Pellet forging of iron.

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PELLET FORGING OF IRON

by

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D e d i c a t e d

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C O N T E N T S

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SYNOPSIS

A study has been made of a new particulate forging process in which pellets are used instead of conventional powder. The experimental procedure was first to produce iron oxide pellets prepared from a mixture of iron oxide superconcentrate powder and an organic binder. After drying, the oxide pellets were reduced and sintered in hydrogen to produce porous sponge iron pellets. Preforms were made from these pellets and subsequently hot forged in a closed die at 1100°C to produce forged discs. The discs were called 'sponge iron pellet forgings'.

One of the purposes of the investigation was to determine whether the new route gave a product which had mechanical properties at least equal to the equivalent powder forged product even though it was made from a potentially cheap and relatively impure raw material. Thus, forgings were also made from pelletised iron powder and iron powder, and the results compared. Mechanical properties appear to be better or similar to that of iron powder forgings.

The effects of process variables, such as pellet size, preform density, type of deformation and annealing treatment, on the final properties were studied. Among these, preform density (varying between 50-95% theoretical) and annealing treatment (1 hour at 700°C in H_2) had significant effects on the final properties.

Microscopically the product is characterised by a fine grain size and a large number of uniformly distributed

fine inclusions. The inclusions derived from residual impurities in the concentrate, most of which were originally in the magnetite lattice. The number, size and size distribution of the inclusions are determined. The inclusions impart a substantial grain-refinement strengthening to the iron matrix during annealing.

CHAPTER 1

INTRODUCTION

The development of alternatives to current methods of metal production and fabrication has created much interest throughout the world. Discounting casting, this is especially apparent in the field of steel drop forging, where good mechanical properties are essential. Successful developments which have emerged are: Powder Metallurgy, Powder Forging and Spray Forging.

Powder metallurgy methods involve the manufacture of a compact with a subsequent sintering operation. The product is porous to a greater or lesser degree depending on the application. Powder forging which involves the hot-forging of a sintered preform between closed dies. The product is nearly fully dense with improved properties. A new developed "Spray Forging Process" is a possible route to a final product with good properties but is still in the early stages of development.

In recent years metal powder and particle technology, including powder metallurgy, has extended beyond conventional powder metallurgy to incorporate other processes involving metal powder in the solid or liquid state. The various terms used in this thesis for processes associated with powder metallurgy are all familiar ones, but it is essential to define them clearly to avoid confusion.

Particle technology is sub-divided into a sub-group called powder, which consists of a solid material in a particular form. The subgroup of powder can be further

divided into metal powder and non-metal powder.[1,2] In this thesis the following convention with respect to forging is therefore used:

(i) The 'particle technology' route for forging is a general term used for any process involving the production or use of particles which may be metal or non-metal, either in the solid or liquid state, and in the hot or cold condition.

(ii) The 'powder technology' route for forging is a sub-group of the general area of the particle technology route, where metal or non-metal powder, either in the hot or cold condition, is produced or used.

(iii) The 'powder metallurgy' route for forging is a sub-group of the powder technology route, where solid metal powder in the hot or cold condition is produced or used.

Since all forgings are derived from ore or ore concentrate, it is logical to envisage these routes starting from ore or ore concentrates. These routes could be operated on a non-integrated, semi-integrated, or fully integrated basis from the ore or ore concentrate to the finished product. Fig. 1.1. shows the classification of these routes for forging from ore or ore concentrate.

The work described in this thesis seeks to establish a new powder technology route to make metal forgings starting from a non-metal powder (ore concentrate). Broadly, high purity sponge iron pellets are produced from iron ore super-

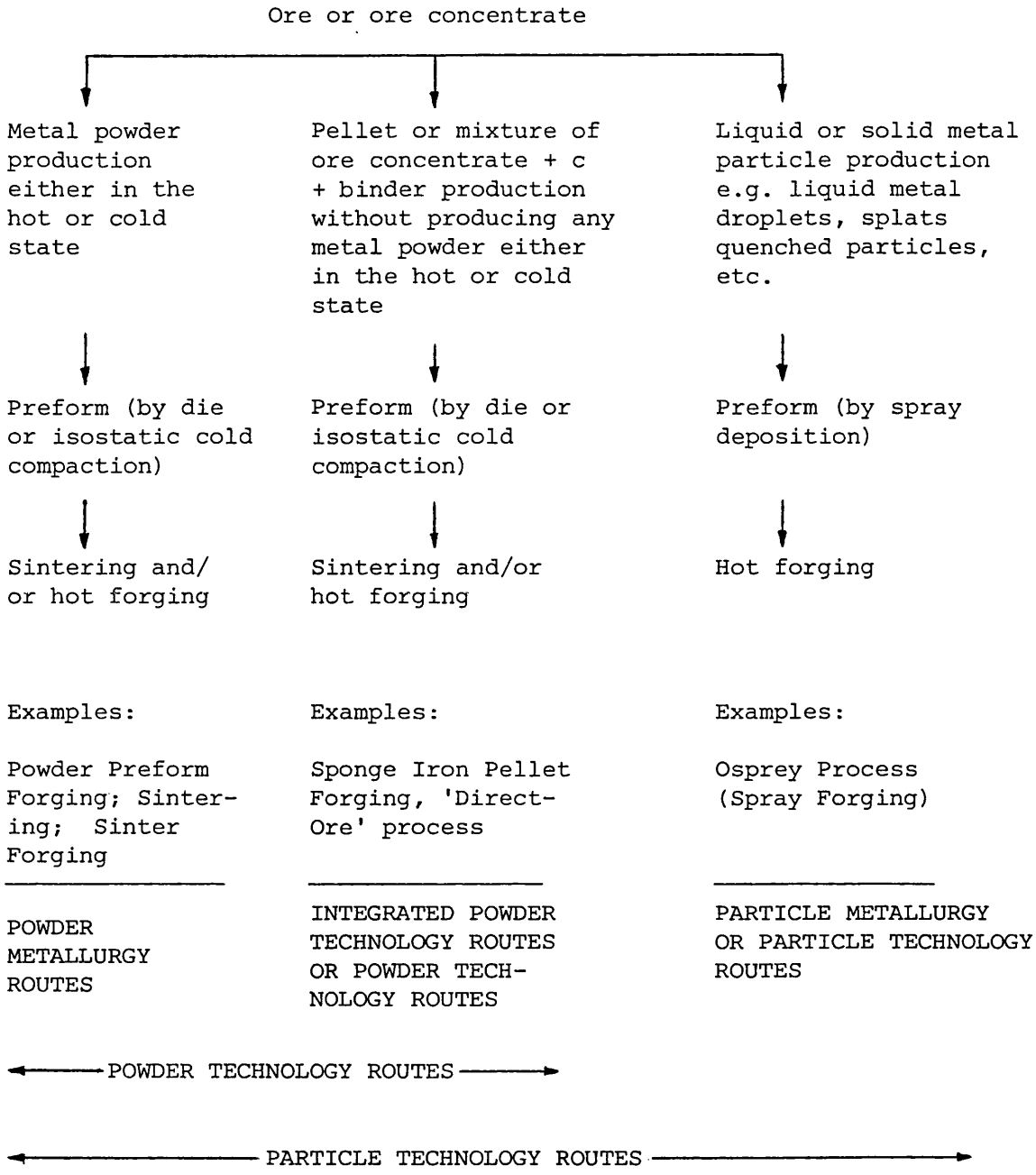


Fig. 1.1. Classification of the Particle Technology Routes For Forging From Ore or Ore Concentrates

concentrates by hydrogen reduction, cold forged to give preforms of discrete shape and density and finally the preforms are hot-forged and heat-treated. The process has been called Sponge Iron Pellet Forging.

To set the scene, a review of the existing routes to steel forgings has been made and this forms the early chapters of this thesis.

CHAPTER 2

REVIEW OF EXISTING ROUTES TO STEEL FORGINGS

2.1. THE CONVENTIONAL ROUTE

Forged parts are produced from iron ore by the 'iron-making-steelmaking-ingot casting-metal working (i.e. forging)' route which will be referred to as the conventional route throughout the present study. In recent years, some improvements have been introduced to the conventional route such as the basic oxygen process and continuous casting. A typical layout of the conventional ironmaking steel processing operational sequence in an integrated steel plant is shown in Fig. 2.1. However, the conventional route has been criticised in many ways. The reasons are generally associated with the dramatic increase in the cost of energy, capital and the following:

(i) The main fuel for ironmaking in the blast furnace is coke, which is an expensive fuel and is a commodity in very short supply throughout the world.[3]

(ii) The major part of the heat supplied (about 45-60%) by the coke is not used in the blast furnace and leaves in the form of unused CO in the top gas.[4] However this outcoming gas is utilised for various heating purposes in the subsequent operations of steelmaking, but this can possibly be obtained from a relatively low grade fuel.

(iii) The coke not only reduces the iron oxide into iron but also saturates the iron with carbon to the extent of 3-4%. This carbon loss, which has also to be

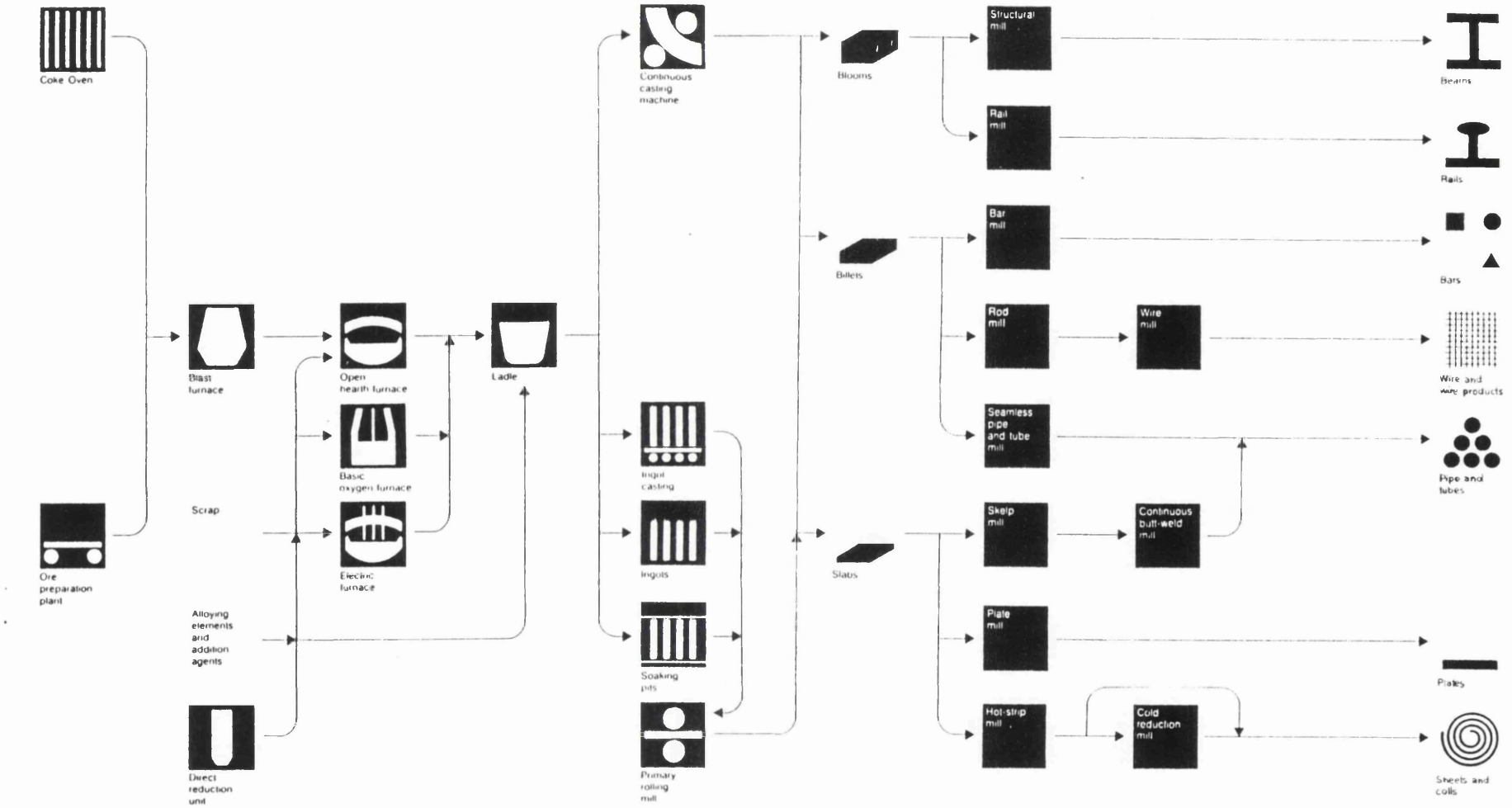


Fig. 2.1. The conventional ironmaking steel processing sequence[10]

removed along with some other metalloids to make steel, constitutes about 8% of the total energy requirements.[5]

(iv) Many individual processes involved in the conventional route, some of which operate at high and some at relatively low temperature, require enormous energy because of frequent heating. The thermal efficiency of the total process is low. About 20% of the total cost of production is represented by energy costs.[6]

(v) Impurities from the original iron ore are removed at a fairly late stage in processing. Some are removed in the slag produced in the blast furnace, some during steelmaking, and some residuals in the teeming ladle. All the impurities are removed by comparatively costly pyrometallurgical methods at high temperatures.

(vi) The steel ingots are cast very large compared to the final forged parts. For example, a 510mm steel ingot is reduced to 11mm to produce forged hand wrenches.[7] The amount of hot and cold working that has to be employed to produce the desired dimensions is so great that it causes a major increase in capital equipment and operating costs.

(vii) Metal loss by scale formation during frequent hot working is substantial when hot metal surfaces are generally exposed to the atmosphere.

(viii) Process scrap and finished product rejects are comparatively high.[8,9]

In spite of the above technological drawbacks of the existing process, the fact is that it works well and cheaply. During the past few decades many attempts have been made to improve the ironmaking-steel processing, based on conventional route, technologically and economically.[10] In particular attention has been paid to reduce the cost of production and improved product quality. As a result large, well-laid-out, modern plants, integrated operations, large blast furnaces, more continuous casting, more instrumentation and computer control, careful attention to operational details, and much lower specific energy consumption have been developed in several countries (e.g. Japan). An integrated works of 2-3Mt or more per year demands large quanities of heavy and elaborate equipment and many other processing equipment. All of these require enormous capital investment. The high amount of capital needed to build an economical integrated iron and steel plant is a matter of serious concern for many countries but excluding the rich O.P.E.C. countries. This is not only because of the magnitude of the investment, but also because of the fact that a substantial part of the investment is required in hard currencies.

Large integrated steel plants can decrease the cost of production but, in fact, the economy of scale depends more on the extent to which any single unit can be operated at a rate close to its full capacity than it does on size alone. Therefore, small scale operations (mini-mill) corresponding to production rates in the region of 300,000 to 1,000,000 tons

per annum may well be the area of future growth in steel production which is located close to the customer and producing a relatively narrow range of products. The experience of the mini-mills in the U.S.A. shows that it is more economical to run smaller plants at a high utilisation factor than large plants at a lower utilisation factor.[11]

The principal driving force for innovation is the need for processes which are low in capital cost, consume less energy and require less labour than the currently employed technology, and at the same time have the capability of providing improved product quality. The starting point for the consideration of new technologies is provided by the technological drawbacks of the existing technology, which are described earlier, and by Fig. 2.2. which indicates the principal cost components associated with the primary and the finishing ends of the conventional ironmaking-steel processing sequence.[10]

However, the application of new technologies is believed to be essential in order to satisfy all the drawbacks of the conventional route mentioned above. Thus, the next sections review the various alternative routes developed and under development for making forged parts with special reference to iron/steel. The alternative routes based on powder metallurgy techniques are dealt with in more detail in order to evolve the concept of the present study.

2.2. POWDER METALLURGY METHODS

Manufacturing routes for making structural parts based on powder metallurgy are the most important among the alternative

Fig. 2.2. The breakdown of energy consumption, labour and capital costs in the primary end and the finishing end of the ironmaking steel processing operational sequence.[10]

routes developed to date. The conventional powder metallurgy routes (Fig. 2.3) involving some form of compaction and subsequent heat treatment (sintering) provide a means for producing parts exhibiting a wide range of tensile strengths comparable to wrought material. In addition, powder metallurgy techniques have proved useful in making unique products like metal filters, controlled density parts and self-lubricating bearings. The economic advantages of the powder metallurgy approach are well known, e.g. reduced material and scrap losses, reduction or elimination of secondary machining and finishing operations. Further improvements have resulted from the use of mass production techniques, improvements in materials etc.

However, in almost all cases, the powder metallurgy parts are characterised by low ductility and impact strength and questionable fatigue resistance. Structural applications are therefore limited. The primary reason for these deficiencies lies in the residual porosity; typical void contents are in the range of 5-25 percent. Improvements in properties (fatigue, impact, ductility) have been achieved through approaches such as repressing, hot-pressing, infiltration, post-heat treatment, or modification of the pore morphology.

Clearly, a significant increase in the utilisation of powder metallurgy materials would follow from the achievement of full density - i.e. it is necessary to eliminate all forms of porosity. As might be expected, many varied approaches are being studied. The majority of these research and development programs involve either (i) the direct and complete

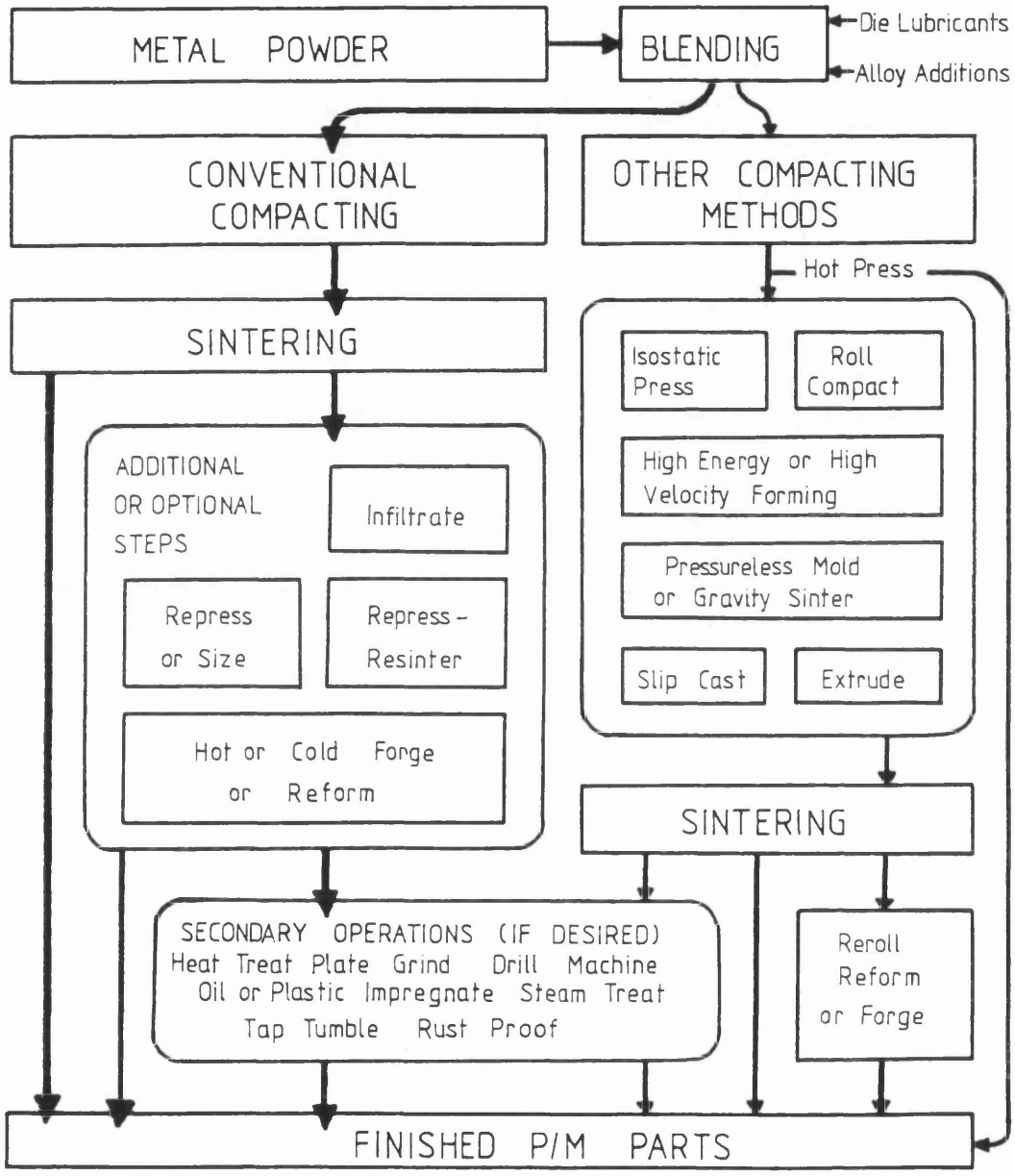


Fig. 2.3. Basic steps in the conventional powder-metallurgy process[12]

densification of the powder with improved compaction technology or (ii) forging of the powder preforms directly into finished shapes. These densification processes produce products from metal powder which are equivalent, and in many cases superior, to those from conventional melting, casting and metal working techniques.

Isostatic compaction, and in particular hot isostatic pressing, is perhaps the most promising method for the direct densification of loose powders or cold compacts. By the simultaneous application of temperature and pressure it is feasible to approach or achieve 100 percent density in a wide range of hard-to-work materials. High speed tool steels, superalloys, berylium, and the refractory metals are particularly amenable to hot isostatic pressing.[12]

A recent innovation in compaction, in which a uniaxial stress and an isostatic state of stress are combined (triaxial compaction), appears to offer further potential for densification. Developed and proven for cold compaction, the approach can be utilised at elevated temperatures.

The other general approach to the achievement of full density involves the preparation of an intermediate porous powder preform. Densification of the preform is then brought about by a hot or cold working operation. The procedure is illustrated schematically in Fig. 2.4. The porosity in the preform, which can vary from 30% to <1%, determines the subsequent mode of deformation. Most preforms have been prepared by die-compaction, cold isostatic pressing, hot isostatic pressing, or a combination of compaction steps.

