

```
GRAIN SIZE


ANALYSIS OF A 3X3X2 FACTORIAL

TREATMT   RESULT     COL.1      COL.2     EFFECT  DIV  MEAN S.S.    RATIO

(1)       124.50    345.10     829.20    1502.10  18.  125350.24     0.00
A1        104.80    322.60     672.90     -40.70  12.     138.04     8.16
A2        115.80    161.50     -22.90      91.50  36.     232.56    13.75
B1        106.90    330.50     -17.80    -412.50  12.   14179.69   838.29
B1A1      111.90    240.80     -12.30       4.70   8.       2.76     0.00
B1A2      103.80    101.60     103.80    -123.30  24.     633.45    37.45
B2         54.40     -8.70    -183.60    -188.10  36.     982.82    58.10
B2A1       63.80     -3.10    -228.90     -58.70  24.     143.57     8.49
B2A2       43.30    -11.10      -2.40      21.30  72.       6.30     0.00
C1        129.50    -17.00       7.10    -156.30  18.    1357.20    80.24
C1A1       88.50      9.10     -60.60       5.10  12.       2.17     0.00
C1A2      112.50     -9.90     -62.70     116.10  36.     374.42    22.14
C1B1       81.80     30.70    -138.60     -45.30  12.     171.01    10.11
C1B1A1     68.10    -13.10     -49.50       9.50   8.      11.28     0.00
C1B1A2     90.90    -29.90     -13.60      -2.10  24.       0.18     0.00
C1B2       39.20     65.00     -45.10      89.10  36.     220.52    13.04
C1B2A1     33.10     36.50      27.00     -31.50  24.      41.34     0.00
C1B2A2     29.30      2.30      -5.70     -32.70  72.      14.85     0.00

     ERROR MEAN SQUARE =          16.91500
10.09.15 FREE *CR0 ,37 TRANSFERS

10.09.17 FREE *LP0 ,54 TRANSFERS
0.18 :DELETED : 00
```

```
ELONGATION


ANALYSIS OF A 3X3X2 FACTORIAL

TREATMT  RESULT    COL.1    COL.2   EFFECT  DIV  MEAN S.S.   RATIO

(1)       18.20    47.40   194.40   370.00  18.    7605.56    0.00

A1        15.00    58.10   175.60    -6.10  12.       3.10    0.00

A2        14.20    88.90    -2.00    -2.30  36.       0.15    0.00

B1        20.80    41.60    -4.10    83.50  12.     581.02  172.94

B1A1      19.70    50.40     4.80    10.10   8.      12.75    3.80

B1A2      17.60    83.60    -7.10    -5.90  24.       1.45    0.00

B2        27.60    -4.00    41.50    44.50  36.      55.01   16.37

B2A1      28.50    -3.20    42.00    11.90  24.       5.90    1.76

B2A2      32.80     5.20     9.20     4.30  72.       0.26    0.00

C1        14.50    -1.10     0.90   -18.80  18.      19.64    5.84

15.57.29 BROADCAST: JOB DRUN        COMPLETED
C1A1      13.70    -2.80     1.00    -2.10  12.       0.37    0.00

C1A2      13.40    -0.20    -6.90   -11.90  36.       3.93    1.17

C1B1      18.00     2.40    20.10     0.50  12.       0.02    0.00

C1B1A1.   17.20    -1.00    24.40    -8.30   8.       8.61    0.00

C1B1A2    15.20     3.40     7.60    -7.90  24.       2.60    0.00

C1B2      26.90     0.50     4.30     4.30  36.       0.51    0.00

C1B2A1    30.00    -1.20     7.80    -3.30  24.       0.45    0.00

C1B2A2    26.70    -6.40    -3.50   -11.30  72.       1.77    0.00


    ERROR MEAN SQUARE =          3.35972
15.58.53 FREE *CR0 ,37 TRANSFERS

15.58.55 FREE *LP0 ,54 TRANSFERS
0.18 :DELETED : 00
15.59.00 0.18 DELETED,CLOCKED 0.00
DISPLAY: EXECUTION ENDED
```

```
REDUCTION IN AREA



ANALYSIS OF A 3X3X2 FACTORIAL

TREATMT  RESULT    COL.1     COL.2     EFFECT   DIV  MEAN S.S.   RATIO

(1)      39.10    101.50    403.80    751.80   18.   31400.18    0.00
A1       31.60    106.40    348.00    -22.80   12.      43.32    7.74
A2       30.80    195.90    -12.10    -12.60   36.       4.41    0.00
B1       35.90     75.20    -10.70    202.30   12.    3410.44  609.37
B1A1     35.40     89.70     -0.30     11.10    8.      15.40    2.75
B1A2     35.10    183.10    -12.30    -10.10   24.       4.25    0.00
B2       65.60     -8.30     94.40    163.50   36.     742.56  132.68
B2A1     67.70     -0.80    107.90     -5.10   24.       1.08    0.00
B2A2     62.60     -3.00      5.30      5.70   72.       0.45    0.00
C1       27.10     -5.70      5.80    -55.80   18.     172.98   30.91