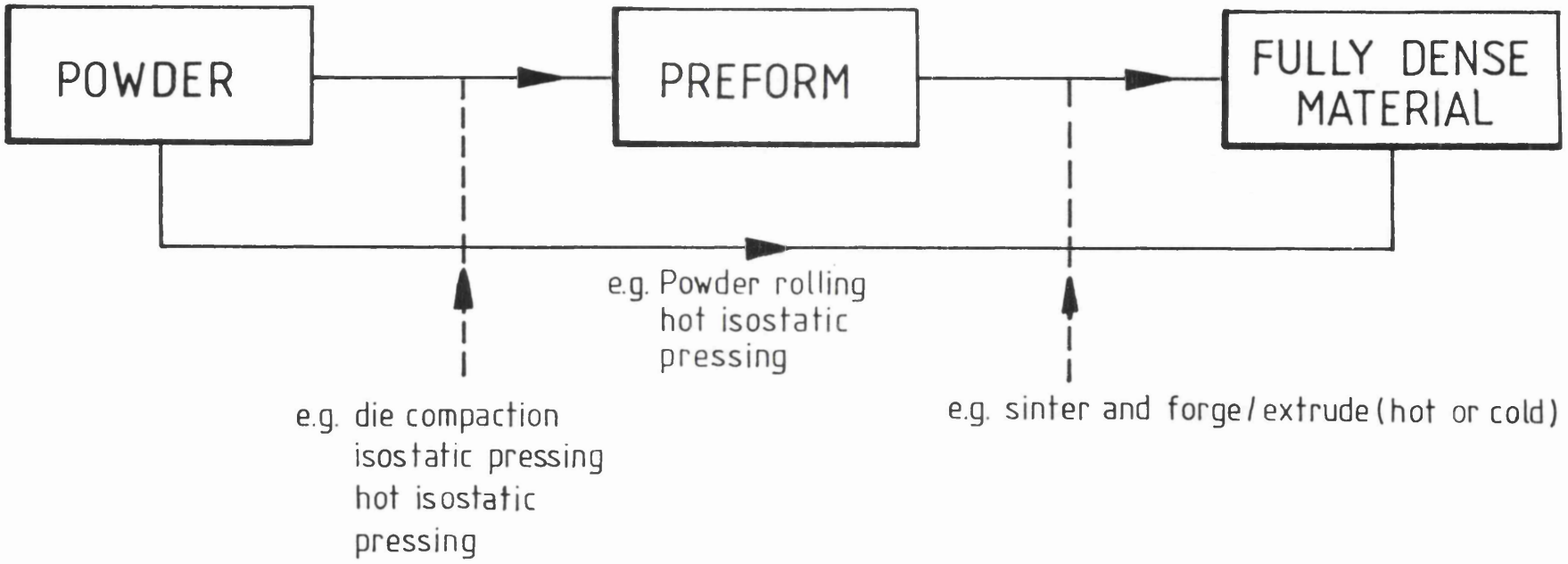


Fig. 2.4. Basic steps in the production of a fully dense material via an intermediate powder preform.[12]

The most frequently used working operations are forging, extrusion or rolling at ambient or elevated temperatures.

The primary aim of the following sections is to review the alternative powder metallurgy routes and to provide a background for the powder preform forging and the present study involved. Each of the powder metallurgy techniques will be briefly considered with respect to the same process variables, the more important uses and limitations, and the basic process operations employed in practice.

2.2.1. FORMING OF POWDERS BY APPLICATION OF PRESSURE

2.2.1.1. DIE PRESSING

Die pressing or compaction is considered as the 'conventional' technique in the powder metallurgy field and the most widely used of all the powder compaction techniques. The procedure is as follows: powder or powder mixture (i.e. mixed with lubricant or alloying powder) is placed into a rigid die and compressed by a press by means of an upper and/or lower punch situated on the upper and/or lower ram. Ejection of the compact from the die is assisted by using lower punch. A general view of the steps in a single action die compaction system is shown in Fig. 2.5. Variations of the basic procedure above have been developed; double action, double action floating die compaction, multiple motion compaction, multiple motion floating die withdrawal compaction, reflex action, and rotary compaction.[13,14,15]

The basic component of the die compaction system is a source of energy or pressure, usually mechanical or hydraulic. Mechanically operated presses are used to a large extent because

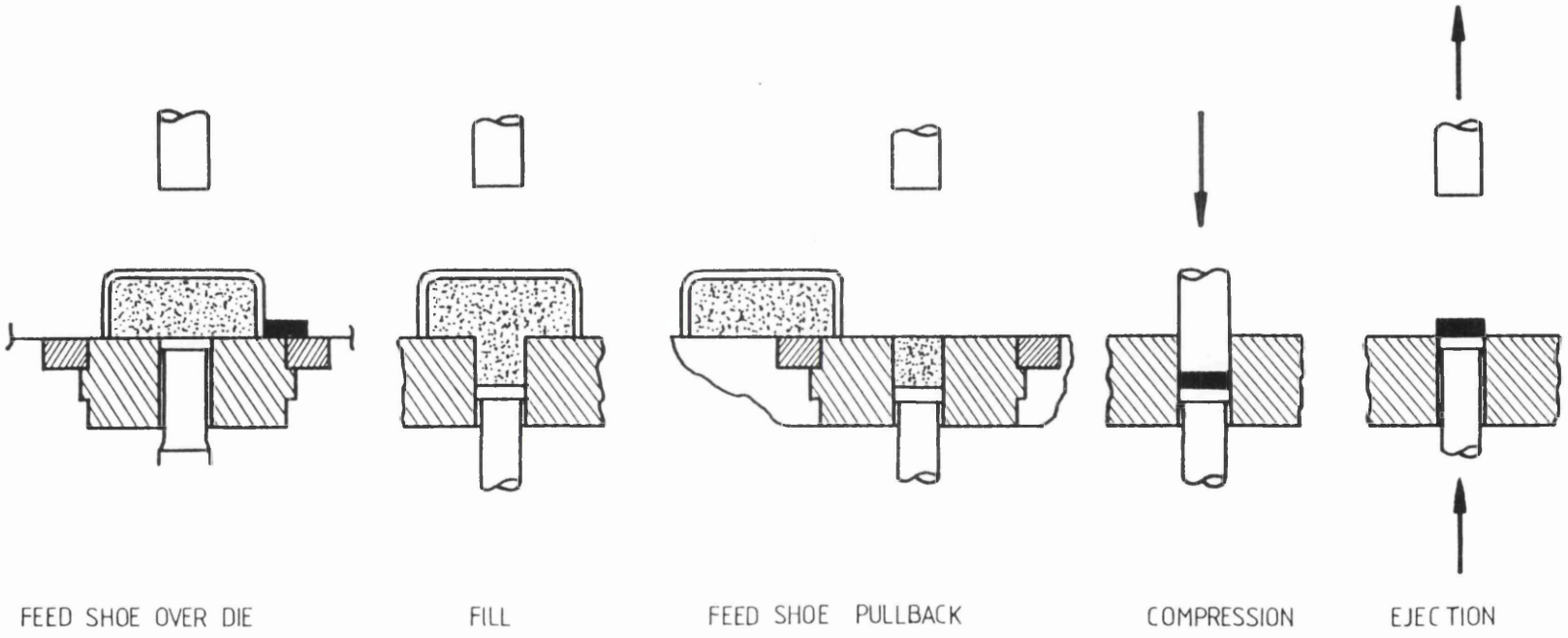


Fig. 2.5. Example of die compaction of powder.[14]

of the high-speed production rates, flexibility in die design, simplicity and economy in operation, and relatively low investment and maintenance costs. On the other hand the hydraulic presses used display superiority over mechanical presses in respect of power level, safety against overload, and flexibility in performance. The only disadvantage of the hydraulic presses is the low rate of production. A combination of mechanical and hydraulic presses can also be used.[13]

The second important component of this system is a properly designed die, without which satisfactory pressure distribution and precision of form in the compact cannot be obtained. Therefore, the die has to be tailor-made for each particular shape and for each different powder to be used. It must also withstand the pressure applied without deflection and resist the wear caused by friction of the fine particles against the side wall. Together with the die, punches and other necessary components (i.e. core rods and pressure pads) should be obtained with adequate strength and proper design.

The greatest advantage of die compaction is that parts of relatively intricate geometry can be pressed to close tolerances and high production rates can be obtained. Combined with well controlled subsequent operations, the parts produced require little or no final machining. The main disadvantage of die compaction is high tooling costs in order to make a compact of uniform density and strength.

2.2.1.2. ISOSTATIC PRESSING

Particulate matter can be consolidated into shapes by the application of pressure in a fluid by a flexible, non-

porous membrane. This process is referred to as 'hydrostatic' or 'isostatic' pressing.[16]

The technique was first used by H.D. Madden and followed by Fehse for the manufacture of tungsten products.[15] During the last two decades, there has been a great deal of research and development in this field by both powder metallurgists and ceramists.

In isostatic compaction, the powder is placed in a tightly sealed flexible mould, immersed into a pressure vessel, and subjected to a pressure acting simultaneously and equally from all directions. A typical isostatic pressure vessel is shown in Fig. 2.6. The two principal tooling methods are the wet-bag (free mould) and dry-bag (fixed mould) techniques.

In the wet-bag technique, the mould is filled with powder, well sealed, and placed into a pressure vessel which contains the pressurising fluid. The pressure chamber is subsequently sealed, the isostatic pressure is applied and then the mould is removed after pressure release. Finally, the compacted part is displaced from the flexible mould. This method offers considerable flexibility in terms of part geometry but its long cycle time makes it impractical for mass production.

In the dry-bag technique the elastic mould is fixed in the pressure chamber. This approach lends itself to higher production runs because automatic presses are available which use the 'rotational' or 'several cavities-in-line' principle.[14,16,17,18] Cycle times of the order of 10s

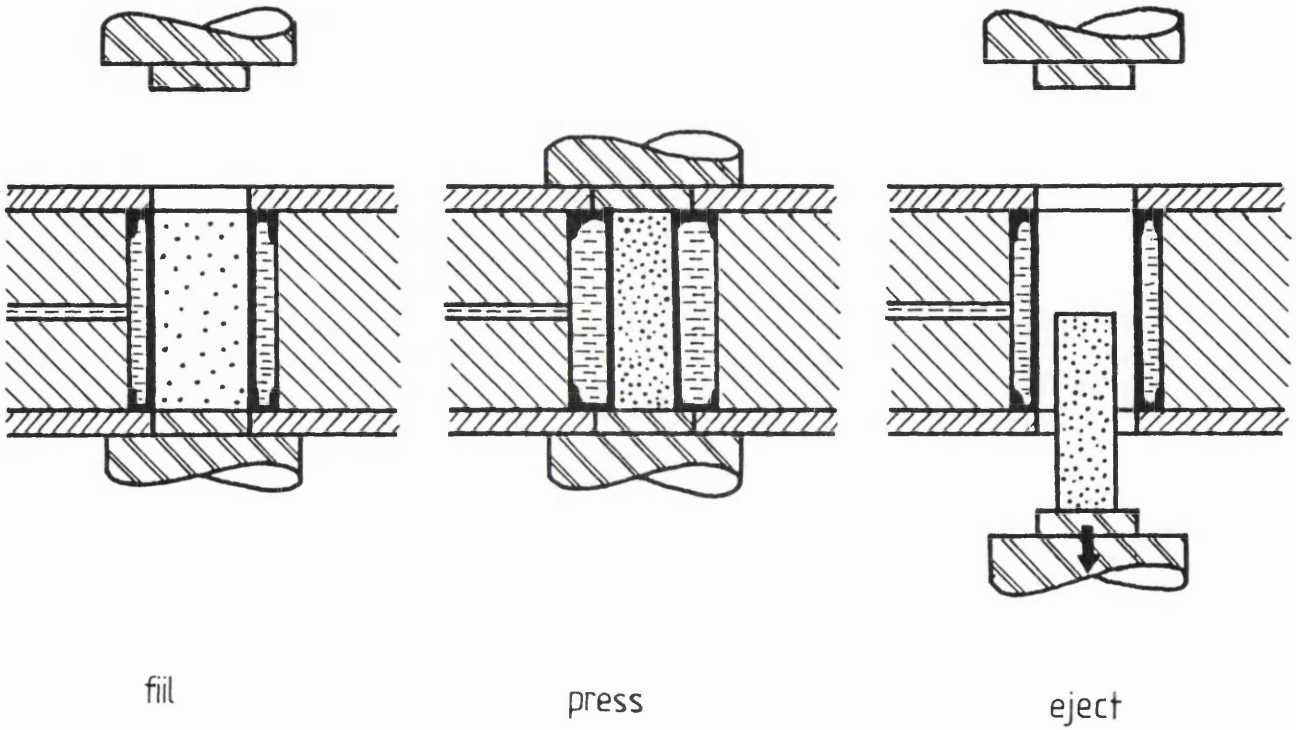


Fig. 2.6. Example of dry-bag isostatic compaction of a preform.[18]

appear feasible for the production of compacts (i.e. preforms).

The advantages obtained from isostatic compaction are:

(i) simple tooling,

(ii) uniform green density

(iii) high degree of uniformity of properties.

The green strength of an isostatic compact can be twice as high as that of an uniaxial compact made by uniaxial compaction because powder lubrication can be eliminated. This leads to simplified furnace design (no lubricant burn-off zone) and eliminates possible subsequent residues which could act as crack initiators.

Furthermore, the isostatic stress allows flexibility in part size and geometry. Very complex shapes and large length to diameter ratios can be achieved that would be impossible to attain with die compaction.

A comparison between isostatic and die compaction in regard to both technological and economic aspects is shown in Table 2.1. The only serious technological drawback of isostatic compaction is poor dimensional accuracy.

2.2.1.3. TRIAXIAL COMPACTION

The most widely used compaction technique is die compaction because of the economic advantage of short cycle time when compared to other compaction techniques. The major disadvantage of the technique is associated with density variation when a large length to diameter ratio is required. Many attempts have been made to eliminate or

Table 2.I. Comparison Between Isostatic And Die Compaction Of Preforms[18]

	Isostatic Compaction	Die Compaction
Technological advantages	Uniform density High green strength	Density gradients can be engineered into preforms
Technological disadvantages	Dimensional accuracy poor	Low green strength because of lubrication Density gradients because of die-wall friction
Economic advantages	Low tooling cost Flexibility Adaptable for induction heating	High production rates
Economic disadvantages	Low production rates	High tooling cost

reduce this problem but none have been fully successful, e.g. lubrication. As an alternative, isostatic compaction has been utilised, which solves the problem if the part lends itself to an economic solution by this method.

The triaxial compaction technique, however, makes use of combined stress-state wherein isostatic pressure is applied to loose powder and is followed by axial load.[19,20] In this compaction technique, the powder to be compacted is placed in a fixed mould (or a dry-bag tooling) with rigid end platens. This assembly is placed in a high-pressure cell and isostatic pressure is applied which eventually becomes the minor stress, σ_3, Fig. 2.7. At this point, the stress σ_3 acts in all directions, lateral as well as axial. Axial pressure is then imposed by a piston to increase the axial stress to σ_1, while the lateral stress remains at σ_3. The resulting principal stress difference $\sigma_1 - \sigma_3$ is numerically equal to twice the shear stress τ. Therefore the required compact density can be obtained by application of various sequences of pressure and by varying the magnitudes of confining and axial pressures. A comparison is made in Table 2.II for atomised iron powder compacted by die, isostatic and triaxial compaction techniques.

However, Koerner[20] concluded in his work that triaxial compaction offers significant density and strength advantages over other compaction techniques because of the sheer stress developed. The main disadvantage in the equipment requirement for addition of an independent axial pressure operation to what is essentially a fixed mould (or dry-bag) isostatic pressure.

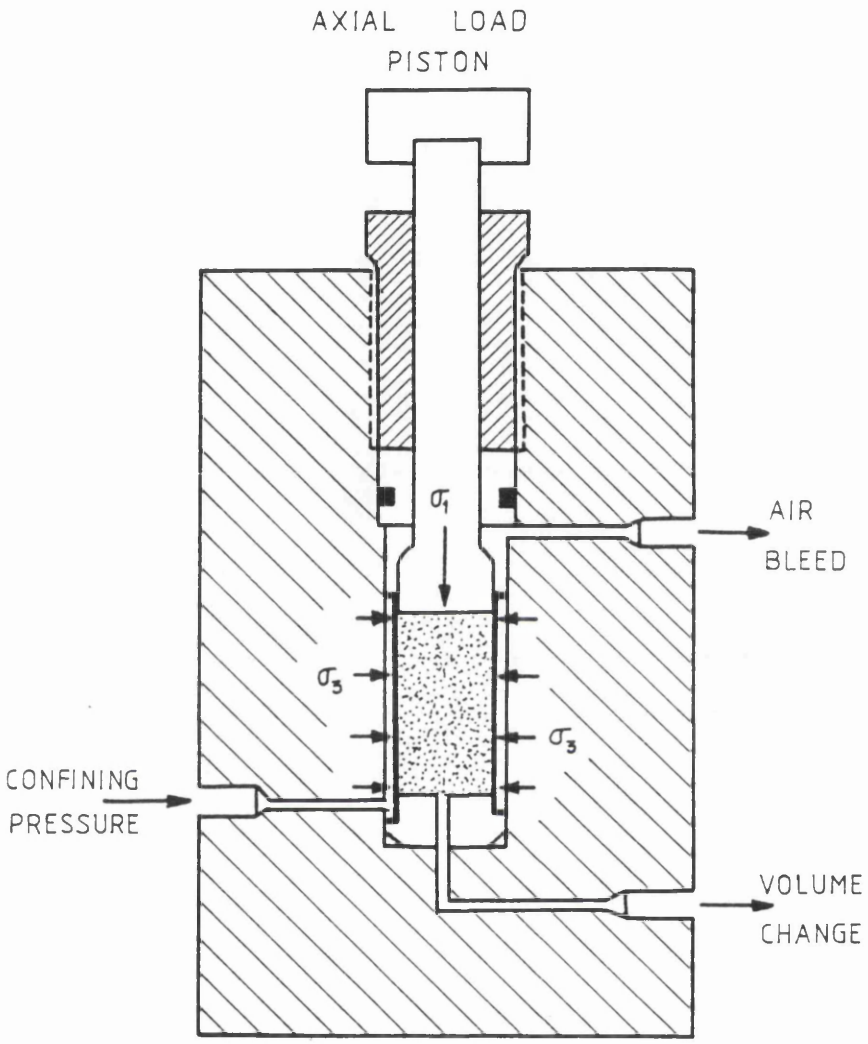
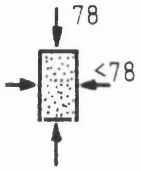
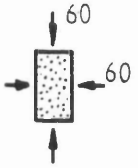
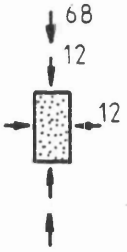
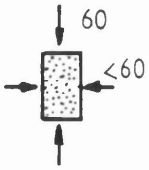
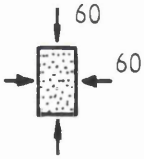
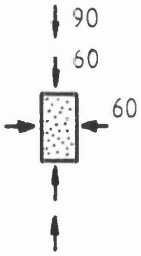


Fig. 2.7. Triaxial Compaction Chamber.[19]

Table 2.II. Comparison of Die, Isostatic and Triaxial Compaction Techniques.[20]

(a) CELL PRESSURE COMPARISON			
COMPACTION METHOD	DIE	ISOSTATIC	TRIAXIAL
PRESSURES (ksi)			
THEO. DENSITY	85 %	85 %	85 %
STRENGTH	20 ksi	25 ksi	55 ksi
(b) GREEN DENSITY COMPARISON			
COMPACTION METHOD	DIE	ISOSTATIC	TRIAXIAL
PRESSURES (ksi)			
THEO. DENSITY	78 %	85 %	94 %
STRENGTH	15 ksi	25 ksi	73 ksi

2.2.1.4. EXPLOSIVE COMPACTING

The technique of explosive compaction of metal powders is a logical extension of the familiar technique of hydraulic pressure consolidation.[21] The idea of explosive charges has been used by several workers over a number of years to study stress waves, fracturing, and plastic flow in materials.

The earliest reported process which utilised explosive in the powder metallurgy industry was that cited by McKenna, Redmond and Smith.[21] Simply, the process uses a piston explosively forced into a chamber containing a fluid and a rubber-bagged powder mix. The advantages cited were high pressure, producing high density, uniform density, and elimination of subsequent warpage. The technique was applied for compacting titanium carbide base cermets and refractory materials.

Apart from the above, other techniques have been used such as the use of single and double acting presses in which the pistons were activated by explosives.

Important variables in explosive compacting processes are the type of explosive, quantity of explosive, shape of explosive, duration and repetition of explosive force, and relationship of charge to batch weight and part shape. The problem, however, is complicated by economic and safety considerations.

2.2.1.5. VIBRATORY COMPACTING

Vibratory compaction is very similar to the conventional die compaction method but both pressure and vibration applied

simultaneously to the powder which greatly promotes densification and uniformity using low pressure; capital costs are thus relatively low.[22] This technique is also applicable to the compacting of nonductile materials.

The major factors influencing the green density of the compacts are the amplitude and frequency of vibration and the applied pressure. It has been reported[14] that there is an optimum amplitude and frequency associated with any pressure and also that there is an optimum pressure. The main problem in commercial exploitation of this method is the availability of suitable vibratory equipment.

2.2.1.6. FORGING

Forging of loose powders as a means of compaction has been used in a limited way[14] and was developed for the production of fuel elements for nuclear reactors. In this technique a metal container is filled with the powder (canning), heated if desired and finally given a conventional forging sequence.[14,23] When forging is accomplished the can material is removed by chemical or mechanical means. The resulting compact can have a very high density and may require no sintering if the powder was heated prior to the forging step.

It should be noted that the forging of powder metallurgy preforms will be considered later in this thesis.

2.2.1.7. OTHERS

These various methods for the compaction of powders briefly reviewed above are the basic methods for powder forging.

However, there are other techniques apart from these powder forming routes such as ; high energy rate forming, continuous compaction[14,15,26] rolling[14,27,33] and extrusion. Among these only high energy rate forming will be mentioned.

High energy rate forming[14,21,24,25] techniques have different methods of energy production including pneumatic, mechanical, explosive and spark discharge techniques. The main features of this technique are the very short time of pressure application and the very high amounts of energy imparted to the material. The advantages of this method are:

(i) increased lateral and axial dimensions of parts;
(ii) improvement in the magnitude and uniformity of density;
(iii) cheap, low grade powders can be used;
(iv) increased green strength removing the need for sintering in some applications.

The main drawbacks are:

(i) excessive punch and die wear;
(ii) reduced dimensional control;
(iii) difficulty of automation for rapid production rates;
(iv) high cost of safety precautions;
(v) expensive dies, punches and rods because of the high strength required.

2.2.2. FORMING OF POWDERS WITHOUT APPLICATION OF PRESSURE

2.2.2.1.LOOSE POWDER SINTERING IN A MOULD

The process simply refers to filling a die with the loose powder and sintering the powder in this die, which is often referred to as "pressureless moulding", "gravity

sintering" or "loose sintering".[14,34] The obvious factor of concern is the chemical inertness of the die with respect to the powder at the high sintering temperatures. For metals, various ceramics, such as oxides and carbides, and graphite may be used as the die material. The process is not used to any great extent on a commercial basis except for the production of P/M filters. This is due not only to the above noted problem but also because of such factors as :

(i) difficulty of part removal after sintering;
(ii) difficulty of die filling, especially for complex shapes;
(iii) high amounts of shrinkage due to sintering;
(iv) necessity of large amounts of moulds for high production rates;
(v) sensitivity of powder to vibrations.

2.2.2.2. SLIP CASTING

Slip casting is usually a forming process utilising fine powders, and slip casting techniques are generally appropriate to producing hallow ware or intricate shapes which cannot be produced by other techniques such as pressing or extruding.

The description of the slip casting process is briefly that a dilute suspension of the powder is cast into a porous mould of the required design. After casting the dispersed powder is deposited on the mould walls by the carrier action of the slip solution being absorbed by the mould. Excess slip is then drained out of the mould once the required thickness has formed, and the cast piece left to dry and

harden until it has sufficient strength to be handled. After removal of the piece from the mould thorough drying is carried out and the slip casting is completed. A sintering operation is required for densification of the slip-cast obtained.

This technique is widely used to produce clay and ceramic products in industrial practice, but the slip casting of metals is a relatively new idea. A number of papers have been published on the production of metal parts by slip casting.[34,36]

Some of the problems associated with this process are slip suspension stability, viscosity and slip solution, interaction between the slip and the mould, mould release and casting cracks. There are practical difficulties associated with the process due to ion exchange between the mould and slip solution which tend to stiffen the casting, fine particles from the slip tending to penetrate in to the mould by a capillary action which results in mechanical adherance of the mould casting leading to cracking on removal.

The literature reports that a number of common metals and alloys can be formed into complicated shapes by slip casting methods and more recently attempts have been made to utilise the process for more difficult materials. For instance Weber[35] has stated that Lidman and Rubina have prepared stainless steel slip, using 0.35 percent ammonium alginate, 0.25 percent polyvinyl alcohol and 25 percent H_2O. However, only solid castings were made and difficulties due to lack of green strength, reproducibility, and segregation were reported.

2.2.3. HOT PRESSING

Powder metallurgical products made by a cold compressing and sintering operation require high pressure and contain voids. These voids can be substantially eliminated by subsequent severe working and heat treatment, but such practice has not proved economical in most applications.

By applying heat during the compressing operation, plastic deformation of the powder particles is considerably increased, and therefore the pressure used is lowered and the resultant compact is practically free of voids.

Hot pressing is usually carried out by applying heat and pressure together which may be considered intermediate between ordinary cold pressing and sintering. However, this method has been widely applied to refractory type metals and ceramic materials with high melting points and high hardness and strength. The method has also been used for the more common metals and alloys. There have been several individual techniques developed,[13-15,18,37,38] which differ primarily in the types of pressure applied, in the types of die, and in the method of heating employed. Three methods are generally used for supplying heat to the work piece being hot pressed:

(i) induction,

(ii) passing current through the highly resistant powder mass;

(iii) enclosing the entire die assembly in a furnace.

Hot pressing is generally carried out in steel, graphite or ceramic dies. The pressure is exerted by presses (including isostatic) or by dead weights. Generally, the advantages of

hot pressing are as follows:

(i) Hot pressing combines pressing and sintering in one process, and therefore the time taken up in sintering would be eliminated. Furthermore handling and losses due to breakage of the green compacts would be minimised.

(ii) Closer size control is possible.

(iii) Because of (ii) a subsequent sizing or coining operation would not be required.

(iv) Hot pressing is less sensitive to the powders origin and to particle characteristics.

(v) It is possible to compress large compacts of high densities.

(vi) It is possible to obtain compacts having better physical properties than with cold pressing techniques.

Difficulties and limitations are:

(i) It is difficult to devise an equipment for mass production. The loose powder must be heated to the required temperature before pressing, therefore time must be provided to permit the flow of heat, and this slows down the process.

(ii) The choice of die materials capable of withstanding high pressures at elevated temperature is limited.

(iii) At elevated temperatures, die wear is substantially higher, and there are difficulties connected with welding on to the die surfaces.

(iv) In many practices, the material under compaction at high temperatures must be protected from oxidation with a suitable atmosphere. This may become inconvenient from a practical point of view.

(v) Provision must be made for ejecting the hot pressed piece into a neutral atmosphere until reasonably cold.

CHAPTER 3

POWDER FORGING

The previous Chapter covers the different conventional powder metallurgy processes for producing engineering components, in which metal powder is compacted to produce a green blank, and then sintered in a furnace at an appropriate temperature. The finished product is generally porous, typically in the range 5-25%. This inherent porosity is used in the production of filters and porous bearings. But it is undesirable when the mechanical properties of structural parts are considered so that it is necessary to attempt to remove as much porosity as possible. Methods that have been used are: infiltration, additional pressing and sintering steps, hot pressing and rolling and extrusion of powders. In general these are expensive operations and with some of these methods it is not possible to reach a level of dynamic properties similar to those of conventionally forged products of similar compositions.

However, the technique that offers the greatest potential mainly for ferrous materials, but also for non-ferrous metals, is the hot forging of powder metallurgy preforms in closed dies (Powder Forging). Before going any further, it would be appropriate to define the term powder forging. 'Powder Forging' is defined as follows: Hot densification by forging of unsintered, presintered or sintered preforms, made from powder, with accompanying significant change of shape. Often the designation 'Sinter Forging' is also used if a separate sintering step is carried out.[18]

The forging of powder preforms directly into finished shapes has been known for years although it is a relatively new technology, especially for ferrous materials. The method has been recently developed to a commercial level. The general process is illustrated in Fig. 2.4 showing that two different technologies are involved: preform making and forging. It is difficult to evaluate the process completely due to lack of necessary published data. But, in the following sections, a discussion of the technical and economical aspects of the process together with the process variations will be reviewed.

3.1. PREFORM MAKING

3.1.1. POWDER

The feed powder for this process can be made by a number of methods. Among these powder production processes[39,40] atomising and reduction processes are most widely used in high quantity production, while mechanical crushing and electrolysis are used primarily for the production of speciality materials in small quantities. The most flexible process is atomisation since it provides the capability to produce alloy powders and affords control over powder properties. Reduced powders from ore contain relatively large amounts of non-reducible oxide inclusions resulting in reduced ductility and/or impact properties of the final product.

Each production method gives a powder with different characteristics which affect subsequent powder forming processing steps such as compaction and sintering to a great extent. In conventional powder metallurgy the important

characteristics are chemical composition, particle size distribution, particle shape, apparent density, flow rate, surface conditions, and compressibility. In the forging of powder preforms, the most important characteristics are powder purity and the precise nature and form of the impurities present. Because it is considered that all P/M preform forgings are at or near full density, the impurities determine the properties of the final product as mentioned earlier. The particle size distribution may also have a significant effect on the preform forgeability and on the properties of the final product because of differences in impurity content, grain size, and the amount and nature of porosity[41]

3.1.2. PREFORMING

After a suitable powder has been selected, the next step in the fabrication sequence is to form the powder into a shape which can then be sintered and/or preheated for forging.

Generally the powder is preformed by die compaction in mechanical or hydraulic presses. Isostatic compaction is one alternative that is receiving considerable attention. Other methods, the loose-pack process, slip casting, and the Osprey process (which will be mentioned in the next Chapter), which eliminate completely the need for compaction equipment, are also used to make preforms for forging. Comparisons between mechanical and isostatic compaction, with a brief description of the processes, have been reviewed in Section 2.2.

Powder preforms are usually of simpler shape than conventional forging blanks or sintered parts. The dimensional requirements for a preform are less important because the finished shape and dimensions will be given during the final hot forging operation. Weight control of the preform has greater importance than dimensional accuracy in achieving the required tolerances. Preform design and control will be discussed later in more detail in Section 3.3.

3.2. SINTERING-PREHEATING

In the normal powder metallurgy process it is customary to sinter green compacts in a suitable protective atmosphere. The main aim in carrying out the sintering operation is to impart sufficient strength to the green piece to permit safe handling of the piece in the subsequent operation. It also removes undesirable impurities, e.g., surface oxide films, sulphur, etc., from the powder mass provided a suitable atmosphere is used. In addition, alloying, mainly with carbon, is introduced.

For powder forging, sintering is either carried out separately or combined with preheating, depending on the chosen fabrication sequence. Generally when sintering is considered separately standard conventional powder metallurgy equipment is used, such as continuous mesh belt, walking beam, roller hearth and pusher type furnaces.[13] The furnaces, heated electrically or by gas, contain a preheating zone for lubricant burnoff, a high temperature sintering chamber, and a cooling zone, and maintain a neutral or reducing atmosphere.

Powder preforms usually do not require admixed lubricant because high compressibility is no longer a major consideration and the preforms will have relatively low densities. Therefore simplication of such furnaces, e.g., elimination of the lubricant burn-off section, would be possible. But, utilisation of these furnaces for sintering-preheating of the preforms is still restricted. The production rates of traditional sintering furnaces are too low to match the forging cycle. However, recent developments in sintering processes showed that it is possible to reach production rates of up to 1000 $kg.h^{-1}$ with new conveyor-belt sintering furnaces,[42] an increase of 5 to 6 times over previous techniques.

Some of these furnaces of the pusher or rotary type are oil or gas fired, allowing no provision for atmospheric control. Use of resistance heating is also limited because preforms usually have non-uniform geometry as well as non-uniform intrinsic electrical resistivity.

The process can be improved further if the forging temperature is lowered. For example, sintering of ferrous materials is normally carried out at 1120-1150°C, whereas work to date indicates that forging of ferrous powder metallurgy preforms can be carried out at temperatures 150-370°C lower.[43]

Induction heating, another approach to sintering, has been studied for sintering-preheating of ferrous preforms.[44-4 It appears to be rather attractive for automated preheating of powder preforms. High temperature can be attained in a

very short time, a protective atmosphere can be easily incorporated and gas usage for the protective atmosphere can be reduced to a minimum. Floor space, capital equipment, operating, and maintenance costs can be comparatively low. Some technological problems still remain to be solved including alloying, reduction of surface oxides, bonding, and applying the heat uniformly.

In addition to the above economics of sintering-preheating equipment, various heating parameters influence the metallurgical process. According to Huppman and Hirschvogel[18] three main processes occur during sintering of the compacted powder, and these determine the final product properties:

(i) formation and growth of necks between adjacent particles by diffusion.

(ii) reduction of oxides present on the particle surface or as inclusions, and

(iii) alloying, mainly with carbon.

All three processes depend in varying degrees on temperature, time and atmosphere.

Sintering temperature, and to some extent also sintering time, influence the final properties of the conventional sintered parts. The difference in final properties directly reflects the degree to which solid necks have developed between adjacent particles and their strength determines the performance characteristics of conventional sintered parts.[46] For parts which are forged to full or nearly full density after sintering, the degree of necks formed

is less important. Arén et al[47] showed that there is no difference between the impact properties of iron powder forgings presintered for various lengths of time as soon as the forged density exceeds 90% of theoretical density. Neck strength and neck shape (pore geometry) also influence penetration of air and thus oxide formation in the preform during the transfer from preheating to forging. It is thought that this effect should be controlled by proper automatic handling and optimised preform design, respectively, rather than by changing the sintering parameters.[18] As far as interparticle bonding is concerned, elimination of a separate sintering operation seems possible.

Oxidation of the preforms during transfer from the preheating furnace to the forging press was considered by Cook[48] who showed that the degree of oxygen penetration increases with the exposure time to air and demonstrated that in the temperature range 800-1050°C oxygen penetration is very slow, while it becomes rapid above 1100°C. Arén et al[47] have shown that for preforms of better than 80% density, oxygen penetration is negligible if the transfer time does not exceed 1s.

Reduction of oxides present on the particle surface or as inclusions can be largely eliminated by using H_2 or carbon to form a gaseous product which escapes through the interconnected porosity during sintering. Graphite is admixed for its alloy strengthening capabilities but can also be used to reduce the oxides. Because a substantial carbon content in metal powders would impair compressibility, carbon is not usually prealloyed into powders, but admixed prior to preforming.

Guichelaar and Pehlke[49] studied the gas-metal reactions during sintering, and explained oxide reduction in some detail and how it depends on sintering conditions. Admixed graphite reacts with the oxides present on the particle surfaces to give CO_2 and CO. Below 700°C the reaction

$$C + CO_2 \rightleftharpoons 2\ CO$$

establishes a CO/CO_2 ratio. The degree of reaction of the oxides depends on this ratio, which approaches equilibrium with the oxides: Fe_2O_3, Fe_3O_4, and FeO. Above 700°C the excess carbon establishes a CO/CO_2 ratio which is reducing to the lowest iron oxide. As the reaction proceeds in the presence of excess graphite, the iron oxide is completely reduced to iron. At higher temperatures the CO/CO_2 equilibrium shifts still further towards CO, which as the predominant component causes the atmosphere to become even more reducing. This is the reason why at very high temperatures even such difficult oxides as those of Mn or Cr can be reduced if excess C is available. Guichelaar and Pehlke[49] found that at temperatures of $\sim$ 1100°C the reaction kinetics are so fast that the reduction of iron oxides is practically complete after about 3 minutes of total heating time. This time span would be compatible with induction heating.

The reduction of other oxides requires higher reduction temperatures than iron oxide. Mn and Cr oxides require reduction temperatures in excess of 1160° and 1120°C, respectively. Such high temperatures will present considerable problems because they would be quite close to the melting temperatures of eutectics in the respective multicomponent systems.

The solution of carbon in iron is not as well understood as the oxide reduction in the presence of C, but volume diffusion starting at contact points between iron and graphite particles seems the most likely process.

3.3. PREFORM DESIGN AND CONTROL

In the design of a preform, the fundamental considerations are the shape of the preform (dependent on the degree and the type of flow), the preform size (dependent on the density) and the weight of the forging required. Unfortunately, the fundamental data that would be required for formal design criteria have not been completely developed. However, some of these data have been published for cold and hot deformation, and will be considered later.

In most of the P/M preform design studies up to date, four types of deformation have been examined:

(i) Uniaxial compression: preforms compressed in one direction between parallel plates with no lateral constraint in the other directions.

(ii) Plain strain: preforms compressed in one direction in a confined die that provides constraint in the second direction and no lateral constraint in the third direction.

(iii) Repress: preforms compressed in one direction in a confined die and constrained in other directions, no lateral flow.

(iv) Closed upsetting: the limited uniaxial compression applied in the confined-die flashless forging which comprises simple upset that occurs

in the first stage and confined after limited flow allowed. This is, in fact, the most common practice of all types of deformation used in powder preform forging.

Antes[50] studied the room temperature deformation characteristics of iron and steel powder preforms to determine the formability and densification characteristics of these materials. In this study the first three types of deformation mentioned above were evaluated and it was found that the rate of densification increased with increasing amount of die restraint. Practical experience, however, shows that a certain amount of material flow is beneficial to densification and in many cases even indispensable for reaching optimum properties.[18-51]

Guest et al[52] studied the effect of final forging diameter to initial preform diameter ratio, D/Do, during deformation and found a critical diameter ratio of about 1.40. Above this critical diameter ratio there was evidence of slight radial cracking when the preform reached the die wall. No surface defects were observed in the fully compacted specimens. Kuhn[53] also studied the densification, flow and fracture behaviour of powder preform material and approached the preform design graphically by considering the allowable height to diameter ratio of preforms $Ho/_{Do}$, to fracture and then accomplishing the remaining densification by repressing in one operation.

In addition to these factors, the shape of the preform has to be considered whether to use a relatively unshaped or 'slug' type of preform, which is easier and cheaper to produce,

or one the shape of which is nearer to that of the finished component. The second approach appears to offer great potential and also better dimension control.

Preform density should be carefully evaluated. A major advantage of powder forging is the improved forgeability of the P/M preforms as compared to similar material at full density.[41] This improvement is directly related to the presence of porosity and the concomitant reduction in strength of the preform. In addition, lower density preforms are cheaper to produce and tend to have better filling capacity into thin sections or fine details. Higher density preforms, however, tend to require somewhat lower loads to achieve a given final fixed density for both cold and hot forging.[54] On the other hand, Brown[55] reports that a low density is advisable to be able to move the material under lightest loads. The difficulty here is that because of the higher relative porosity there is a tendency for the preform to crack. The other problems that may occur with relatively large amounts of interconnected porosity in the preform are oxidation and for carbon steels, decarburisation prior to and during hot forging. These could possibly be eliminated by the use of proper sintering-preheating atmosphere and well shaped preforms.

Finally, the weight of the preform may have to be controlled to within ± 1% to achieve the desired dimensional control, densification, and elimination of flash.

3.4. FORGING

Fundamentally, powder preform forging should be a very simple process which results in high-precision parts with a very good surface finish and improved properties.

This can be achieved only by optimisation and possibly automation of each step in the forging cycle.

3.4.1. FORGING CYCLE

The forging cycle of powder preform forging consists of fewer steps than are necessary in conventional die forging (see Fig. 3.1.) The first step, transfer from the preheating furnace to the forging machine, should be carried out as quickly as possible in order to reduce the danger of preform oxidation, as mentioned earlier. Mechanised preform handling is therefore advisable.[18]

Powder preform forging, as compared to conventional die forging, requires only one forging stroke because powder preforms can be made of a shape which require comparatively little material flow. Because of the absence of flash the total forging force for a given part can be reduced substantially. Therefore the forging energy is reduced because less material needs to be deformed. Thus lighter forging equipment can be used. Accurate temperature control (best carried out in an automated operation) is also essential for producing high-precision forged parts. Varying preform temperature would impair precision, not only by the varying thermal contraction on ejection from the die but also by its influence on the forging stress and thus the elastic straining of the forging machine. For similar reasons close temperature control is also necessary for the forging tool, and preheating to temperatures around 300°C is general practice.[18]

Oxidation of parts after ejection from the forging tool would also impair their precision and surface quality.

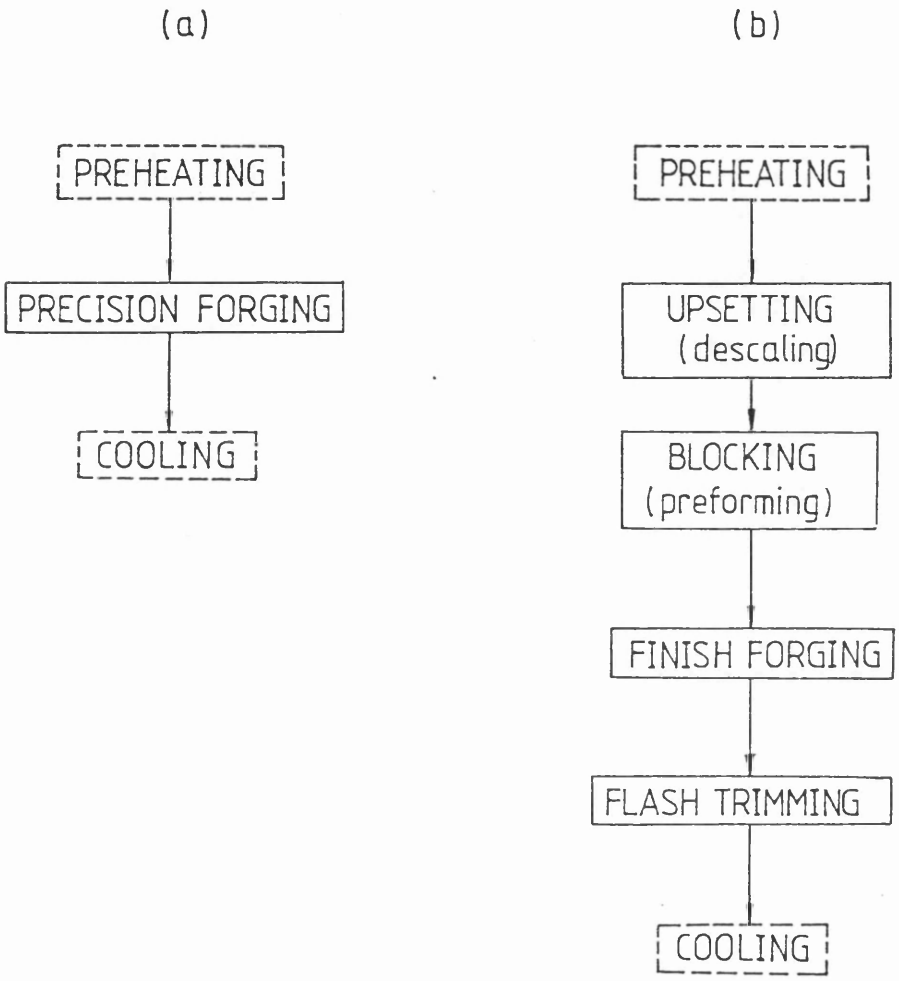


Fig. 3.1. Forming steps in (a) powder preform forging and (b) conventional die forging.[18]

Therefore, provision should be made for either quenching or cooling in a protective atmosphere or a protective medium like graphite.

Proper lubrication for hot forging is important because it extends die life, gives better densification of the surface, can reduce forging loads and promote more homogeneous deformation. In hot forging, the lubricant provides thermal insulation between the work piece and the die surface, and a colloidal dispersion of graphite in water is commonly used.

3.4.2. FORGING PARAMETERS

The three principal variables in powder forging are preform density, temperature of forging and applied load.

Preform density was considered in Section 3.3. In brief, preform density should be carefully evaluated before hot forging because lower density preforms are found to be more economical to produce and tend to have better die filling capacity. The influence of preform density on the final properties of the forgings was evaluated by several investigators. Fischmeister et al[56] showed that the yield stress in hot compaction is proportional to preform density. Moyer[57] found that forgings at formed densities greater than 7.7 $g.cm^{-3}$ (approximately 2% porosity or less) from the lower density preforms (6.2 $g.cm^{-3}$) provided the highest impact strength. Later, the effect of preform density on the properties of the final product will be discussed in more detail.

In principle, the influence of forging temperature on the flow of porous preforms is not different from the temperature dependence of forging dense materials, i.e., the flow stress

decreases as the forging temperature increases. Simultaneously, an improvement in formability is obtained. However, the absolute values of flow stress and formability at a given temperature differ considerably for porous and fully dense material. Densification proceeds in a manner similar to that found in simple upsetting experiments, and barrelling takes place until the material reaches the die wall. Because of the porosity still present during this stage, deformation can still continue under comparatively low loads. Once the preform is in contact with the die wall the densities and the load increase more rapidly as in recompaction,[18,41,54,55,58] (see Fig. 3.2.) This is the reason why relatively small deformation ratios, as mentioned earlier, are used.[52,53] The preform flows at low loads in the early stage of densification, and its mechanical properties are correspondingly low because the preform still contains porosity. Therefore, hot-tearing will occur when large densification ratios are used.