C1A1     26.70     -5.10    -13.90      1.40   12.       0.16    0.00
C1A2     21.40      0.10      3.80    -12.00   36.       4.00    0.00
C1B1     31.40      6.70     84.60     13.50   12.      15.19    2.71
C1B1A1   32.00      0.20     78.90      0.50    8.       0.03    0.00
C1B1A2   26.30     -7.20     -9.70     17.70   24.      13.05    0.00
C1B2     60.80     -4.90      4.60     -5.70   36.       0.90    0.00
C1B2A1   61.40     -6.30     -0.90     14.30   24.       8.52    0.00
C1B2A2   60.90     -1.10      6.60      7.50   72.       0.78    0.00

     ERROR MEAN SQUARE =              5.59667
16.18.27 FREE *CR0 ,37 TRANSFERS

16.18.36 FREE *LP0 ,54 TRANSFERS
0.40 :DELETED : 00
16.18.38 0.40 DELETED,CLOCKED 0.00
DISPLAY: EXECUTION ENDED
```

```
DENSITY


ANALYSIS OF A 3X3X2 FACTORIAL

TREATMT  RESULT     COL.1     COL.2    EFFECT  DIV  MEAN S.S.    RATIO

(1)       95.00    282.50    864.30   1725.80  18.  165465.87     0.00

A1        93.70    286.40    861.50      0.30  12.       0.01     0.00

A2        93.80    295.40      0.30     -1.90  36.       0.10     0.00

B1        94.70    280.20      0.00     27.50  12.      63.02   173.98

B1A1      95.50    286.50      0.90      0.60   8.       0.04     0.00

B1A2      96.20    294.80     -2.80     -1.00  24.       0.04     0.00

B2        98.40     -1.20     12.90      7.10  36.       1.40     3.87

B2A1      98.60      1.50     14.60     -4.20  24.       0.74     2.03

B2A2      98.40      0.00      1.20      3.80  72.       0.20     0.00

C1        93.10      0.30     -0.60     -2.80  18.       0.44     1.20

C1A1      93.70      0.00     -1.80     -0.30  12.       0.01     0.00

C1A2      93.40     -0.30      0.80     -3.70  36.       0.38     1.05

C1B1      95.20      1.40      5.10      1.70  12.       0.24     0.00

C1B1A1    96.10     -0.10      2.00     -1.80   8.       0.41     0.00

C1B1A2    95.20     -0.40     -4.20      2.60  24.       0.28     0.00

C1B2      98.40     -0.90      0.00     -3.10  36.       0.27     0.00

C1B2A1    98.30     -1.80      1.20      4.20  24.       0.74     0.00

C1B2A2    98.10     -0.10      2.60      1.40  72.       0.03     0.00


     ERROR MEAN SQUARE =          0.36222
10.01.35 FREE *CR0 ,37 TRANSFERS

10.01.37 FREE *LP0 ,54 TRANSFERS
0.09 :DELETED : 00
10.01.42 0.09 DELETED,CLOCKED 0.00
DISPLAY: EXECUTION ENDED
END OF MACRO
10.01.53-
```

```
HARDNESS


ANALYSIS OF A 3X3X2 FACTOPIAL

TPEATMT   FESULT     COL.1      COL.2     EFFECT   DIV   MEAN S.S.     PATIO

(1)       109.70    316.90     939.10   1874.90    18.   195291.67     0.00
A1        110.10    340.40     935.80    -27.60    12.       63.48     0.00
A2         97.10    281.80     -16.80      8.00    36.        1.78     0.00
B1        111.80    285.00     -10.80     -2.30    12.        0.44     0.00
B1A1      111.60    333.00       7.60      8.30     8.        8.61     0.00
B1A2      117.00    317.80       0.40    -22.70    24.       21.47     0.00
B2        101.20    -12.60     -35.10   -145.30    36.      586.45     7.15
B2A1       88.80      5.20      32.80    -63.30    24.      166.95     2.04
B2A2       91.80     -9.40       3.20    -83.50    72.       96.84     1.18
C1        102.90    -11.30       5.10     -3.30    18.        0.61     0.00
C1A1       90.50      6.70      28.80      6.00    12.        3.00     0.00
C1A2       91.60     -6.20     -51.50     -7.20    36.        1.44     0.00
C1B1      111.80    -13.40     -82.10     67.90    12.      384.20     4.68
C1B1A1    102.70      5.60     -63.20      1.90     8.        0.45     0.00
C1B1A2    118.50     15.40     -32.40    -80.30    24.      268.67     0.00
C1B2      102.70     13.50     -30.90     18.90    36.        9.92     0.00
C1B2A1    118.60     24.90      -9.20      1.50    24.        0.09     0.00
C1B2A2     96.50    -38.00     -74.30    -65.10    72.       58.86     0.00

     EPPOR MEAN SQUARE =           82.01917
15.52.01 FREE *CR0 ,37 TRANSFEPS

15.52.04 FPEE *LP0 ,54 TPANSFEPS
0.09 :DELETED : 00
15.52.07 0.09 DELETED,CLOCKED 0.00
```