Usually higher forging temperatures, e.g., 1100°C, are used in P/M preform forging practices. It is also found that in forging of pure iron powder preforms very low stresses are required just below the α-γ transformation temperature than in the austenitic region[59,60] (see Fig. 3.3) but alloying additives can considerably change this behaviour. Relatively low forging temperatures can reduce oxidation but could cause higher tool wear.

One of the indirect influences of forging temperature on residual porosity is, in regions near the surface of forged parts, the chilling of the preform in contact with the relatively cold die walls. This can be minimised by using

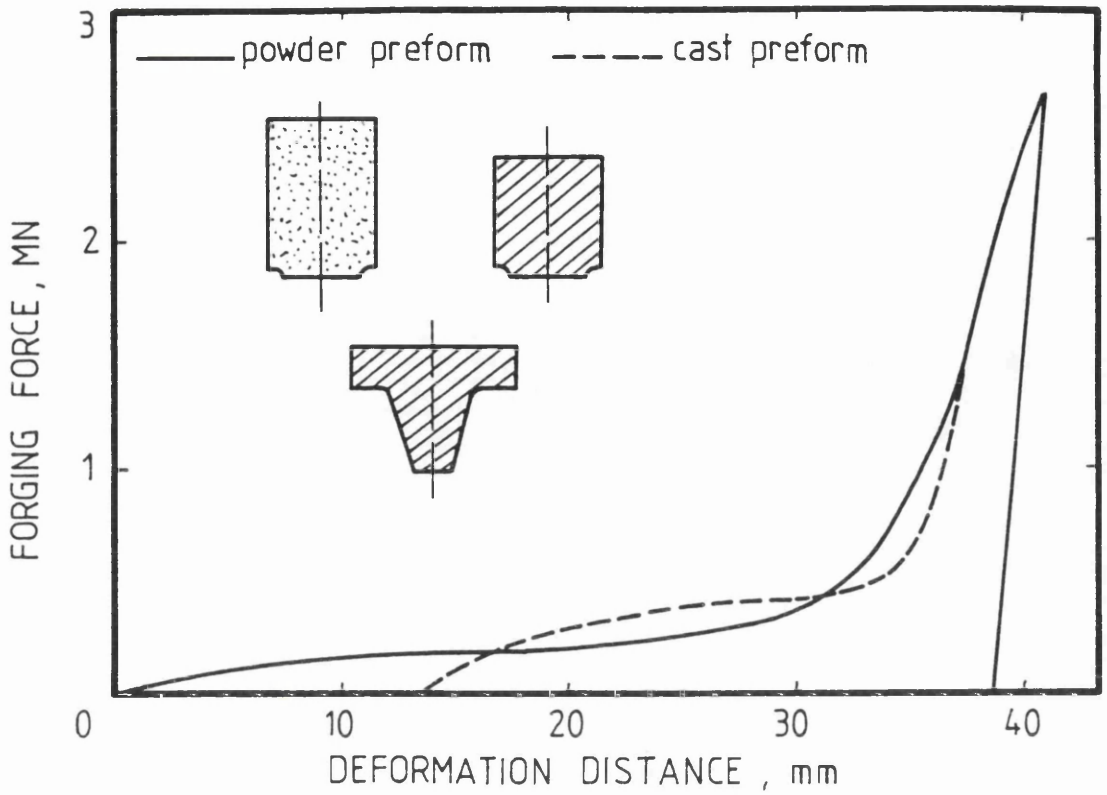


Fig. 3.2. Forging force v.deformation distance curves for porous and fully dense material.[18]

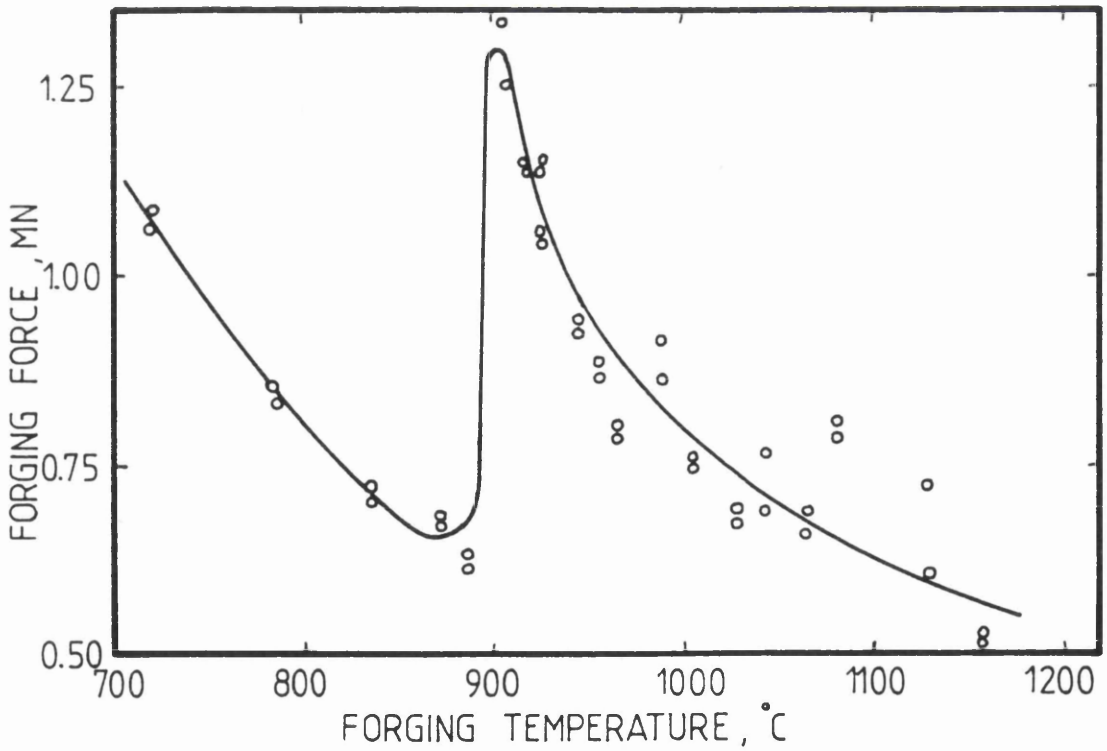


Fig. 3.3. Forging force as function of forging temperature for pure Fe (water atomised Fe powder; 80% dens preforms, 40 mm dia., 40 mm height; reduced 50% in height by upsetting.)[59]

higher forging temperatures and by heating forging tools up to appropriate temperature. It should be mentioned here that this surface porosity is also influenced by two closely interrelated forging parameters, namely, deformation rate and contact time. Huppmann and Hirschvogel[18] revealed that low forging rates produce more porosity in the outer region of the forging than high deformation rates. Under high deformation rates preform material flows faster and reaches the die walls in a very short time. This results in less chilling, therefore less porosity, of the forged part. In fact, surface porosity depends more on tool temperature and lubrication.

Forging temperature, force and the relative amount of shear are also of primary importance on the interparticle welding which occurs when pores are eliminated. Although there is not sufficient quantitative data available about this pore-welding mechanism, it is generally agreed that when shear forces are strong enough to break residual surface oxide layers on powder particles, highly effective interparticle bonding occurs.[18,61] It is therefore desirable to use high forging temperatures and deformation rates. This also promotes the welding of transient cracks.

To extend die life, for uniform pressure distribution during compaction and to facilitate ejection of the part from the die after pressing, lubricants are used. Furthermore, proper lubrication can reduce forging loads and promote more homogeneous deformation. In the case of hot forging, the lubricant provides insulation between work piece and the die surface and prevents, to some extent, the work piece from

oxidation.[62,63]

3.5. PROPERTIES OF POWDER FORGED MATERIALS

During the development of the powder-forging process, there has been considerable effort put into the measurement of mechanical and physical properties of powder metal forgings.[41,54,64-70,73] As a result it is now generally accepted that if full theoretical density is achieved during forging, properties will be comparable, and in some instances superior to, cast and wrought alloys of similar composition. Very small departures from full density can result in large reductions in resultant properties. (See Fig. 3.4) The investigation by Bockstiegel and Blande[65] showed that in the case of ASC 100.29 iron powder forgings, and in the case of ATST-A steel powder forging, the endurance limit as well as the tensile strength decrease relatively slowly as porosity increases to approximately 4 percent and somewhat faster as porosity further increases to approximately 10 percent. The endurance ratio in all cases remains practically constant over the entire range of investigated porosities. Elongation decreases much faster than tensilestrength. Ishimaru et al[66] also reported that the mechanical properties are decreased as the residual porosity increases and proposed a relationship between apparent density and tensile strength of sintered ferrous alloys. In the formula, tensile strength is proportional to the third power of the porosity.

The most severely affected property is impact strength. Fig. 3.4(c) shows that forging to full density is essential if dynamic applications are being considered. It is also important to consider the distribution of porosity in a forged part. As

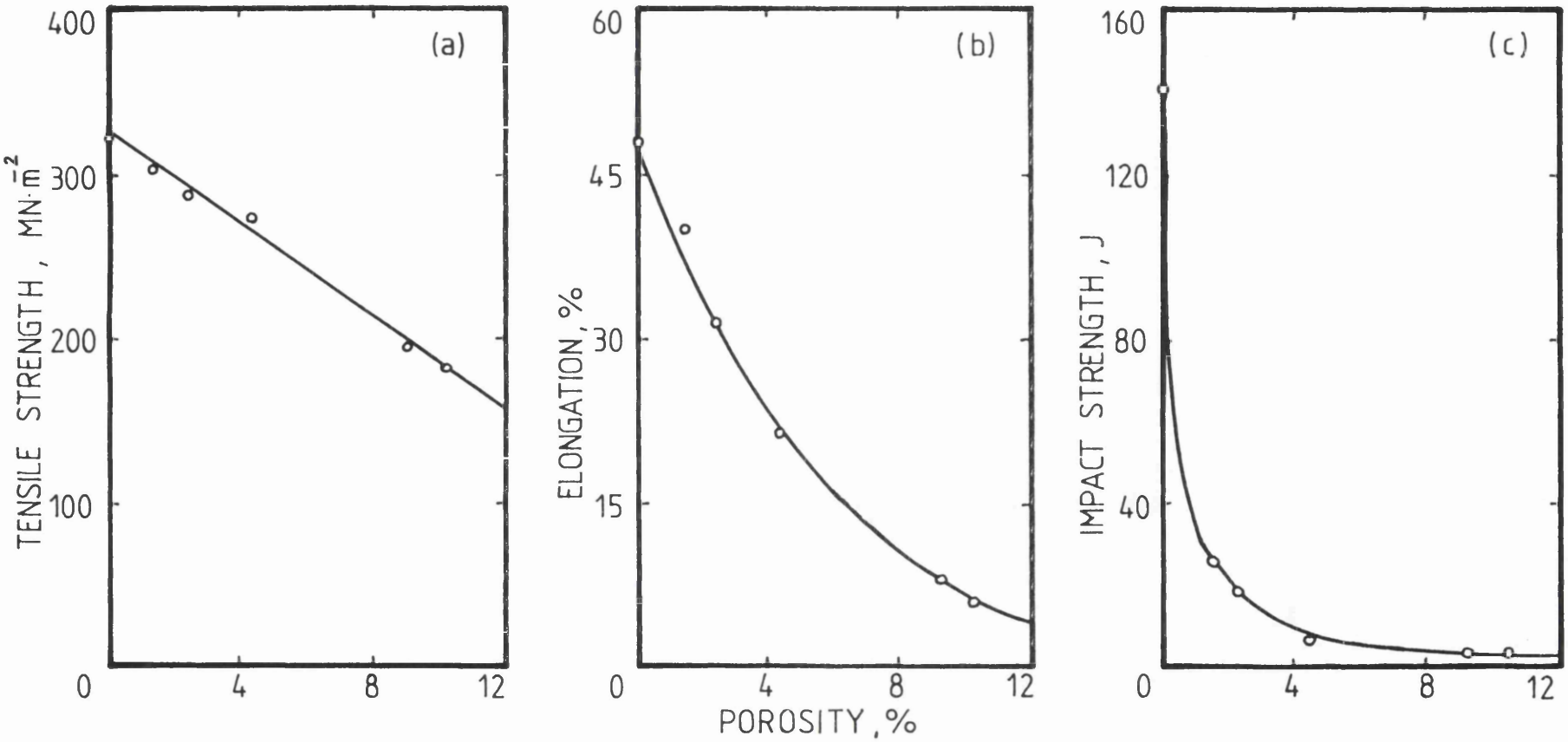


Fig. 3.4. Influence of porosity on mechanical properties of hot-repressed pure iron.[65]

mentioned above, chilling by the die walls and insufficient lubrication leads to increased porosity near the surface of a product where usually the highest stress will be encountered in service. Not only absolute porosity but also size and shape of pores influences the properties of forged parts. It is reported[64,65,69] that small evenly distributed, spheroidised pores will give better mechanical properties.

Probably of even greater importance than residual porosity is the effect of different non-metallic inclusions on the mechanical properties of powder forgings.[55,65,71-73] Some methods of powder production tend to produce non-metallic inclusions in the resulting powder. This is usually the case for reduction techniques, such as the reduction of iron ores or mill scale. Unreduced oxides or other mineral impurities are likely to be unaffected by subsequent processing. Powders produced by atomisation of a molten metal or by electrolytic deposition tend to be purer and have fewer, if any, non-metallic inclusions. Brown[55] has listed five reasons to account for the formation of inclusions in water atomised powders: (a) slag entrapment from the melt; (b) the presence of deoxidation products; (c) refractory breakdown; (d) sulphide formation; and (e) metal droplet interaction with the atomising water. These inclusions can be eliminated largely by careful atomising techniques.

Bockstiegel and Blande[65] studied the influence of slag inclusions on mechanical properties of forged iron and steel. In their investigation, plain iron powder forgings and tough-hardened steel powder forgings were deliberately contaminated with varying amounts of coarse and fine slag inclusions. With

increasing amounts of slag inclusions, impact strength was severely decreased and elongation somewhat reduced, while tensile strength and endurance limit were hardly affected. On the contrary, in the case of plain iron, the endurance limit slightly increased as slag inclusions increased to a certain level of inclusions. Equal percentages of slag additions, coarse or fine, had approximately the same effect on properties. For the same number of inclusions, the coarser inclusions are more harmful to impact strength than small inclusions.

Borland et al[72] also showed that the presence of impurities has a markedly deleterious effect on impact properties - coarse slag inclusions having a greater influence than the same volume percentage of finer inclusions.

The influence of preform density on the impact properties of P/M forged atomised iron powder was studied by Moyer.[57] In this investigation atomised iron powder preforms ranging in density from 6.2 $g.cm^{-3}$ (79% theoretical) to 7.2 $g.cm^{-3}$ (92% theo.) were hot forged using plane strain deformation to give a range of final densities from 7.4 $g.cm^{-3}$ to 7.83 $g.cm^{-3}$. Moyer found that lower density preforms yielded the highest impact strengths, >244J at deformed densities greater than 7.7 $g.cm^{-3}$ (approximately 2% porosity). Higher density preforms, 7.0 $g.cm^{-3}$, yielded a maximum impact strength of 149J to 163J and preform density of 7.2 $g.cm^{-3}$ yielded maximum impact strength of only 88J to 137J. However, impact strength increased with increasing final density and with reduced preform density. Uniform impact strength was found throughout the deformed preforms except where grain growth

occurred.

The basic nature of the metallurgical structure of P/M forgings is likely to be quite different from that of conventional forgings. The most important differences are homogeneity of structure, grain size and directionality of structure. In P/M forgings, the segregation will be minimum because alloy powder, as starting material, is practically segregation free.

It is reported[72] that the relatively small particle sizes in the initial powder and limited grain growth during sintering-preheating and forging should lead to smaller grain sizes in P/M forgings than in conventional forgings,[74] the controlling factors being powder particle size and purity, and sintering time.

It is also reported by several investigators[57,75] that the grains at the edges of forgings are coarser than at the centre. The grain sizes at the centre are smaller and more uniform. Moyer[57] measured the grain size of atomised iron powder forgings and found that the section of the outside specimen closest to the die surface of the forging had a coarse grained structure, ASTM 0-1 (319-226 μm); the remaining structure was fine grained, ASTM 8-9 (20-14 μm). The depth of the coarse grained structure varied but averaged about 3.2 mm deep.

The properties of powder forgings tend to be more uniform in all directions. Typically the elongation value of a powder forging may be slightly lower than that of an equivalent wrought steel, tested longitudinally, while the

reverse is true when the direction of testing is transverse.[55]

3.6. ECONOMIC CONSIDERATIONS

Powder metallurgy has become increasingly popular as a manufacturing technique. The principal motivating factor behind this growth has been economics.

Considering the simplicity of the process, as shown in Fig. 3.1, Hirschhorn and Bargainnier[41] listed the most important economical advantages as follows:

(i) reduced forging steps - ideally one blow in one die.

(ii) reduced forging pressure allowing the use of smaller forges.

(iii) reduced forging temperatures making preheating less costly.

(iv) reduced tool costs due to above factors.

(v) reduction in skilled forging personnel.

(vi) reduction in scrap due to elimination of flash and secondary machining operations.

(vii) reduction or elimination of various secondary operations due to elimination of flash, improved dimensional control and improved surface finish.

(viii) improved reliability, less rejections related to fewer and more simplified operations and greater consistency of forging stock (preforms).

Many engineering factors, such as improved mechanical properties and greater shape complexity, can also be included in the above advantages.

In order to understand the economics of powder metallurgy as a manufacturing process, it is helpful to know about the total cost. The total cost analysis made by Anderson and Winquist[76] is interesting. They consider that the factors affecting the total cost are material, direct labour, direct and indirect overheadsand administrative costs, including profit. As a simplified approach to analysing costs they used the following formula:

$$TC = M + DL + OH + A$$

where TC = Total cost

M = Material cost per part

DL = Direct labour cost per part

OH = Direct and indirect overhead

A = Administrative costs, including profit

They analysed the above formula and concluded that labour and overheadsbecome far more significant in small parts relative to material cost. In which case it makes little difference as to the selection of material. On the other hand, in large parts both material and labour costs are of utmost importance.

Hupmann and Hirschvogel[18] also made a cost analysis for the production of parts weighing about 0.5 kg. at a rate of 10 min^{-1}. The figures of 2.21 DM/part and 4.42 DM/kg (after making an addition to allow for a profit) were considerably higher than the price of die forging at 1978 prices. The above authors suggested that cost reduction could be made by utilising automation, high productivity equipment and simplifying design to reduce operations. For example, a separate sintering step could be eliminated which costs

0.30 DM/kg of total cost. The material cost could also be reduced by a careful selection of material. In summary, however, the total cost could possibly be lowered to a rather favourable price. Huppmann and Hirschvogel[18] also made a comparison between powder preform forging and other alternative fabrication techniques on the basis of a selected part and its service requirements, which is shown in Table 3.1.

Table 3.I. Compaction Of Powder Preform Forging With Competing Technologies[1]

Process	Powder Preform Forging	Sintering	Die Forging	Cold Forging	Precision Casting
Part weight, kg	0.1-5	0.01-1	0.05-1000	0.01-35	0.1-10
Height/dia	$\leqslant 1$	$\leqslant 1$	Not limited	Not limited	Not limited
Shape	No large variations in cross-section openings limited	No large variations in cross-section openings limited	Any, opening limited	Mostly of rotational symmetry	Any, any opening possible
Material utilization, %	100	100	50-70	95-100	70-90
Tolerances	IT 8-10	IT 6-8	IT 13-15	IT 7-9	IT 8-10
Surface roughness, μm	5-30	1-30	30-100	1-10	10-30
Production begins to become economical at number of parts (for 0.5 kg/part)	20,000	5,000	1,000	5,000	2,000
Main goal	High strength no machining,	Moderate strength porous materials, no machining	High strength machining to final shape	High strength, minimal machining	Intermediate strength, minimal machining
Cost of one production unit (sintering=100%)	250	100	150	150	100
Possibilities for automation	Good	Good	Limited	Very good	Limited
Price, DM $3kg^{-1}$ (1978)	4-5	3-4	3	4	6

CHAPTER 4

POWDER TECHNOLOGY (P/T) ROUTES

4.1. INTRODUCTION

The three basic steps for making parts from ore by a powder metallurgy route are:

(i) Extraction of metal from ore or ore concentrate in the liquid or solid form, other than powder.

(ii) Production of metal powder.

(iii) Processing of metal powder into a forged part.

The production of metal powder, either in the cold or hot (or liquid) condition, is common to all P/M forging routes which are a sub-group of the larger powder technology (P/T) route, see Fig. 1.1. There are some routes which are more removed from the traditional P/M routes described in this chapter. These start from non-metal powder, e.g. powdered ore or ore concentrate. They do not involve the production of metal powder either in the hot (or liquid) or cold condition at any intermediate stage, and yet operate on the basic principles of powder technology. Such routes will be called 'powder technology' or 'direct powder technology' since it is possible to produce forgings directly from ore or ore concentrate without handling any metal powder.

There has not been much research and development activity to date in the area of direct powder technology routes as previously outlined. However, during the past decade, a new such process for forging, namely 'The Direct Ore' process was announced[77] and some development on a similar line has been carried out in Sweden.[78] In the present context the

direct ore process is the only available example of the P/T route, but the spray forging process is also discussed in this chapter. In the case of the spray forging of metals, liquid metal droplets or particles are involved, and therefore, in a strict sense, it is an example of forging by particle metallurgy, see Fig. 1.1, but for the purpose of the present discussion it will be classified under the powder technology route.

4.2. SPRAY FORGING

Recently a particle technology process, which is termed the Osprey Process, has been under development in the U.K. by Ospray Metals Ltd., in conjunction with BOC Ltd.[18,79] The three basic stages of this process are : atomisation of molten metal; spray deposition of preforms; hot working of these preforms to produce forgings.

A spray of hot particles from an atomising unit is directed into shaped moulds. On impact the particles flatten and weld together to form a high density preform. The deposited preform is then subsequently forged and trimmed. It is claimed that the rate of deposition can be substantial and that steel forgings can be produced from the molten state within seconds. Osprey preforms are also claimed to be homogeneous, fine grained and with low oxide levels, with an average density of at least 95% and, more typically, over 98% of the theoretical value.

Huppman and Hirschvogel[18] stated that accurate weight control is difficult and it is also impossible to produce flashless high-precision forgings with this process. Further-

more, it appears doubtful whether the process can be fully automated and still be well controlled under large scale production conditions. However, the process is still in an early stage of development and it is reported[79] that a pilot production plant has been commissioned in order to establish its production potential.

4.3. DIRECT - ORE PROCESS

Because of the very high cost of the iron powder raw materials, consideration has been given to alternative cheaper raw materials for the powder forging process.[1] Different approaches such as direct-ore, spent shot and the Fibriron processes have been investigated to determine whether low cost forgings could be made by using different cheap raw materials including high-purity iron ore concentrates, spent shot and cast iron turnings. Of these the most important and most relevant to this thesis is the direct-ore process.

In the direct-ore method, the procedure is to mix the concentrate with carbon powder and a binder, followed by pressing into the shape of a preform. The preform is pre-heated in a neutral atmosphere to the forging temperature during which time it is reduced to sponge iron but retains its original shape. The sponge iron preform is quickly transferred to a press and forged.

Some of the problem areas given by Brown[77] are; binder, reducing agent and heating stage. The binder materials tried ranged from hot to cold setting resins, starches, gums, etc., right through to wall paper paste. In spite of the high cost, cellulose wallpaper paste was found to be satisfactory.

Reducing agent graphite used in the P/M industry is considered to be too expensive and gas-coke and petroleum are possible alternatives. The difficulty encountered during heating is the length of time to achieve complete reduction. When complete reduction is achieved then the tendency is to have a very slight excess of carbon in the final product or a small amount of unreduced oxide. Neither alternative is attractive. In terms of mechanical properties, excess iron oxide was found to be less detrimental than excess coke. Strengths of 379 $Mn.m^{-2}$ with 14% elongation in the former case and 621 $Mn.m^{-2}$ with nil elongation in the latter were achieved.[77] On the other hand Singer[1] suggested that the difficulty could be avoided at the cost of longer reduction times by using a reducing atmosphere in conjunction with the lower carbon content.

Another problem that could arise during the heating stage is control of dimensions. It is observed that during heating the green preform swells and cracks.[78] However, this process clearly deserves further attention, especially with the advent of cheaper methods of producing iron oxide superconcentrate in bulk.

4.4. A NEW P/T ROUTE FOR MAKING STEEL FORGING: SPONGE IRON PELLET FORGING

Current work at the University College of Swansea has shown that good ductilities ranging from 15-30% can be obtained by hot rolling compacts made from sponge iron pellets derived directly from iron ore concentrates. The pellets would be low in cost (possibly less than half the cost of powder) though less pure, but the higher degree of deformation during compaction

ensures good properties from fully dense material. 'Sponge Iron Pellet Forging' thus evolved from the established technique of powder forging and hot rolling of sponge iron pellets[80-82] by combining the advantages of both. The route schematically shown in Fig. 4.1. consists of mixing iron oxide superconcentrate powder with a binder. The mixture is then made into porous pellets in a pelletiser with the addition of water. The oxide pellets are dried and reduced at high temperature with either pure hydrogen or a mixture of carbon monoxide and hydrogen. The high temperature during reduction brings about some sintering in the particles to form sponge iron pellets. The sponge iron pellets are subsequently cold compacted into pellet preforms which are then preheated and hot-forged in a closed die in one blow to a finished forging.

The pellet forging route has similarities with the direct-ore process and powder preform forging process in the sense that the number of process steps is kept to a minimum. It differs from the direct-ore process because porous metal pellets are produced and made into porous metal pellet preforms. Therefore shape control of the preform is no longer a problem as mentioned in the previous section. It differs from the powder preform forging process because porous metal pellets are used instead of powder to make relatively low density preforms. This ensures high plastic deformation in the closed dies.

The process outlined above involves direct reduction of iron ore concentrates. Most of the impurities present in the iron ore are not reducible by hydrogen or carbon monoxide, even

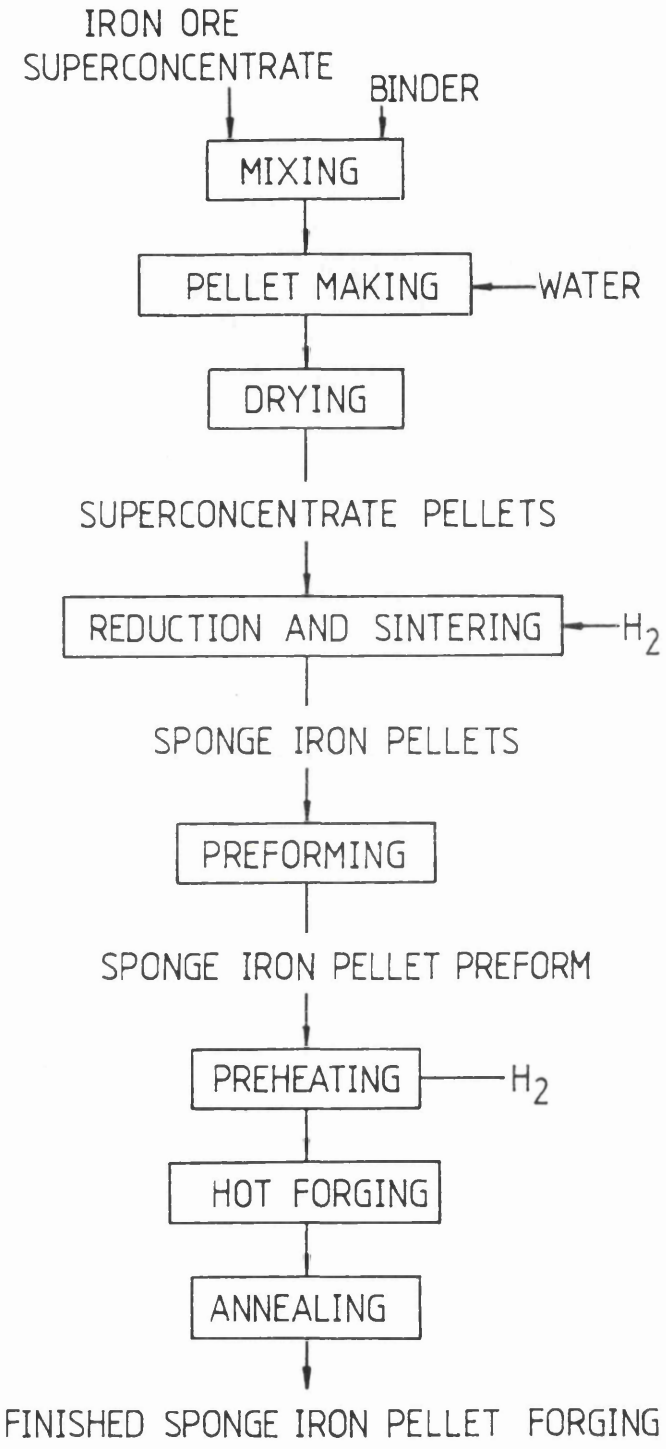


Fig. 4.1. A new direct powder technology route for the production of steel forgings from iron ore superconcentrate.

at a temperature as high as 1200^{o}C. Therefore all the impurities in the starting raw material would ultimately appear in the final product. To maintain a suitable final steel composition, it is therefore necessary to remove as much of the undesirable impurities as possible from the iron ore at the very beginning of the process, and therefore a high purity concentrate, often called superconcentrate should be used.

The following terminology will be used in relation to the material mentioned at the various stages of the proposed sponge iron forging route:

(i) 'Sponge iron pellets' is the term used for pellets obtained after complete reduction of the magnetite superconcentrate pellets.

(ii) 'Sponge iron pellet preform" is the term used for a preform obtained after cold compaction of sponge iron pellets.

(iii) 'Sponge iron pellet forging" is the term used for a forged piece obtained after the hot forging of a sponge iron pellet preform in a closed die.

(iv) 'As-forged' the term used for a forged piece as above

(v) 'Annealed' the term used for a forged piece as above and annealed.

CHAPTER 5

PELLETISATION OF IRON ORES

5.1 INTRODUCTION

Direct reduction of iron ores has become an important step in the worldwide steelmaking industry and there are many commercial plants in operation using 15 different processes.[84]

However, inadequate attention has been paid to the various granulation techniques that have made significant contributions towards bringing direct reduction to its current stage. The areas of interrelationship between direct reduction processes and granulation techniques are many and approximately 55% of the feedstock to direct reduction is in the form of pellets of one kind or another.[85]

Granulation, systematically manufactured agglomerates, involves a number of different processes and operations in which material in powder or lump form or in solution is converted into granules of fairly uniform size and shape; these processes and operations include crystallization, spray-drying, rolling, extrusion and pressing.[83,91] In the following sections agglomeration techniques, mechanism of agglomeration and compaction mechanism are briefly reviewed to provide a background for pelletising, and then pelletising is reviewed in more detail.

5.2. AGGLOMERATION TECHNIQUES

Agglomeration of iron ores and iron-containing waste products, flue dust, pyrites residues, etc., has been actively pursued since the end of the last century. Several agglomeration methods have been developed, the rate of

development increasing markedly during the Second World War, when a general ore shortage required that all available raw materials be used. The methods may be classified as follows:

(i) Briquetting

(ii) Nodulizing (rotary kiln sintering)

(iii) Vacuum extrusion

(iv) Sintering (grate sintering)

(v) Pelletising

Briquetting involves the pressing of ore fines, with or without a binder, into a block of some suitable size and shape, and then subjecting the block to a hardening process. The Grondal process, hot briquetting, and carbon bonded briquettes are a few of the know briquetting processes that have been used.[86,87]

In the nodulizing (rotary-kiln sintering) process,[88] iron bearing fines and carbon are passed through a rotary kiln (inclined at a few degrees to the horizontal) as a counter-current to hot gases produced by a gas fired burner. The agglomerate builds up in the walls of the sinter zone to form rings and then these are removed at frequent intervals by means of a scraper. The product is dense, slaggy, and difficult to reduce.

Vacuum extrusion has been mainly used in the ceramic industry to make strong dense shapes. In the 1950s attempts were made to adapt the process to agglomerating iron ores.[89] In this process the ore fines and/or flue dust are mixed with water and a binder such as bentonite is added if necessary.

The mix is fed into a de-airing chamber, which is connected to a vacuum pump, by means of a scroll feeder, and extruded through a series of dies by means of an auger. Short cylinders are produced by means of a knife which slices through the extruded material at regular intervals. The cylinders fall on to a conveyor, where they remain for a curing time of approximately forty-five minutes. They may be charged to the furnace as-cured or may be fired.

In the sintering process moist iron-ore fines are mixed with fine fluxes and solid fuel (normally coke breeze). The mix is loaded onto a permeable grate, the upper surface is raised to a high temperature by oil or gas burners and air is drawn downwards through the bed and grate. After a short ignition period, heating of the bed top is discontinued and the narrow combustion zone (1200-1500°C) moves downwards through the bed. In advance of the combustion zone water is evaporated and volatile compounds are driven off. In the combustion zone bonding takes place between the grains and a strong agglomerate is formed. Most of the heat in the gases leaving the combustion zone is absorbed in drying, calcining and preheating the lower layers of the bed. When the combustion zone reaches the base of the sinter mix the process is complete and the sinter cake is tipped from the grate and roughly broken up. After screening, the undersize is recycled and the oversize is cooled and sent to the blast furnace. Other types of sintering process routes have also been used, such as the Huntington Haberlein pot process, Greenwalt process, etc.[90]

Pelletising consists of two distinct operations: forming the pellets at ambient temperature and then firing

them at a temperature in the region of 1300°C. The pellets are formed by rolling moist fine ore, with or without the addition of a binder, in either a horizontal drum or an inclined disc.

Among all the agglomeration processes briefly described above, only continuous sintering and pelletising are now of any industrial significance. There are a number of reasons for the failure of the other processes such as: high fuel cost, unsuitability for large scale production, poor quality of product, etc. The sintering process is not suitable for agglomerating fine concentrates, these being best handled by pelletising.

The magnetite concentrates from which pellets were originally made were far too fine to be even considered as a blast furnace feed, and such materials can lead to problems such as high fuel consumption and low specific output when an attempt is made to sinter them. These factors, together with the fact that the pelletising process requires less fuel than the sintering process, gave added impetus to the use of this new process on a much wider scale on similarly sized material. Today pellets are not only produced from magnetite concentrates but also from haematite concentrates, natural ores, artificial magnetites and pyrite residues.

5.3. MECHANISM OF AGGLOMERATION

Bonding occurs between particulate materials during the agglomeration process. According to Rumpf,[91] who first published a classification, the main bonding mechanism can be classified into five major groups:

(i) solid bridges.

(ii) interfacial forces and capillary pressure.

(iii) adhesion and cohesion forces at not freely movable bonding bridges.

(iv) attractive forces between solid particles, and

(v) form-closed bonds (interlocking).

Solid bridges between particles in an agglomerate occur by sintering, chemical reactions, melting at the point of contact, hardening of bonding agents and by the crystallisation of dissolved materials.

Capillary pressure and interfacial forces in liquid bridges can form strong bonds. The magnitude of these forces, which are associated with the surface tension of the liquid, is determined by the size of the particles, the structure of the granule and the moisture content. Interparticle liquid can influence adhesion in different ways.[83] These are:

(i) the pendular state, when water is present at points of grain contact only and surface tension holds the particles together.

(ii) the fanicular state, when some pores are filled with water.

(iii) the capillary state, when all the pores are filled but the surface is not covered by a coherent liquid film.

Newitt and Conway-Jones[83] determined the green ball strength for each state and found that the cohesive stress for spherical particles in the capillary state was three to four times that in the pendular state. Rumpf[91] took an alternative approach to

the prediction of the tensile strength of green balls and he proposed two models which enable an approximation of the tensile strength of green balls to be made. Rumpf carried out experiments in which the tensile strength of balls was determined by a practical technique. The results were in close agreement with the theoretical values for pendular bonding but were always low for capillary bonding, due possibly to air inclusions in the balls.

Adhesional and cohesional forces in bonding bridges which are not freely movable can form bonds with two possible mechanisms. Firstly, viscous binders, such as tar and other high molecular weight organic liquids can develop bonds very similar to those of solid bridges. The effect of the interfacial forces on the liquid surface is reduced when thin layers of viscous binders are introduced between the particles. Therefore, the reduced mobility prevents the formation of a constant liquid pressure and this plastic material will also retain any surface shape given to it because the energy required for deformation would be much greater than the decrease in surface energy. On the other hand, many of these binders harden after a certain time and form solid bridges. Secondly, thin adsorption layers, less than about 30Å, can transmit the entire force of molecular attraction from one particle to another if they touch or penetrate.

Attraction between solid particles may exist and transmit tensile loads even if no material bridges exist between the particles. The typical short range forces of the Van Der Walls electrostatic or magnetic type can cause solid particles to stick together if they approach each other closely enough.

Decreasing particle size clearly favours this mechanism.

In form-close bonds, fibres, flat-shaped, and bulky particles are involved. With suitable motion and compression, particles interlock or mat with each other.

Bonding mechanisms can alternatively be classified into two groups, whether the bond is due to material bridges between particles or due to attractive forces without any material bridges being formed.

In the agglomeration methods reviewed previously, each involves one or more of the above bonding mechanisms. According to the mechanism involved the agglomeration processes can be categorized as those with binders and those that are binderless.

Many types of particulate matter do not exhibit inherent binding tendencies; therefore, a binder must be added to secure adhesion of the solid particles. The main binding mechanism for such cases are: bridges of highly viscous media; capillary pressure at the surfaces of agglomerates filled with liquid; and freely movable liquid bridges.

5.4. MECHANISM OF COMPACTION

The aim of compaction is to bring small particles into sufficiently close contact so that the forces acting between them are large enough to produce a product which has sufficient strength to withstand subsequent handling. Therefore, it is sometimes necessary to carry the compaction into the bulk compression stage in which the stressing is hydrostatic in character.

Powder compacts, briquette or tablets can be produced

by a number of techniques. All methods involve a common basic compaction mechanism.

When solid particles are compacted into a die, a reduction in volume will occur due to the following mechanism,

(i) At low pressures, rearrangement of the particles takes place to make a closer packing. Friction between particles uses the energy given at this stage and the magnitude of the effect depends on the coefficient of inter-particle friction of the material.

(ii) At higher pressures, elastic and plastic deformation of the particles may occur and material flows into void spaces. Low thermal conductivity and low melting point materials can more easily be deformed because the heat generated at the point of contact may be sufficient to raise the local temperature to a point where increased plasticity facilitates particle deformation. Interlocking of the particles may also occur.

(iii) Stage (ii) continues until the compact density approaches the true density of the material.

The mechanisms discussed may occur simultaneously. The relative importance of the various mechanisms and the order in which they occur depends on the properties of the particles and on the speed of pressing.

Ideally, the stress distribution throughout the compact is uniform with frictionless die walls. In practice, the

presence of frictional shear forces at the wall leads to a non-uniform pressure distribution causing variations in the density of the compact. This will result in distortion during the sintering step. Variation in density increases with the applied pressure and with the height of the compact for constant diameter; decreases with increasing diameter even at constant height to diameter ratio; slightly reduces with the addition of lubricant; and considerably reduces by lubricating the die walls or tools.[92]

A knowledge of the relationship between compacting pressure and density is important because pressure or force, more than any other factor, controls the attainment of high density, high strength and low porosity in green compacts and markedly influences the same properties in the final product. A number of empirical formulae have been proposed to describe the pressure-porosity relationship; however, none of these formulae is universally applicable because each gives acceptable results only over a limited range of pressures.

5.5. PELLETISING

The idea of rolling moist fine ore in a drum to form balls and then drying and firing was first put forward by Andersson in Sweden but was never used commercially.

A similar process was used by Brackelburg in Germany in the early 1930s. In this process sodium silicate was admixed with fine ore and hardening was achieved by heating to a relatively low temperature. A pilot plant was built at Rheinhausen but the process was not developed any further.

In the same decade Barrett and Dean of the U.S. Bureau

of Mines developed a balling process. Fine ore was balled in a drum without using additives and the green pellets were fired at a temperature between 500°C and the softening point of the ore. At about the same time, E.W. Davies and his co-workers at the University of Minnesota developed the pelletising process to treat the ultra fine mineral dressing products obtained from the up-grading of Mesabi ore. They used a drum for balling and a shaft kiln for firing. After the Second World War, work on pelletising was carried out in Sweden under the direction of Jernkontoret.[90]

After 1950 it was clear that the pelletising process was a commercially attractive method of agglomerating fine concentrates. Up to date several pelletising processes have been developed and used all over the world, and further developments continue.

5.5.1. THE PELLETISING PROCESSES

The pelletising processes are generally based on the formation of green balls by rolling a finely ground ore or concentrate to which binder is usually added together with a critical amount of water. These balls are then dried, pre-heated and finally fired to a temperature of 1250-1350°C, all stages under oxidising conditions. Thus, oxide bridging, grain growth and some slag bonding occurs, to produce the final pellet strength. The pellets are then cooled in air.

Green balls are normally produced in balling drums or discs but to a lesser extent, in specialised equipment. The following steps are basic to the pelletising process; each step is modified to suit both the equipment employed and the ore being processed:

(i) Feed preparation.

(ii) Green ball production.

(iii) Green ball induration

(a) drying stage

(b) pre-heating stage

(c) firing stage

(iv) Pellet cooling.

5.5.1.1. MECHANISM OF BALL FORMATION

In pelletising processes, the production of good quality balls is a prerequisite to the success to subsequent stages. Investigations into the mechanism of balling have demonstrated the importance of the moisture content. When the moisture content is below a critical minimum, it is unevenly distributed with the non-granulated material being the drier portion: under these conditions, when balling is attempted sticking to the surface of the balling equipment occurs and when dislodged tends to slide rather than roll. Excess moisture results in the formation of weak lumps and low ball formation rate.

This critical moisture content for balling initiates the formation of nuclei, or seeds, without which the process cannot proceed. Such nuclei are approximately saturated and have a slight excess of water at the surface which imparts a certain degree of surface plasticity. This enables partial deformation to occur when two such nuclei are in collision, and excess moisture is available to effect cohesion across the larger area of contact so formed. Further agglomeration is affected by one of two mechanisms, depending on whether fresh feed material is available or not, and the ultimate size

of the green balls produced is controlled by the residence time in the balling equipment.

Firth[93] postulated that the ease with which fine ores could be balled was proportional to the total contact area or number of points of contact, and that the particle size range had a marked effect on the ability of the material to ball.

Tigerschiold and Ilmoni[94] rejected this concept of contact area and they considered that probably the more important factor was the surface tension of the added water, acting as a compressing force to develop green ball strength but that the rolling action has a high compressing effect on the balls. Support for this hypothesis is seen in that dry powders will not form balls on rolling, and green balls, when immersed in water, can disintegrate quite quickly.

An investigation[83] into balling of closely sized sands showed an apparent linear relationship between ball diameter and balling time. Kapur and Fuerstenau[95] carried out tests with a comminuted limestone, in which the pre-wetted material was charged to a drum with no further addition of feed material; they used a photographic technique, as opposed to sieves, for measuring the size range of the balled product. These tests demonstrated that the average number of green balls as a function of the number of drum revolutions could be divided into three regions which are characterised by three different growth mechanisms:

(i) nuclei growth region.

(ii) transition stage.

(iii) ball growth region.

5.5.1.1.1. NUCLEI GROWTH REGION

When correctly wetted, material is charged to the balling equipment. The growth potential of the nuclei is due to the surface energy associated initially with a thin film of water surrounding the particles. When the particles come into contact with each other the liquid layers coalesce to form pendular bonds at the point of contact, thus reducing the area of water-air interface and the surface energy of the system. This lowering is a function of the size distribution, the shape and the packing of the particles and the thickness of the water layer. On rolling, highly porous loosely held aggregates and crumbs are formed in which the particles are held together by the discrete lens-shaped rings of liquid at the points of contact between particles, i.e., by pendular bonds. After a short induction period in which rearrangement and partial packing of the particles occur, these aggregates and crumbs form small, spherical, stable nuclei. This is the nucleation period, a pre-requisite of the balling process since balls grow from the nuclei. The nuclei formed represent an energetically more stable configuration, are quite porous and, as a result, consist of three phases; water, entrapped air and solid particles. The growth rate of the nuclei appears to be related to their specific surface and not to the specific area of the feed material. In this period the size range is quite small.

5.5.1.1.2. TRANSITION PERIOD

The nuclei, after further rolling action, change from what can be regarded as a bundle of randomly oriented capillaries whose walls are covered by a thin layer of water

and are compacted. The interstitial void volume decreases continuously and the constricted capillaries fill with water. The capillary forces then cause the material particles to be pulled together and this, with the rolling action in the balling equipment, causes the granule to densify rapidly.

This segregation of water occurs over a small number of revolutions and alters the growth mechanism. Apart from pockets of trapped air, the centre consists of two phases, solid and water. The shell of the densified granule is, however, still highly plastic and ball growth by simple coalescence of a number of granules can readily take place. Such granules grow much more quickly than those still present as nuclei. In this transition period the size range of the granules and the balls becomes much wider.