APPENDIX II

Statistical analysis of results obtained from the testing of compacts made from prealloyed powders

U.T.S.

Source	Result	Col 1	Col 2	Div	Mean SS	Ratio
1	270	790.1	1510.7	-	-	-
b_1	271.7	720.6	-17.5	4	76.56	1.44
b_2	248.4	-21.6	-30.7	12	78.54	1.47
c_1	237.2	4.7	-69.5	6	805.04	15.12
b_1 c_1	242.1	-25	25.7	4	165.12	3.10
b_2 c_1	241.3	-5.7	19.3	12	31.04	0.58

RECRYSTALLISATION

Source	Result	Col 1	Col 2	Div	Mean SS	Ratio
1	7.4	104.4	183.2	-	-	-
b_1	12.0	78.8	145.2	4	5270.76	1409.1
b_2	84.0	76.6	128	12	1365.33	336.3
c_1	2.4	68.8	-25.6	6	109.23	26.9
b_1 c_1	5.4	65.4	-8.0	4	16	3.9
b_2 c_1	71.0	62.6	-2.8	12	0.65	0.2

ELONGATION

Source	Result	Col 1	Col 2	Div	Mean SS	Ratio
1	9.4	60.4	127.2	-	-	-
b_1	16.6	66.8	46	4	529	157.4
b_2	34.4	25	25.2	12	52.92	15.74
c_1	14.2	21	6.4	6	6.83	2.03
b_1 c_1	17.4	10.6	-4.0	4	4.0	1.19
b_2 c_1	35.2	14.6	4.0	12	1.33	0.40

PROOF STRESS

Source	Result	Col 1	Col 2	Div	Mean SS	Ratio
1	148.5	364.9	650.1	-	-	-
b_1	126.7	285.6	-99.1	4	2455.2	14.9
b_2	89.7	-58.8	-47.3	12	186.44	1.13
c_1	110	-40.3	-79.3	6	1048.08	6.38
b_1 c_1	105.9	-15.2	18.5	4	4.63	0.03
b_2 c_1	69.7	-32.1	-16.9	12	23.8	0.14

REDUCTION IN AREA

Source	Result	Col 1	Col 2	Div	Mean SS	Ratio
1	31.9	150.3	329.9	-	-	-
b_1	46.9	179.6	63.6	4	1011.24	180.6
b_2	71.5	39.6	23.6	12	46.41	8.3
c_1	50.2	24.0	29.3	6	143.08	25.6
$b_1 c_1$	55.2	9.6	-15.6	4	60.8	10.9
$b_2 c_1$	74.2	14.0	4.4	12	1.61	0.29

GRAIN SIZE

Source	Result	Col 1	Col 2	Div	Mean SS	Ratio
1	108.0	222.0	503.0	-	-	-
b_1	85.0	281.0	-164.0	4	6724.0	397.6
b_2	29.0	-79.0	-124.0	12	1282.0	75.8
c_1	121.0	-85.0	59.0	6	580.0	34.3
$b_1 c_1$	124.0	-33.0	-6.0	4	9	0.53
$b_2 c_1$	36.0	-91.0	58	12	280	16.6