5.5.1.1.3. BALL GROWTH REGION

In the ball growth region (period), it is considered that the granules and balls consist of a tightly packed interior surrounded by a thin, wet shell. Kapur and Fuerstenau[95] considered that ball growth took place by the coalescence of two or more granules regardless of their relative size, i.e., whenever two or more granules or balls were packed together in a favourable configuration, rolling and deformation into a larger sized sphere occurred. Other possible mechanisms occurring during the ball growth region were postulated, which may affect the dynamics of balling:

(i) de-coalescence, where the shape of the clump results in parts of the conglomerate being sheared off;

(ii) fragmentation, where a ball bouncing with sufficient

velocity could lead to breakage:

(iii) abrasion and transference of small volume elements from the surface of one ball to another.

In (i) and (ii) the fragments produced were postulated to combine with smaller granules, or themselves, to form larger particles.

Newitt and Conway-Jones[83] considered that in ball growth the torque produced by the tumbling motion set an upper limit to the size of balls which can stick together long enough to be kneaded into a single larger ball by coalescence.

The most likely explanation of ball growth mechanism is that put forward by Capes and Danckwerts[90] which was confirmed by experimental work. Two mechanisms were identified:

(i) growth by assimilation. This occurs when balling is proceeding without the addition of fresh feed material.

(ii) growth by layering. This occurs when balling is carried out with the addition of fresh feed material.

Initially, the balling of iron ores in commercial plants was carried out for the most part in drums, but now discs are finding increasing application. These two processes are the most widely used.

5.5.1.2. BALLING DISCS

A balling disc consists essentially of a disc, fitted with a peripheral wall, which is rotated whilst inclined to the horizontal. Facilities are provided for the addition of the feed and moisture to the disc, the latter usually in the

form of fine sprays. Scrapers are provided which not only prevent build-up of material on the disc but which can be employed to control the flow pattern of the material on the disc. Ball growth occurs on the disc, and when the balls attain the desired size they are discharged from the disc. Thus the disc acts as a classifier, though in order to produce green balls in the close size range of 10-16 mm demanded in modern practice, the product must be screened to remove undersize and oversize material. This operation may be carried out at the disc or just prior to feeding balls to the firing unit. The undersize material is returned to the disc while oversize material is shredded down and added to the incoming feed material. Material recycled in this way can be as high as 25 percent, though modern practice is tending to maintain a lower level.

The major parameters controlling the disc output and the movement of material on the disc are the disc diameter, the angle of inclination, the speed of rotation and the height of the peripheral wall. Bazilevich[96] developed a mathematical model of the disc operation and considered that the optimum utilisation of the disc occurs when unballed material begins to move down the inclined plane after having reached the highest point on the disc. Under these conditions about 70 percent of the disc surface is being actively employed. The operating conditions are then defined by

$$\omega^2 \frac{d}{2} - g\,(\sin\gamma - \tan \phi_m \cos\gamma) = 0$$

where γ is the angle the disc subtends with the horizontal

d is the disc diameter

ϕ_m is the angle of repose of unballed material

ω is the angular velocity of rotation of the disc

g is the acceleration due to gravity

This equation can be rewritten

$$\frac{\sin(\gamma - \phi_m)}{\cos\phi_m} = 0.56 \times 10^{-3} n^2 d$$

where n is the speed of rotation (revolution per minute)

The speed at which the disc is rotated must be optimised for each material by adjusting the speed and the angle of the disc until maximum utilisation of the disc is obtained. A typical laboratory-scale arrangement of a disc pelletiser is shown in Fig. 5.1.

5.5.1.3. BALLING DRUMS

The balling drum process is shown in its simplest form in Fig. 5.2. It consists of a rotating drum, essentially an inclined cylindrical shell, with a length-to-diameter ratio usually 2.5 to 3.5, the dimensions of which are adjusted to give the necessary output rate. Feed material is introduced at the inlet end of the drum where the water sprays are situated. Ball growth occurs in the drum but, as opposed to the balling discs, no classification occurs and the product must be screened to remove undersize and oversize material. Both are recycled to the feed addition point, the latter first being shredded. Drums are provided with a scraping mechanism to prevent build-up on the shell surface.

Though drums are usually operated at very low slopes, e.g., an angle of 6° to the horizontal, it is usual to fit a retaining ring to the feed end to prevent spill-back of material.

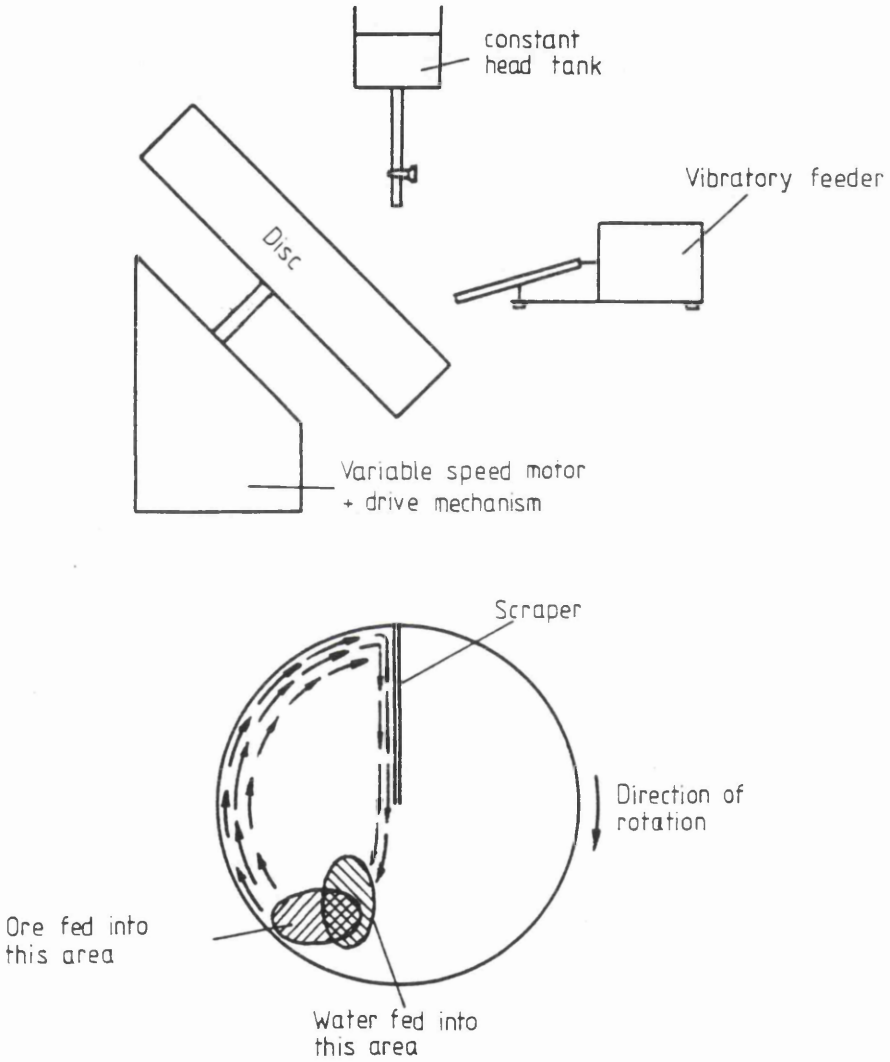
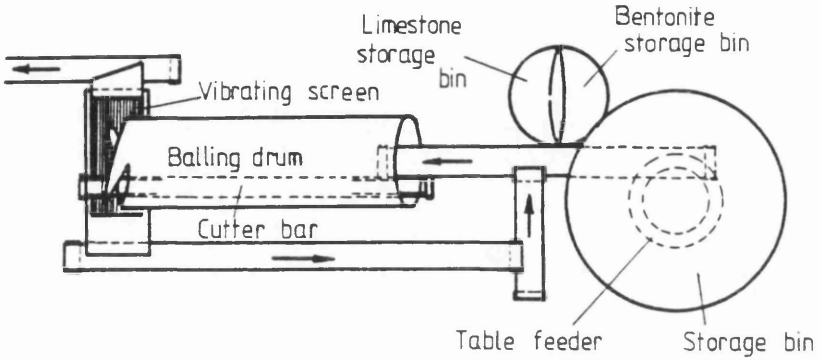
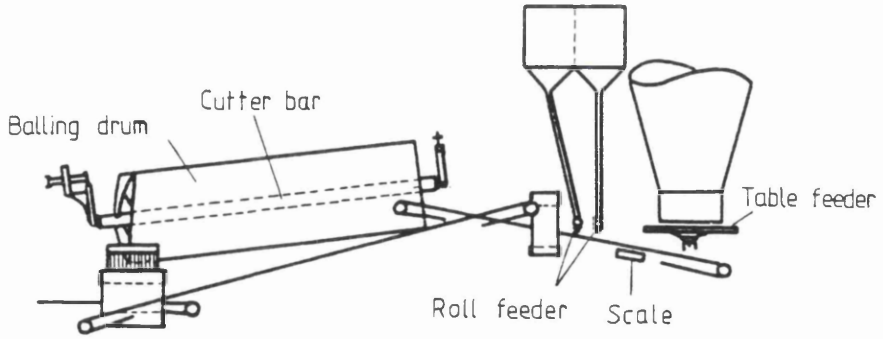


Fig. 5.1. Schematic diagram of disc pelletiser.



Plan



Elevation

Fig. 5.2. Typical balling drum arrangement.

The residence time in a drum is given by

$$T = \frac{0.037\ (\alpha + 24)L}{n\ DS}$$

where T is the residence time (minutes)

L is the drum length (feet)

D is the drum diameter (feet)

n is the speed of revolution (r.p.m.)

S is the slope of the drum (inches/foot length)

α is the angle of repose of the feed material

Ideally it should be possible to operate the drum in the same way as the disc, in which the time for ball growth is equal to the residence time, given by the weight of material actively being balled in the drum divided by the feed rate. This, however, is not possible because the drum does not act as a classifier, and in order to promote the correct rolling and tumbling action to produce good quality balls, there is an optimum speed and bed depth of material in the drum. The drum operation is therefore restricted by the need to satisfy three constraints; the speed of rotation, the depth of material in residence in the drum, and the time required for balling. The output from a drum can be increased by increasing the diameter or the length of the drum, or both.

5.5.2. BINDERS

Throughout the development of the pelletising process, additives have been employed to improve both the operation and the economics of the process. Most of the additives used as binders are either inorganic or organic. Bentonite is the most widely used inorganic binder, and organic binders such as starch, dextrin, celacol and bitumen are also used.

Considerable effort has been expended to find cheaper and more effective binders. This work has been directed towards:

(i) promoting and facilitating the balling of iron ores;
(ii) the improvement of green and dry-ball strength;
(iii) improving the properties of the fired or reduced pellets.

A bonding agent must have strong adhesion to the surface of the solid and must also possess considerable cohesion within the bonding matrix. The bonding agent should be compatible with the chemical process to be subsequently employed and, preferably, should function as a reducing agent. Coal tar pitches and petroleum bitumens come close to being ideal bonding materials. They are sufficiently fluid at 100-120°C to flow and readily wet solid surfaces for good adhesion.[85]

5.5.3. PELLETS FOR DIRECT REDUCTION OF IRON ORE CONCENTRATES

For a number of reasons direct reduction of iron ores has grown rapidly; two main reasons are shortage of coking coal and the need to use iron ore fines.[84,97]

Pelletising, among other agglomeration processes, is widely used for direct reduction of iron ores. Reduced pellets are used in the blast furnace to improve blast furnace performance, and are also used in electric or BOF steelmaking. The chemical characteristics required for each application are dependent on the type of ore concentrate and the process itself.

In recent years, however, some work has been carried out on perfecting low cost powder metallurgy routes for making steel strip and forgings from magnetite/haematite superconcentrates.

For these practices the superconcentrate is reduced in the form of strip, pellets, or any desired shape, and then subsequently compacted into the finished product.[1,77]

Organic binders rather than bentonite have been used for agglomerating iron ore concentrates from which steel strip and forgings are directly produced. Although bentonite is commonly used, it is expensive and requires a high firing temperature and leaves a residue after direct reduction, and this will have an adverse effect upon the properties of the final product. An investigation[85,98] carried out in the Metallurgy Department at University College Swansea on the suitability of binders for pelletising iron oxide particles shows that pitches and bitumens are suitable for these requirements. They leave no harmful residue, are available in large quanitities, are relatively inexpensive and only require a baking temperature of about 200°C to give comparable properties to those pellets using bentonite.

CHAPTER 6

AIM OF THE PRESENT INVESTIGATION

The aim of the present investigation is as follows:

(1) To determine the feasibility of the proposed new direct powder technology route outlined in Section 4.4. for making steel forgings direct from iron oxide superconcentrate on a laboratory scale involving separate operations.

(2) To study the behaviour of the material in various processing stages, and optimise the different processing conditions for making acceptable quality forgings from the chosen iron oxide superconcentrate by the proposed route, investigating the following factors:

(a) Pelletisation and the behaviour of pellets during drying and combined reduction/sintering

(b) Preforming behaviour of the pellets

(c) The effect on the final forging of -

(i) Pellet size

(ii) Preform density

(iii) Deformation mode

(iv) Forging force

(v) Sintering prior to forging

(vi) Annealing treatment.

(d) The effect of annealing atmosphere on the properties of forging.

(3) An assessment of the properties of the hot-forged and annealed forgings produced under the established processing conditions, with special reference to the following:

(a) Mechanical properties

(b) Amount, nature, size, and size distribution of the inclusions.

(c) Grain size and distribution

(4) To compare the properties of the sponge iron pellet forgings as listed in (3) with those of forgings from reduced iron powder and atomised iron powder (including making pellet forgings from these powders by the route adopted for the sponge iron pellet forgings).

CHAPTER 7

EXPERIMENTAL PROCEDURE

7.1. MATERIALS

7.1.1. IRON OXIDE SUPERCONCENTRATE

The most important raw material for this investigation was the iron oxide superconcentrate , a magnetite superconcentrate supplied by L.K.A.B. of Sweden. This was produced from a crushed magnetite ore containing about 50% iron which was ground to 1mm in a rod mill and then magnetically wet-concentrated in three stages. The concentrate then contained about 68% iron, and was reground and magnetically concentrated again in three stages, when the concentrate contained 70.7% iron. Further concentration was carried out by flotation followed by drying. This material can be produced economically on a large scale. The chemical composition and size analysis of the superconcentrate used are shown on Table 7.I.

A typical scanning electron micrograph of the superconcentrate is shown in Fig. 7.1. showing that the magnetite superconcentrate particles were free of porosity.

7.1.2. IRON POWDERS

In order to compare the results with conventional powder forging, sponge iron powder and atomised iron powder were used.

7.1.2.1. SPONGE IRON POWDER

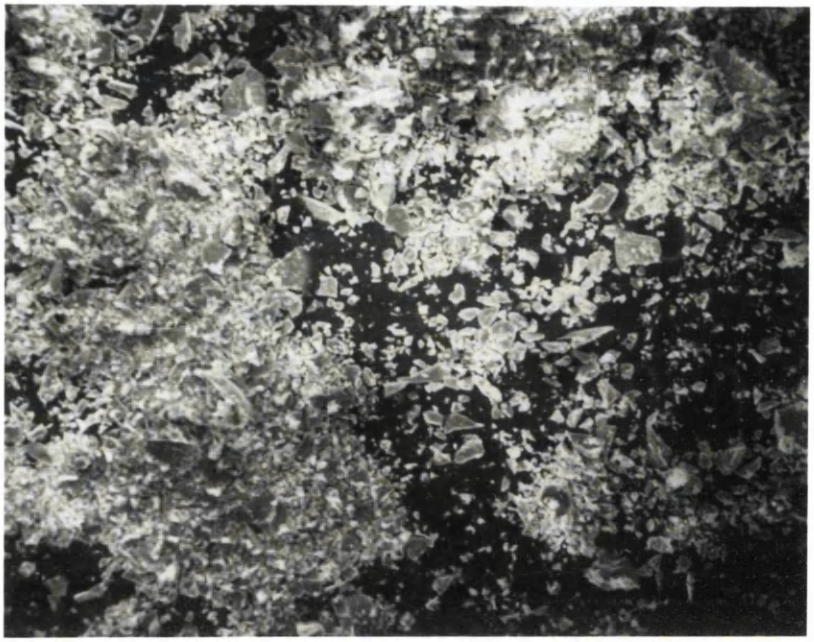
Sponge iron powder, NC 100.24 supplied by Höganäs AB of Sweden, is the most widely used grade for powder metallurgy purposes. This was chosen because of its spongy structure and chemical composition, which is to a certain extent similar to

Table 7.1. Chemical And Size Analyses Of The Magnetite Superconcentrate.

Chemical Analysis	
Constituent	Wt %
Fe	71.90
Fe_3O_4	97.00
Fe_2O_3	2.30
SiO_2	0.06
MgO	0.15
Al_2O_3	0.15
TiO_2	0.14
V_2O_5	0.20
Na_2O	0.023
K_2O	0.015
P	0.004
Cu	0.005
S	0.003

Size Analysis	
Size m	Cumulative Weight %
+ 74	0.6
+ 62	1.1
+ 44	9.7
+ 25	57.2
+ 20	69.0
+ 16	75.2
+ 11	84.1
+ 5	94.3
+ 1	99.1
< 1	0.9

Fig. 7.1. Magnetite superconcentrate particles. x 125



the H_2 reduced magnetite superconcentrate pellets used in this investigation. The compressibility of NC 100.24 is very good. Due to the spongy structure of the powder particles the green and edge strength of the compacts become very high. The chemical analysis and physical properties of the Höganäs NC 100.24 are given in Table 7.II.

7.1.2.2. ATOMISED IRON POWDER

Atomised iron powder, ASC 100.29, supplied by Höganäs AB of Sweden, was also used due to its purity and softness. This was specifically chosen in order to widen the understanding of the mechanism of the deformation involved in this study. The chemical analysis and physical properties of the Höganäs ASC 100.29 atomised iron powder used are shown in Table 7.III.

7.1.3. BINDER

A binder was necessary to form a homogeneous pellet of magnetite superconcentrate and iron powders. A water soluble 'Celacol' powder, manufactured by British Celanese Co. Ltd., was used as a binder, which was essentially methyl cellulose. This binder is available in various viscosity grades, and the M450 grade was used. The ash content of the binder as Na_2SO_4 was 1% max., and the charring temperature was 330°C approximately.

7.1.4. GASES

Pure hydrogen supplied from cylinders, having a dew point of -65°C, was used for reducing and sintering the superconcentrate pellets and iron powders pellets. For flushing the furnace chamber before passing hydrogen and after stopping hydrogen, nitrogen or 96%N_2 + 4%H_2 mixture was used. Pure

Table 7.II. Chemical And Size Analysis* Of Höganäs NC 100.24 Sponge Iron Powder.

Apparent density:	2.40 + 0.10 $g.cm^{-3}$ - 0.05 $g.cm^{-3}$
Flow:	max 32 sec/50g
Green density:	min 6.4 $g.cm^{-3}$

Screen analysis		Chemical analysis	
Tyler mesh	%	Constituent	%
+ 65	0	H_2 - loss	max 0.30
- 65 + 80	max 2	C	max 0.02
- 80 + 100	max 5	SiO_2	max 0.25
- 325	15-25	P	max 0.015
		S	max 0.015

*As supplied by the manufacturer

Table 7.III. Chemical And Size Analyses* Of Höganäs ASC 100.29 Atomised Iron Powder.

Apparent density:		2.95 $g.cm^{-3}$	
Flow:		max 28 sec/50g	
Green Density:		min 6.80 $g.cm^{-3}$	
Screen analysis		Chemical analysis	
Tyler mesh	%	Constituent	%
upper particle size 72	-	H_2 - loss	max 0.15
		C	max 0.01

* As supplied by the manufacturer

hydrogen was also used for preform preheating and annealing purposes. Argon was also used for annealing.

7.1.5. DIES AND PUNCHES

Initial attempts to produce the pellet and powder preforms and hot-forged discs were made using cylindrical die sets of 38mm and 51mm inside diameters of hardened special tool steel. These sets were already available and suitable for initial and some further experimental work. A photograph of the sets are shown in Figs. 7.2. and 7.3

Some of the preforms were also compacted using a die set which was made from USAC tool steel. The tool steel supplied was machined, hardened and ground into die body (57.7mm inside diameter), punch and pressure pads as shown in Fig. 7.4.

Another die set of 63.5 mm inside diameter was also made for use in the 3.985MN (400 tons) hydraulic press. KEA 145 tool steel supplied by Sanderson Kayser Ltd., was machined, hardened and ground as required, shown in Fig. 7.5.

7.2. PREPARATION OF PELLETS

In the experimental programme to investigate the behaviour and properties of forgings made from pellets, the first stage was to prepare pellets from the magnetite super-concentrate and, for comparison, pellets made from reduced iron powder and atomised iron powder.