PORE SIZE

Source	Result	Col 1	Col 2	Div	Mean SS	Ratio
1	59.9	231.1	468.3	-	-	-
b_1	82.4	237.2	57.1	4	815.1	209.5
b_2	88.8	28.9	-33.3	12	92.4	23.8
c_1	62.1	28.2	6.1	6	6.2	1.6
$b_1 c_1$	84.8	-16.1	-0.7	4	0.12	0.03
$b_2 c_1$	90.3	-17.2	-1.1	12	0.1	0.03

DENSITY

Source	Result	Col 1	Col 2	Div	Mean SS	Ratio
1	98.5	297.8	595	-	-	-
b_1	99.4	297.2	2.4	4	1.44	4
b_2	99.9	1.4	-0.6	12	0.03	0.08
c_1	98.47	1.0	-0.6	6	0.06	0.16
$b_1 c_1$	99.16	-0.4	-0.2	4	0.04	0.11
$b_2 c_1$	99.47	-0.2	0.2	12	0.003	0.008

HARDNESS

Source	Result	Col 1	Col 2	Div	Mean SS	Ratio
1	103	296.6	538.7	-	-	-
b_1	94.4	269.1	-51.6	4	665.6	8.12
b_2	72.2	-30.8	-12.4	12	12.8	0.16
c_1	100.3	-20.8	-0.5	6	0.04	0.00
b_1 c_1	89.3	-13.6	-10.0	4	25.0	0.30
b_2 c_1	79.5	1.2	-12.4	12	12.8	0.15

APPENDIX III

X-ray traces obtained of intensity versus Bragg angle 2 θ following the testing of compacts with the following treatment combinations:-

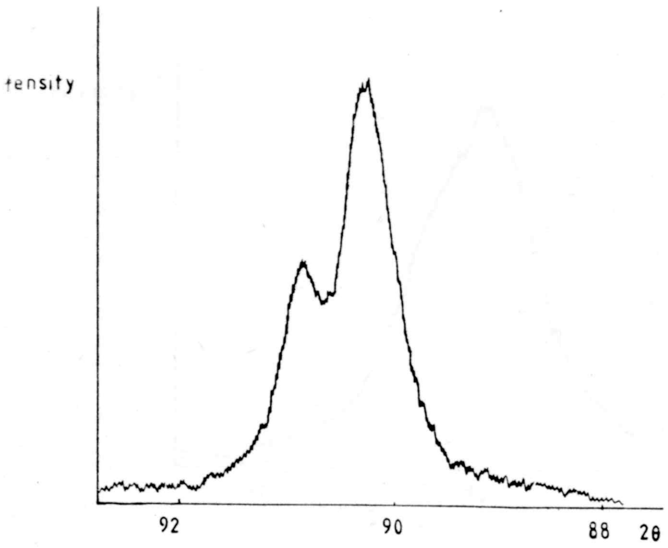
1) Prealloyed powder ; high temperature, high particle size

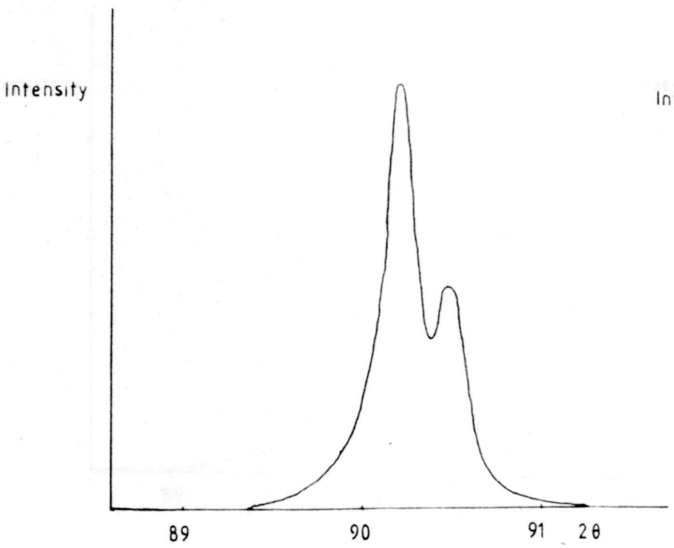
2) Elemental powder ; medium temperature, high particle size, low particle size ratio

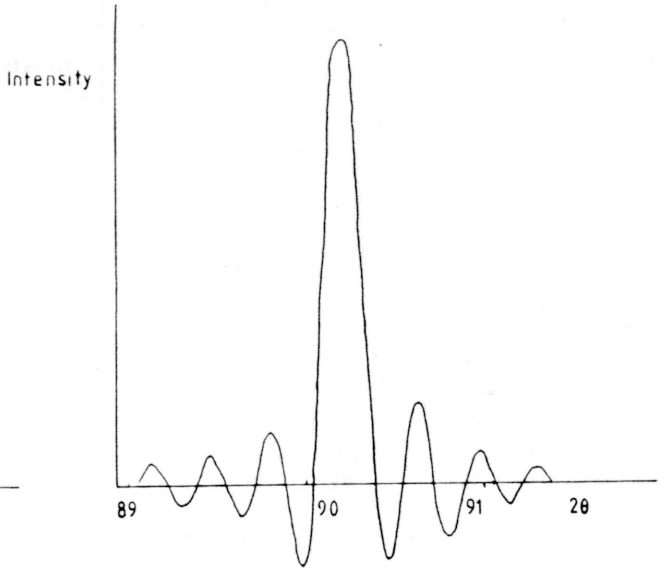
3) Elemental powder ; sintered only

APPENDIX III

(1)