7.2.1. PREPARATION OF SPONGE IRON PELLETS

Sponge iron pellets were made by tumbling high purity magnetite superconcentrate with an organic binder, adding water

Fig. 7.2. 38mm diameter die set. X0.5

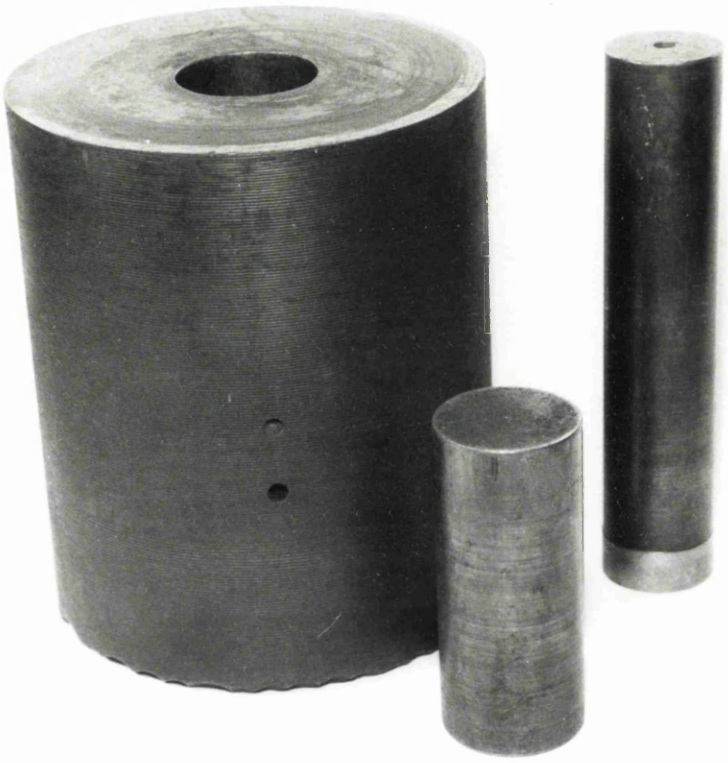


Fig. 7.3. 51mm diameter die set. XO.41

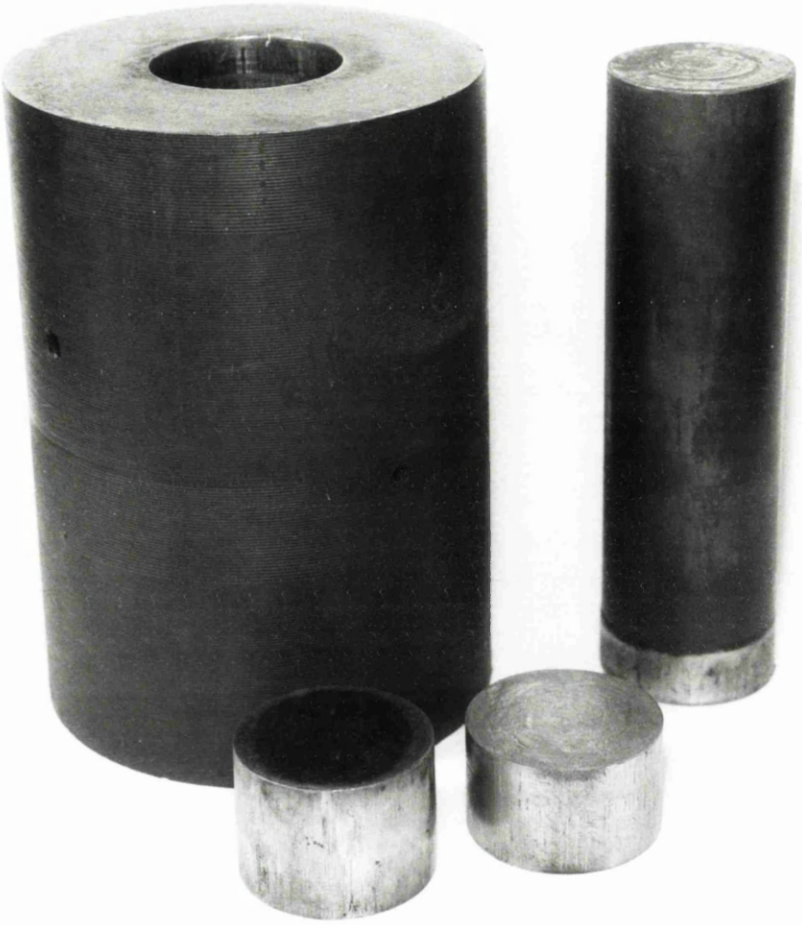


Fig. 7.4. 57.7mm diameter die set. XO.52

Fig. 7.5. 63.5mm diameter die set. XO.5

in a disc pelletiser, drying, and then reducing with hydrogen to form high porosity sponge iron pellets. The sequence of the process is shown in Fig. 7.6 and detailed below.

7.2.1.1. BLENDING

Using a bench-type mixer, see Fig. 7.7 in a number of preliminary trials, the following blending sequence was found to be satisfactory.

The superconcentrate was loaded into the mixer, an addition of 1% Celacol was added and blending carried out for 15 minutes.

7.2.1.2. PELLETISING

The balling of the mixture was carried out using a disc pelletiser, the Erich Pelletiser Type TRO4, as shown in Fig. 7.8. The operating procedure is outlined below.

The inclined pelletising pan, 400mm in diameter, which rotates in a clockwise direction, is flange mounted onto a drive unit. The major parameters controlling the disc output are the disc diameter, the angle of inclination and the speed of rotation. At around 125 rpm pellets of up to 20mm dia. were produced with an angle of inclination of 45^{o}.

1 kg of the blended material was gradually fed into the pan as it was rotating and water in the form of a fine spray was applied. Prevention of build-up of material on the disc and control of the flow pattern of the material was provided by a scraper supplied with the equipment. As the pellets reached acceptable size they were removed manually and fresh material was fed continuously by hand.

The pellets were placed in a steel tray to dry.

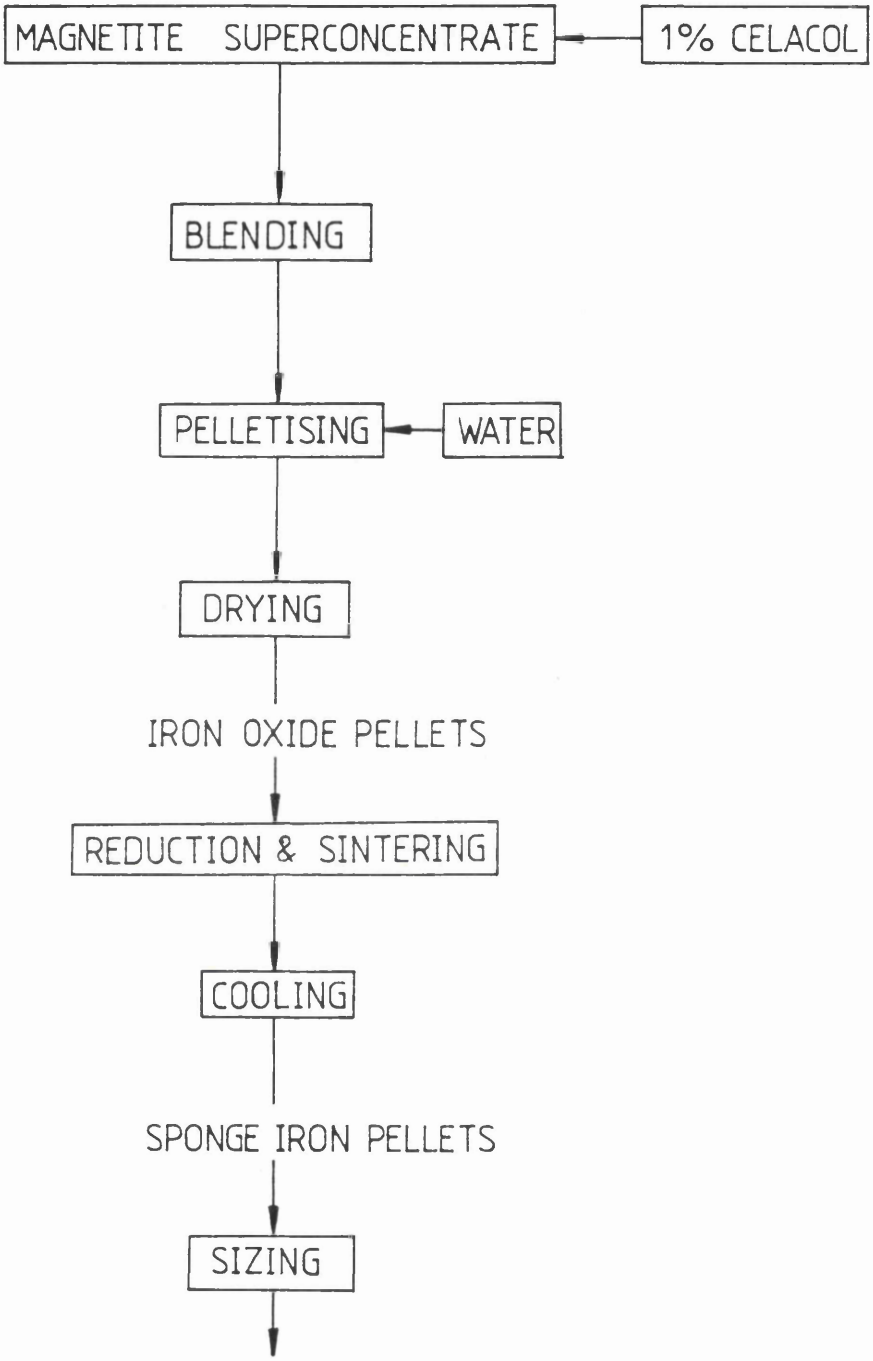


Fig. 7.6. Basic steps in the preparation of sponge iron pellets from magnetite superconcentrate.

Fig. 7.7. Powder mixing unit. XO.14

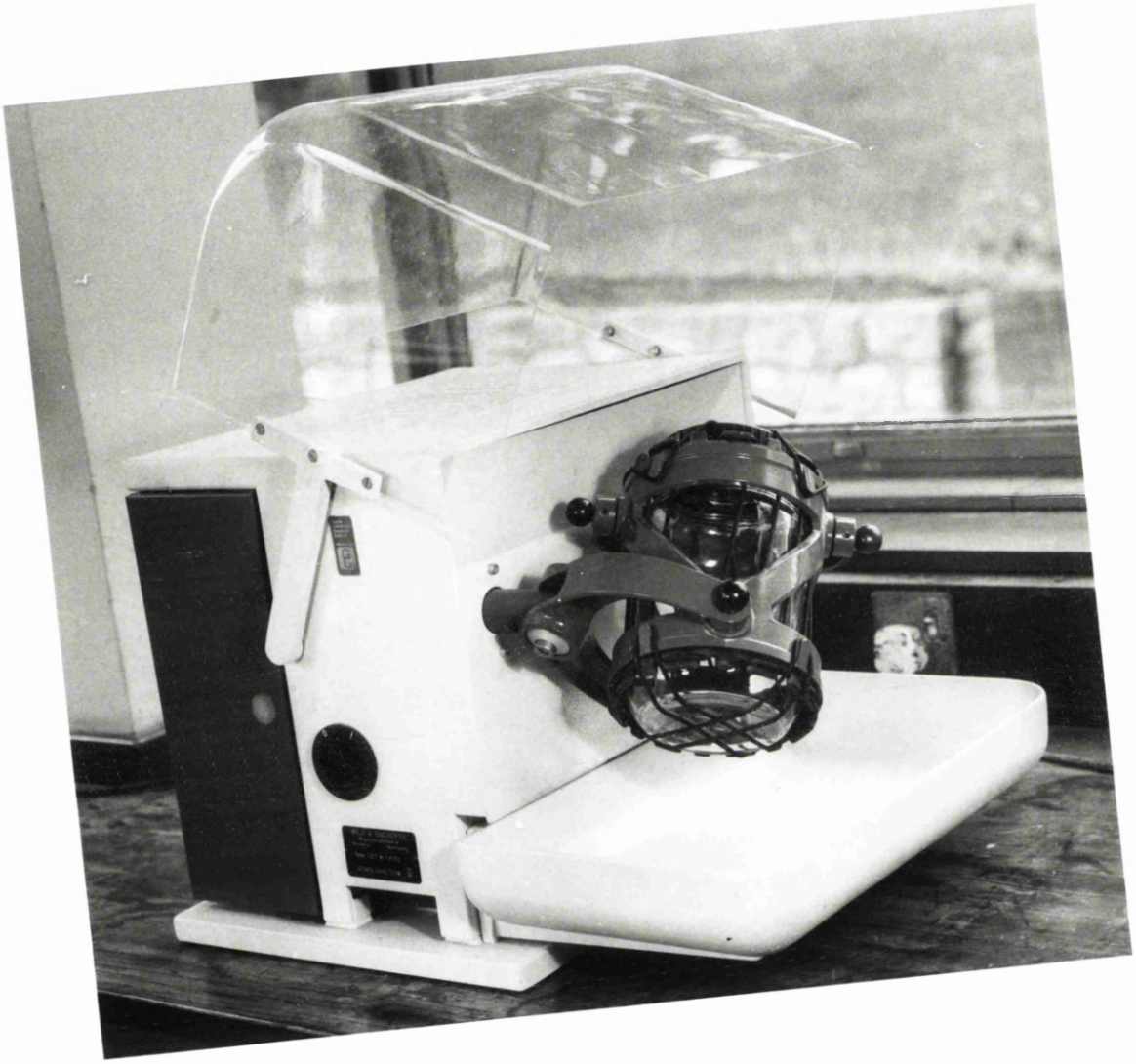


Fig. 7.8. Disc pelletiser. XO.16


ERWEKA
AR 400

7.2.1.3. DRYING

The tray full of wet pellets was placed in an air oven to dry at approximately 125^{o}C for 24 hours and subsequently allowed to cool in air.

7.2.1.4. COMBINED REDUCTION AND SINTERING OF THE MAGNETITE SUPERCONCENTRATE PELLETS

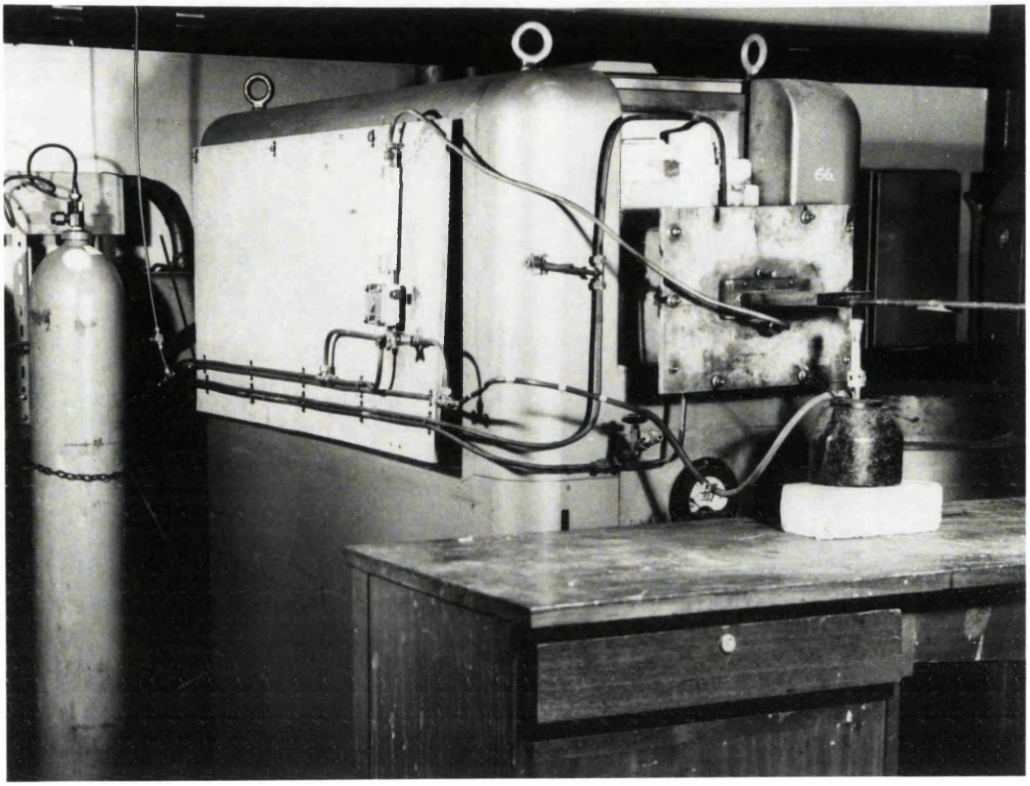
Reduction and sintering of the dried pellets was carried out in a muffle furnace in a hydrogen atmosphere.

The furnace tube consisted of a 10cm dia. Inconel tube, 60cm long, closed at one end, heated by SiC rods giving a 20cm hot zone in which the reduction/sintering was carried out. Welded to the open end of the muffle furnace was a water-cooled chamber. At the closed end gases were supplied via a 0.6cm dia. Nimonic tube, the gases exiting through the cooling chamber to atmosphere.

Experimental procedure was as follows:

The reduction zone of the furnace tube was raised to 1100^{o}C and the furnace tube was flushed with Nitrogen-Hydrogen mixture gas with 4% hydrogen for approximately 5 minutes. Pure hydrogen was then passed into the muffle at a flow rate of 3.77 L/min. The pellets, contained in a perforated Nimonic tray, were then carefully introduced through the cooling chamber and into the hot zone of the muffle. A reduction/sintering time of 2 hours was allowed. The tray was then transferred into the cooling chamber and cooled for 15 minutes, while maintaining the flow of hydrogen. Finally the tray was removed from the cooling chamber to complete the cooling in air. A photograph of the reduction furnace is shown in Fig. 7.9.

Fig. 7.9. The equipment used for combined reduction and sintering. X0.06


66.

7.2.2. PREPARATION OF IRON POWDER PELLETS

As received Höganäs NC100.24 sponge iron powder and Höganäs ASC100.29 atomised iron powder were also pelletised in the same manner as the superconcentrate pellets. The flow chart for the preparation of iron powder pellets is given in Fig.10. The only difference was that the combined reduction and sintering time was lowered to 1 hour. The reason for this was that the iron powder only had a relatively thin oxide surface film requiring a short reduction time compared to that for the superconcentrate.

7.2.3. PELLET SIZING

After combined reduction and sintering, strong pellets were obtained which were sized into different fractions for the purpose of :

(i) general use,

(ii) to determine the effect of pellet size on properties of forgings.

For general use, the pellets were prepared in size up to 16mm diameter and then sized into 5-14mm in diameter.

For determination of the effect of pellet size on properties, the materials were pelletised up to 20mm dia. and then screened into three different fractions:

(a) up to 8mm

(b) 8-14mm

(c) 14-20mm

7.3. PREPARATION OF PELLET AND POWDER PREFORMS

In P/M forging one of the methods adopted to obtain full density involves the preparation of a porous preform. This

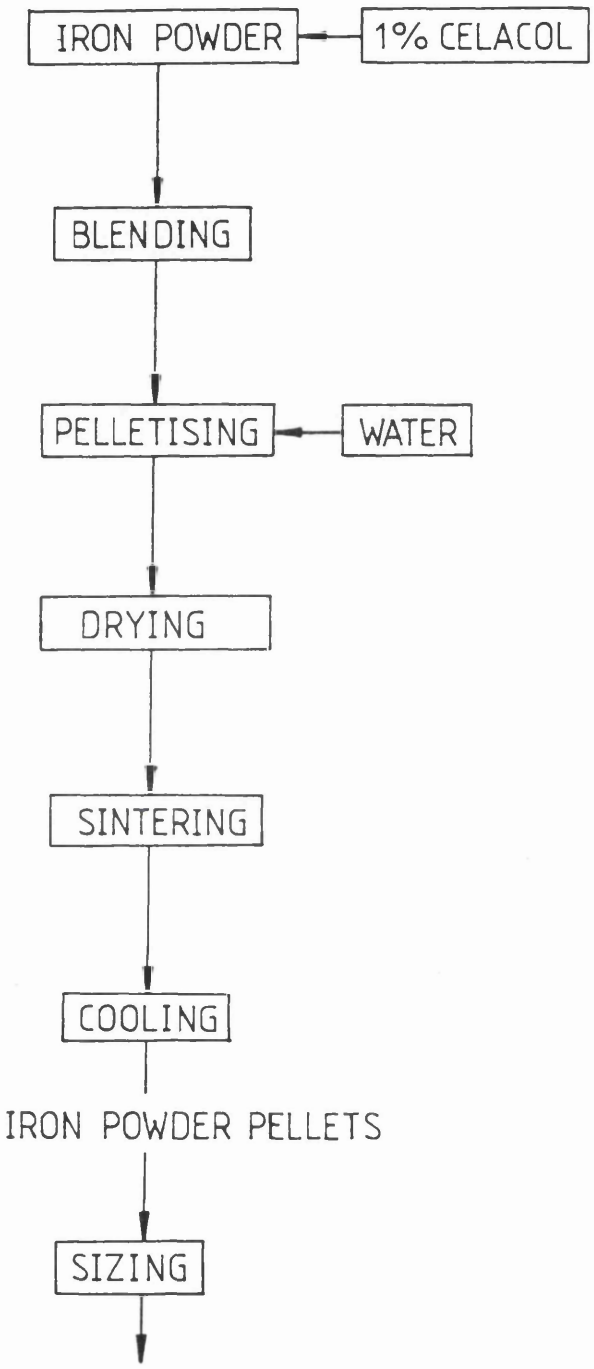


Fig. 7.10. Basic steps in the preparation of iron powder pellets from NC 100.24 or ASC 100.29.

route was followed in this work and the investigation therefore examined the effect on the final forgings made from the various pellets of preform density, pellet size, flow during hot compaction, hot compaction pressure and annealing atmosphere.

7.3.1. EQUIPMENT

Most of the preform making was carried out by using the 1.07 MN (110 tons) single action hydraulic press with a maximum speed of 50.8 $mm.s^{-1}$. The speed and the load could be adjusted within the above limits. The general view of the hydraulic press is shown in Fig. 7.11.

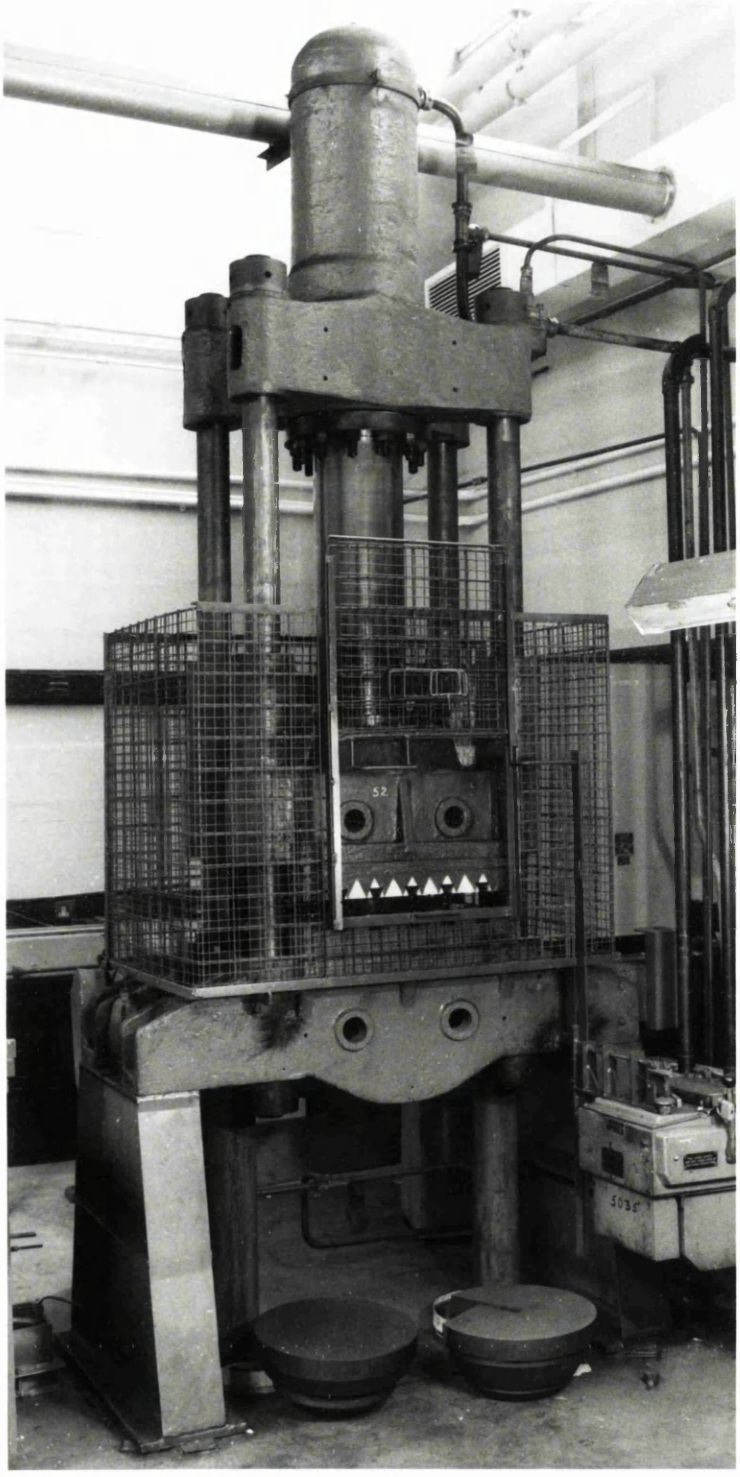
The confined die sets used were in 38mm, 51mm and 57.7mm inside diameter of hardened tool steel with punches and pressure pads, as shown in Figs. 7.2, 7.3 and 7.4.