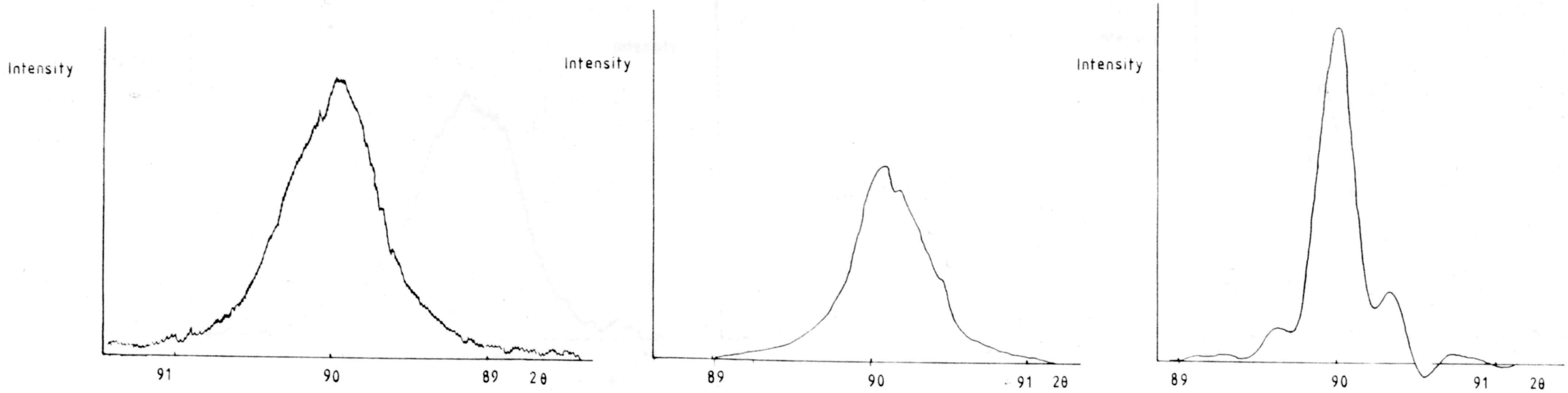






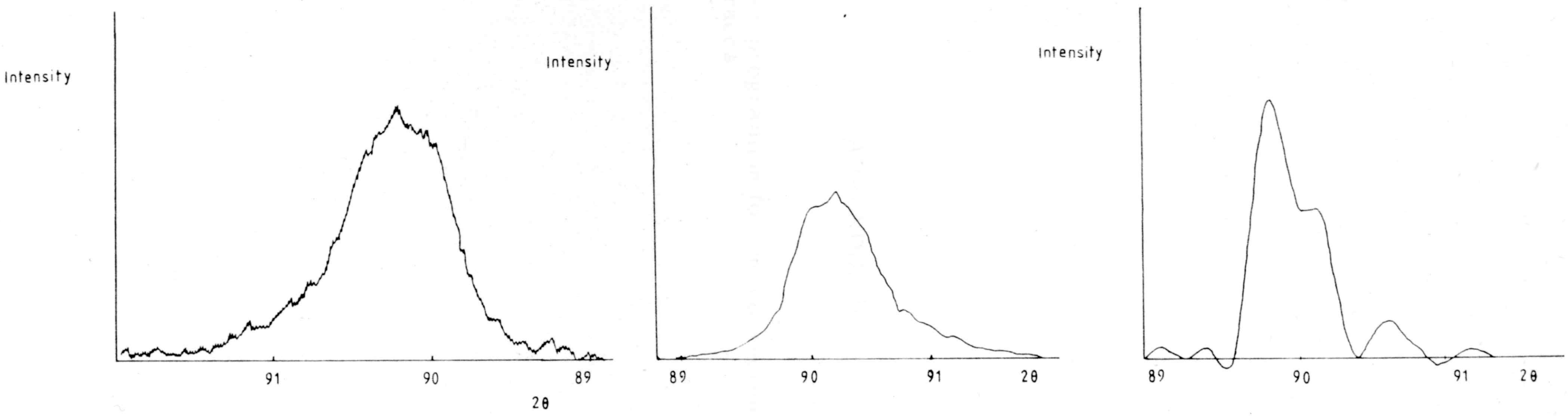
APPENDIX III

(2)



APPENDIX III

(3)



APPENDIX IV

Computer programme for the correction of the x-ray traces

```
      PROGRAM (REFO)
      COMPACT DATA
      INPUT 1=  CR0
      OUTPUT 2= LP0
      OUTPUT 3= CP0
      END
      MASTER REFOURIER
      DIMENSION TITLE(6)
      DIMENSION GR(100),GI(100),HR(100),HI(100),FR(100),FI(100)
      DIMENSION F(113),Y(113)
      DIMENSION XD(113),HD(113),GD(113),X(113),H(113),G(113)
      DIMENSION FD(113)

C     DATA INITIALIZATION

      XD(1)= 88.5
      A=3.5/112.
      DO 10 I=2,113
   10 XD(I)= XD(I-1)+ A
      READ(1,8)NN
      WRITE(3,8)NN
    8 FORMAT(I2)
      DO 200 L=1,7
      M=L*16
      MM=(L-1)*16+1
      READ(1,2)(GD(I),I=MM,M)
  200 CONTINUE
      READ(1,2)GD(113)
      DO 9000 IJ=1,NN
      READ(1,9)TITLE
      WRITE(3,9)TITLE
    9 FORMAT(6A6)
      DO 201 L=1,7
      M=L*16
      MM=(L-1)*16+1
  201 READ(1,2)(HD(I),I=MM,M)
      READ(1,2)HD(113)
    2 FORMAT(16F5.0)
      NC=57
      N=113

C     SCALE H TO SAME AREA AS G

      CALL INTEGRATE(A,HD,HIN,N)
      HA=HIN
      CALL INTEGRATE(A,GD,HIN,N)
      GA=HIN
      FACT=GA/HA
      DO 1020 I=1,N
      H(I)=HD(I)*FACT
      G(I)=GD(I)
 1020 X(I)=XD(I)-XD(NC)
```

```
      PROGRAM (REFO)
      COMPACT DATA
      INPUT 1=  CR0
      OUTPUT 2= LP0
      OUTPUT 3= CP0
      END
      MASTER REFOURIER
      DIMENSION TITLE(6)
      DIMENSION GR(100),GI(100),HR(100),HI(100),FR(100),FI(100)
      DIMENSION F(113),Y(113)
      DIMENSION XD(113),HD(113),GD(113),X(113),H(113),G(113)
      DIMENSION FD(113)

C     DATA INITIALIZATION

      XD(1)= 88.5
      A=3.5/112.
      DO 10 I=2,113
   10 XD(I)= XD(I-1)+ A
      READ(1,8)NN
      WRITE(3,8)NN
    8 FORMAT(I2)
      DO 200 L=1,7
      M=L*16
      MM=(L-1)*16+1
      READ(1,2)(GD(I),I=MM,M)
  200 CONTINUE
      READ(1,2)GD(113)
      DO 9000 IJ=1,NN
      READ(1,9)TITLE
      WRITE(3,9)TITLE
    9 FORMAT(6A6)
      DO 201 L=1,7
      M=L*16
      MM=(L-1)*16+1
  201 READ(1,2)(HD(I),I=MM,M)
      READ(1,2)HD(113)
    2 FORMAT(16F5.0)
      NC=57
      N=113

C     SCALE H TO SAME AREA AS G

      CALL INTEGRATE(A,HD,HIN,N)
      HA=HIN
      CALL INTEGRATE(A,GD,HIN,N)
      GA=HIN
      FACT=GA/HA
      DO 1020 I=1,N
      H(I)=HD(I)*FACT
      G(I)=GD(I)
 1020 X(I)=XD(I)-XD(NC)
```

```
      PROGRAM (REFO)
      COMPACT DATA
      INPUT 1= CR0
      OUTPUT 2= LP0
      OUTPUT 3= CP0
      END
      MASTER REFOURIER
      DIMENSION TITLE(6)
      DIMENSION GR(100),GI(100),HR(100),HI(100),FR(100),FI(100)
      DIMENSION F(113),Y(113)
      DIMENSION XD(113),HD(113),GD(113),X(113),H(113),G(113)
      DIMENSION FD(113)

C     DATA INITIALIZATION

      XD(1)= 88.5
      A=3.5/112.
      DO 10 I=2,113
   10 XD(I)= XD(I-1)+ A
      READ(1,8)NN
      WRITE(3,8)NN
    8 FORMAT(I2)
      DO 200 L=1,7
      M=L*16
      MM=(L-1)*16+1
      READ(1,2)(GD(I),I=MM,M)
  200 CONTINUE
      READ(1,2)GD(113)
      DO 9000 IJ=1,NN
      READ(1,9)TITLE
      WRITE(3,9)TITLE
    9 FORMAT(6A6)
      DO 201 L=1,7
      M=L*16
      MM=(L-1)*16+1
  201 READ(1,2)(HD(I),I=MM,M)
      READ(1,2)HD(113)
    2 FORMAT(16F5.0)
      NC=57
      N=113

C     SCALE H TO SAME AREA AS G

      CALL INTEGRATE(A,HD,HIN,N)
      HA=HIN
      CALL INTEGRATE(A,GD,HIN,N)
      GA=HIN
      FACT=GA/HA
      DO 1020 I=1,N
      H(I)=HD(I)*FACT
      G(I)=GD(I)
 1020 X(I)=XD(I)-XD(NC)
```

```
C     FOURIER COEFFICIENTS FOR K=0

      GI0= 0.
      HI0= 0.
      CALL INTEGRATE(A,G,HIN,N)
      GR0= HIN/3.5
      CALL INTEGRATE(A,H,HIN,N)
      HR0= HIN/3.5

C     FOURIER COEFFICIENTS FOR K=1,10

      DO 50 K=1,10
      RK=K
      DO 60  I=1,N
   60 Y(I)= G(I)*COS(1.7952*RK*X(I))
      CALL INTEGRATE(A,Y,HIN,N)
      GR(K)=HIN/3.5
      DO 70 I=1,N
   70 Y(I)= G(I)*SIN(1.7952*RK*X(I))
      CALL INTEGRATE(A,Y,HIN,N)
      GI(K)=HIN/3.5
      DO 80 I=1,N
   80 Y(I)= H(I)*COS(1.7952*RK*X(I))
      CALL INTEGRATE(A,Y,HIN,N)
      HR(K)=HIN/3.5
      DO 90 I=1,N
   90 Y(I)= H(I)*SIN(1.7952*RK*X(I))
      CALL INTEGRATE(A,Y,HIN,N)
   50 HI(K)= HIN/3.5

C     FOURIER COEFFICIENTS OF F

      FI0=0.
      FR0= HR0/GR0
      DO 100 K=1,10
      FAC= GR(K)**2+GI(K)**2
      FR(K)=(HR(K)*GR(K)+HI(K)*GI(K))/FAC
  100 FI(K)=(HI(K)*GR(K)-HR(K)*GI(K))/FAC

C     CALCULATE VALUES OF F

      DO 120 I=1,N
      S1=0.
      S2=0.
      DO 110 K=1,10
      RK=K
      XX=1.7952*RK*X(I)
      S1=S1+ FR(K)*COS(XX)
  110 S2=S2+ FI(K)*SIN(XX)
  120 F(I)= FR0 + 2.*(S1+S2)
```

```
C     SCALE F TO SAME AREA AS G

      CALL INTEGRATE(A,F,HIN,N)
      FACT=GA/HIN
      DO 130 I=1,N
  130 FD(I)=F(I)*FACT

C      OUTPUT DATA

      WRITE(2,131)TITLE
  131 FORMAT(//////1X,6A6)
      WRITE(2,3)
    3 FORMAT(//1X,'FOURIER COEFICIENTS')
      WRITE(2,4)
    4 FORMAT(/1X,'  K           GR           GI           HR           HI
     1       FR          FI'/)
      WRITE(2,13)GR0,GI0,HR0,HI0,FR0,FI0
   13 FORMAT(1X,E15.4,5E12.4)
      DO 150 K=1,10
  150 WRITE(2,5)K,GR(K),GI(K),HR(K),HI(K),FR(K),FI(K)
    5 FORMAT(1X,I3,6E12.4)
      WRITE(2,6)
    6 FORMAT(///1X,'        XD        X     GD      G     HD      H
     1 H           HD')
      DO 160 I=1,N
  160 WRITE(2,7)XD(I),X(I),GD(I),G(I),HD(I),H(I),F(I),FD(I)
    7 FORMAT(1X,2F9.5,4F6.1,2F13.5)

C     OUTPUT DATA FOR PLOTTING


       DO 5050 I=1,113
 5050 WRITE(3,5051)XD(I),G(I),H(I),FD(I)
 5051 FORMAT(4E15.5)
 9000 CONTINUE
      STOP
      END


      SUBROUTINE INTEGRATE(A,Y,HIN,N)
      DIMENSION Y(113)
      S1=0.
      S2=0.
      DO 10 I=2,N-1,2
   10 S1=S1+Y(I)
      DO 11 I=3,N-2,2
   11 S2=S2+Y(I)
      HIN= A/3.*(Y(1)+Y(N)+4.*S1+2.*S2)
      RETURN
      END
      FINISH
```

```
      PROGRAM(REPO)
      COMPACT DATA
      INPUT 1=CR0
      END
      MASTER REPLOTX
      DIMENSION X(113),G(113),H(113),F(113)
      DIMENSION ITITLE(40)
      DO 1 I=1,113
    1 READ(1,2)X(I),G(I),H(I),F(I)
    2 FORMAT(4E15.5)
      CALL CILN
      CALL MOVTO2(100.,240.)
      CALL CHAASC(103)
      CALL MOVTO2(400.,240.)
      CALL CHAASC(104)
      CALL MOVTO2(700.,240.)
      CALL CHAASC(102)
      CALL SHIFT2(50.,50.)
      CALL PENSEL(1,0.2,2)
      CALL AXIPOS(1,0.,0.,250.,1)
      CALL AXIPOS(1,0.,0.,200.,2)
      CALL AXISCA(3,4,88.,92.,1)
      CALL AXISCA(3,4,0.,200.,2)
      CALL AXIDRA(2,1,1)
      CALL AXIDRA(-2,1,2)
      CALL PENSEL(2,0.2,2)
      CALL GRACUR(X,G,113)
      CALL PENSEL(1,0.2,2)
      CALL SHIFT2(300.,0.)
      CALL AXIPOS(1,0.,0.,250.,1)
      CALL AXIPOS(1,0.,0.,200.,2)
      CALL AXISCA(3,4,88.,92.,1)
      CALL AXISCA(3,4,0.,200.,2)
      CALL AXIDRA(2,1,1)
      CALL AXIDRA(-2,1,2)
      CALL PENSEL(2,0.2,2)
      CALL GRACUR(X,H,113)
      CALL PENSEL(1,0.2,2)
      CALL SHIFT2(300.,0.)
      S=0.
      DO 10 I=1,113
   10 IF(F(I).GT.S)S=F(I)
      IF(S.LT.200)FIN=200
      IF(S.LT.400..AND.S.GT.200.)FIN=400.
      IF(S.LT.800..AND.S.GT.400.)FIN=800.
      CALL AXIPOS(1,0.,0.,250.,1)
      CALL AXIPOS(1,0.,0.,200.,2)
      CALL AXISCA(3,4,88.,92.,1)
      CALL AXISCA(3,4,0.,FIN,2)
      CALL AXIDRA(2,1,1)
      CALL AXIDRA(-2,1,2)
      CALL PENSEL(2,0.2,2)
      CALL GRACUR(X,F,113)
      CALL DEVEND
      STOP
      END
      FINISH
```

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