Operational procedure used was as follows:

The main die body was placed on the lower platform of the press and the first pressure pad inserted into the die cavity. Then pellets or powder was put in, levelled and the second pressure pad fitted on top, followed by the punch. Pressure was then applied as required, released and the die body raised on two steel blocks by hand to allow the compact to be ejected. Pressure was applied again to eject the compacted preform. Visual examination and dimensional measurement then took place.

However, further examination showed that the hot forged products contained small amounts of residual porosity. The results also indicated that it was necessary to use a minimum

Fig. 7.11. 1.07 MN hydraulic press. X0.05



of 926MNm^{-2} pressure to eliminate porosity (the press mentioned above was capable of producing only 525MNm^{-2} in 51mm dia. die). Also to obtain Izod impact test specimens, forgings larger than 51mm diameter were needed and it was necessary to use a larger capacity press to produce these larger preforms and forgings with full density. For the highest density preforms, another single-acting hydraulic press with a capacity of 3.985MN (400 tons) was used following the above operational procedure.

7.3.2. PRELIMINARY INVESTIGATION

In the experimental programme, it was initially decided to find out the feasibility of the novel pellet forging process. Thus preforms were prepared from:

(i) sponge iron pellets
(ii) NC 100-24 iron powder pellets, and
(iii) NC 100-24 iron powder.

In the meantime attempts were made to analyse the effect of the major process parameters on the properties of forgings. The parameters investigated were :

(i) Type of deformation
    (a) Closed upsetting (limited flow)
    (b) Repressing (no flow)
(ii) Preform density.

All final hot forgings were compacted in a 51mm diameter cylindrical closed die set in this part of the study. Therefore, preforms of 38mm diameter were prepared to give limited flow during hot deformation and preforms of 51mm diameter were produced for repressing. 51mm diameter preforms were then machined to 49mm dia. to allow for swelling (and expansion)

during heating before hot forging.

The amount of material (pellets or powder) was always 160g and average pellet size was 10mm in diameter.

Preform density was varied in the range 50-85% theoretical. The pellets could produce preforms with a density as low as 50% theoretical and still have acceptable strength. This applied particularly to the sponge iron pellets which have a high inherent porosity and densities in the range of 1.7 - 1.9 gcm^{-3}. Pellet preforms (38mm and 51mm in diameter) were cold pressed to densities of 50%, 70% and 85% theoretical without using any die wall lubrication.

160g of pellets for each compact was filled into a confined cylindrical die. The pressure was adjusted to give the required density. After ejection from the die the density was checked by measuring the height of the compact using a micrometer.

The same procedure was applied to NC100.24 iron powder, but it was difficult to make low density preforms because of the low green strength. Therefore only preforms of 70% and 85% theoretical density were prepared.

It was then necessary to machine the 51mm diameter preforms on a lathe taking 2mm off the diameter. Apart from the 50% density NC100-24 iron powder pellet preforms, all the others withstood the machining operation satisfactorily.

7.3.3. FURTHER STUDIES

After analysing the preliminary work, it was decided to investigate the effect of additional process parameters on

the properties of the finished products while varying the other parameters used before. The new parameters that were studied are given below:

(i) pellet size,

(ii) sintering time (preform sintering before hot forging),

(iii) annealing atmosphere,

(iv) hot compaction pressure.

7.3.3.1. PELLET SIZE

To investigate the effect of pellet size, three different size fractions were obtained by screening and preforms were produced varying the other parameters as before. The size fractions were:

(i) $<$ 8mm

(ii) 8-14mm, and

(iii) 14-20mm.

Using the same equipment as before, a series of preforms were produced using the above three sizes of pellets and cold compacted under varying conditions. The full experimental programme is shown in Fig. 7.12.

7.3.3.2. SINTERING TIME

Although in powder preform forging, preforms are directly hot forged after they are cold preformed, sintering of the preforms before hot forging is also used as one of the powder preform forging techniques.

As mentioned in the previous sections pelletised iron ore concentrates or iron powders were reduced and sintered to form strong individual pellets. The amount of sintered

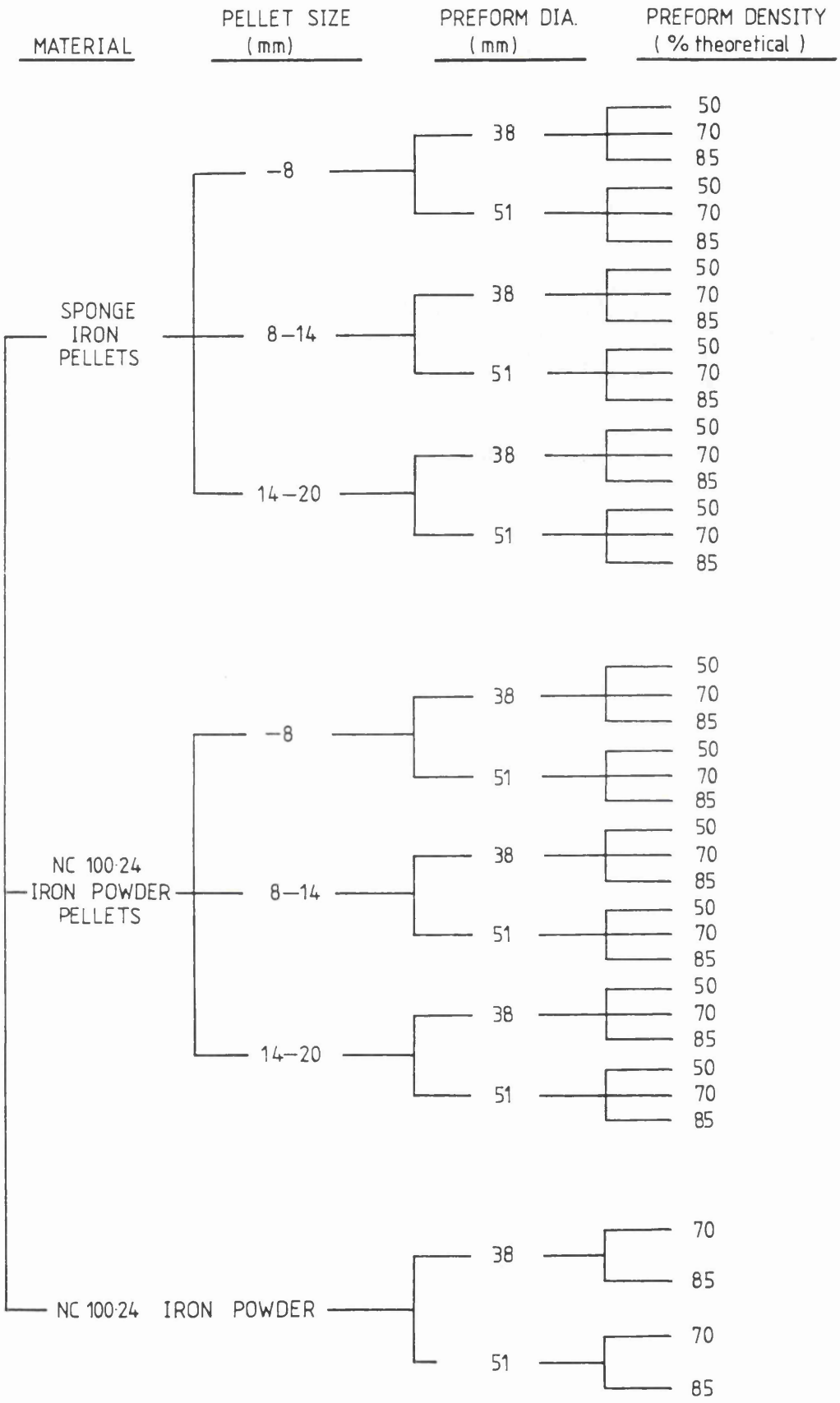


Fig. 7.12. Statistical experimental programme.

material in the preforms was consequently high prior to hot forging compared to iron powder preforms. Therefore it was planned to investigate the effect of sintering prior to hot forging, and a comparison was made between NC 100.24 iron powder and sponge iron pellets.

Four preforms, each 160g, from both materials were compacted to 70% theoretical density using the 38mm diameter die, sintered for 1,2 or 3 hours at 1100^{o}C in dry hydrogen and cooled. The fourth specimen was not sintered. Pre-heating and hot forging then took place using the 51mm diameter die. The mechanical properties of the finished discs were examined and compared according to sintering time.

7.3.3.3. ANNEALING ATMOSPHERE

Generally one mechanical test specimen from each final forging disc was annealed in dry hydrogen at 700^{o}C for 1 hour. Some of the mechanical test results showed that higher strength and lower elongation were obtained with this annealing treatment compared to the as-forged condition. Initially it was thought that this effect was due to hydrogen atmosphere used. Later a series of experiments were carried out to find the effect of annealing atmosphere by using argon and vacuum.

Preforms for this part of the investigation were made by using again:

(i) sponge iron pellets,

(ii) NC 100.24 iron powder pellets, and

(iii) NC 100.24 iron powder.

The same experimental procedure as explained in Section 7.3.2. was followed to prepare these preforms.

7.3.3.4. HOT COMPACTION PRESSURE

The density of the final forged product is basically dependent on the hot forging pressure in most of the P/M work. The pressure necessary for hot forging a given material is dependent on hot forging temperature and type of forging.

Using a pressure of $525MNm^{-2}$ in the preliminary work gave final forging densities close to but less than the theoretical density of the material. The force of the press was limited to 1.06MN, and an experiment was conducted to determine the optimum hot forging pressure for comparatively impure sponge iron pellets. Therefore, a 38mm dia. die set, a temperature of $1100^{o}C$ and 70% theoretical density of 38mm diameter preforms were planned to be used for hot repressing. 110g of sponge iron pellets for each preform was used (instead of 160g as before used) to provide as uniform a pressure distribution as possible during hot forging. 16 sponge iron pellet preforms were compacted to 70% theoretical density applying pressure of $340MNm^{-2}$ in 38mm dia. die without a lubricant. These preforms were then hot forged using four different pressures. The forged densities were then measured and compared to mechanical properties.

However the results showed that the higher the forging pressure the lower was the final porosity and it was found that it was essential to use a minimum hot forging pressure of $926MNm^{-2}$ to produce full density forgings. In addition to this it was necessary to have impact test specimens which

requires a final forging bigger than 51mm in diameter. The results also indicated that pellet size and the type of deformation used had a small effect on the final properties. The process variables which affected the properties most were preform density and annealing treatment. Therefore another set of experiments was designed to make forgings at full density using a 3.985MN single-acting hydraulic press made available by Imperial College, London.

The preforms for this part of the study were pre-compacted again from:

(i) sponge iron pellets
(ii) NC100.24 iron powder pellets, and
(iii) NC100.24 iron powder.

Preform density and annealing treatment were considered as the process variables for hot-repressing. Thus the preforms were made in a 57.7mm diameter cylindrical die (Fig. 7.4) to be hot-repressed in a 63.5mm diameter (Fig.7.5). 400g of material was used for each preform to give a height to diameter ratio similar to previously designed preforms. Six pellet preforms varying in density between 49% - 96% theoretical density and four iron powder preforms varying in density between 61% - 89% theoretical density were prepared.

Preforms were compacted using the 1.07MN press, applying the same operational procedure as before, except for the highest density preform of each material. These were prepared using a 3.985MN press in a 63.5mm dia. die (Fig. 7.5) but these preforms were then machined to give 57.7mm diameter preforms.

7.3.3.5. ATOMISED IRON POWDER AND PELLET PREFORMS

ASC 100.29 atomised iron powder pellets and ASC 100.29 iron powder preforms were prepared in a similar way to the preforms prepared in the preliminary investigation. The difference was that only preforms for hot-repressing were prepared.

7.4. HOT FORGING OF PREFORMS

The preforms obtained after cold compaction were preheated and subsequently hot forged to densify the preforms. The reheating of such a material must be carried out in a protective atmosphere to avoid internal oxidation, so preforms were reheated in a muffle furnace under a hydrogen atmosphere. One end of the reheating chamber was closed, while the other end had an extended zone projecting outside the furnace, as shown in Fig. 7.13. The reheating (preheating) chamber contained a flat plate as a base for the preforms. The preforms were preheated at 1200^{o}C for 20 minutes before hot forging; assuming a 100^{o}C fall in temperature during transfer of the hot preform into the die cavity, the temperature of the preform at impact was 1100^{o}C.

Most of the hot forgings were produced on the 1.07MN single-acting hydraulic press having a maximum speed of 5.08cm.s^{-1} which was described previously and shown in Fig.7.11. Most of the preforms were forged in a cylindrical die set of 51mm diameter die cavity, shown in Fig. 7.3., and a maximum force of 1.07MN was given in a single blow. The standard procedure used for hot forging was as follows:

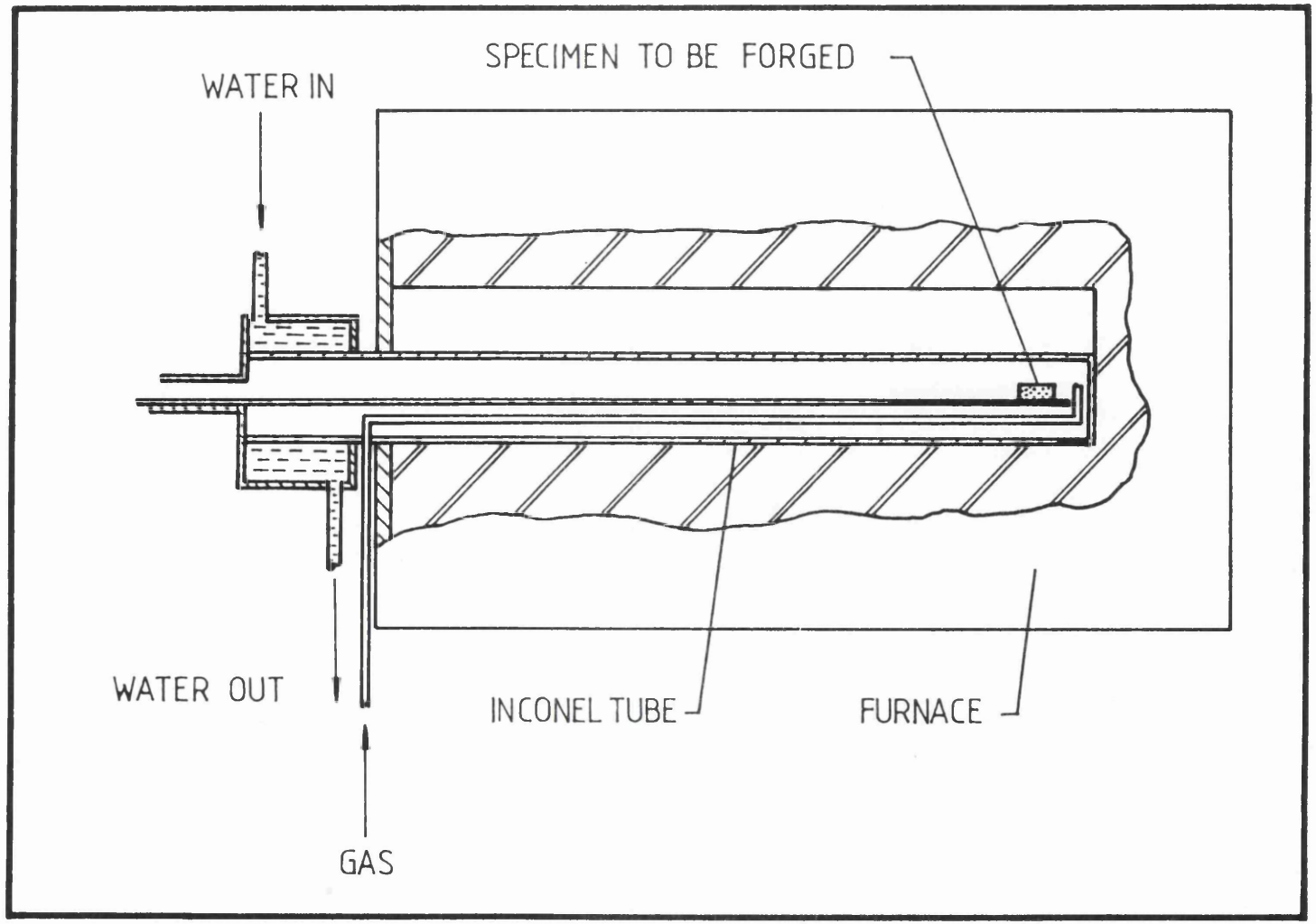


Fig. 7.13. Preform preheating furnace.

(i) The die body was placed on the lower platform of the hydraulic press. The pressure pad was inserted into the die hole and the top of the pressure pad was covered with asbestos sheeting to minimise heat loss during hot forging.

(ii) The reheating furnace was placed in front of the hydraulic press so that the extended exit end was very close to the die set. The preform then placed on a flat plate was inserted into the hot zone of the furnace.

(iii) The press was set to its maximum pressure of $525MNm^{-2}$

(iv) Using a hooked wire the heated preform was pulled from the hot zone of the furnace, placed into the die cavity and the upper punch inserted with asbestos paper on top of it. These procedures were carried out in 2-3 seconds. Immediately the press was actuated to compress the preform in a single blow.

(v) The press was raised so that the die body could be supported on two steel bars of equal thickness, spaced so that when pressure was again applied the forging was ejected. The forging was then air-cooled.

Some of the preforms, as mentioned in Section 7.3.3.4, were hot forged on another single-acting hydraulic press which had a capacity of 3.985MN. This press could undergo speed changes during compacting in order to allow for an escape of trapped air. It had very accurate pressure control, because the pump short-stroked itself as soon as the preset pressure was reached.

The operational procedure with this press was the same as before, with the following differences. The punch was

fixed on the upper ram and the die body was inserted into the lower main body which rested on the bed of the press, and moved vertically to assist ejecting the forged disc. This main die body was also heated at constant temperature of 300^{o}C.

7.5. ANNEALING

Annealing consisted of heating the specimens at 700^{o}C in a muffle furnace in a hydrogen atmosphere for 1 hour.

In order to investigate the effect of annealing atmosphere on properties, some specimens were annealed in an argon atmosphere at 700^{o}C in a muffle furnace for 1 hour and some specimens in vacuum for 1 hour at 700^{o}C at pressure of 0.075 Torr.

7.6. METHODS OF TESTING AND INSPECTION

7.6.1. DENSITY MEASUREMENT

The density of the preforms were calculated from weight and volume measurements. The densities of the hot forged materials were determined by the immersion technique, distilled water being the immersion fluid. The results were checked by calculation using weight and volume of the carefully prepared test specimens. For this, rectangular specimens (27 x 21 x8)mm^{3} were machined from forged discs, the surfaces being finely ground.

7.6.2. MECHANICAL TESTING

The U.T.S, yield stress, and percentage elongation of the hot forged discs were determined using a Houndsfield Tensometer at room temperature. The specimens used were machined and polished from discs (Fig. 9.1).

Izod impact test specimens were prepared from the specially made forged discs. Single V-notch square section specimens were machined and polished according to BS 131 (Fig. 9.1). Testing was carried out at room temperature.

7.6.3. METALLOGRAPHY

The structures of the materials at the different stages of processing were examined under optical and scanning-electron microscopes.

7.6.3.1. OPTICAL MICROSCOPY

The sponge iron pellets, iron powder pellets, and all preforms were soft and porous. It was therefore necessary to impregnate the pores with some material. A mixture of Metaserve Resin 'SW' and hardener in the proportions 98.75 : 1.25 by volume was used for this purpose. Standard preparation for metallographic examination of the dense forgings was carried out. A mixture of Resin 'FT' and hardener in the proportion 97.5 : 2.5 was used for mounting the specimens in a plastic mount.

It became clear that the most important structural characteristic of the sponge iron pellet forgings was the presence of a large amount of fine non-metallic inclusions distributed throughout the matrix which could be dragged out during mechanical polishing, unless proper care was taken. The following precautions were therefore taken in order to reduce this tendency:

(i) A special nylon cloth was used for polishing, and diamond paste was used as an abrasive.

(ii) A heavy load was applied to the specimens during polishing. This was accomplished by holding the mounted specimen in one spot on the wheel while oscillating it slowly about its own vertical axis.

(iii) Fine polishing time was kept to a minimum because the degree of preservation of the inclusions deteriorated significantly with long polishing times. Some compromise was necessary, between freedom from polishing scratches and the preservation of inclusions.

Etching of the polished high inclusion content specimens was difficult. Since the etching action was more rapid around the inclusions, a long etching time caused the inclusions to appear larger than they were. Therefore a compromise was reached between revealing all the grains and the preferential attack of the etchant around the inclusions. 2% Nital solution was used as an etchant in all cases.

7.6.3.2. SCANNING ELECTRON MICROSCOPY

S.E.M. observations were made to examine the fracture surface of the hot compacted material and the non-metallic inclusions in the matrix material. The specimens were coated with gold before observing them under the microscope.

7.6.4. EVALUATION OF INCLUSIONS

Quantitative parameters related to the inclusions present in the forgings were determined as follows:

(i) Total volume percentage of the inclusions.

(ii) Total number of inclusions.

(iii) Size distribution of the inclusions.

The Quantitative Television Microscope (QTM) manufactured by Metals Research Ltd. was used for determining the above parameters for the unetched specimens.

The QTM equipment measured the total area of the non-metallic inclusions in (picture points)^2 which was converted into an area percentage. The area percentage of the inclusions was assumed to be equal to the volume percentage.[99] From a sampling point of view, the lower the magnification, the better the sample. Consequently, it was desirable to use a low power objective. The use of a low power objective, however, presents a problem since the very small inclusions are not detected. As a compromise an objective of x63 was generally used throughout the investigation and objectives of x32 and x100 were used to check the results. In all cases about 70 measurements were taken, and the data were statistically analysed. The conditions under which the specimens were examined on the QTM are shown in Table 7.IV.

7.6.5. GRAIN SIZE MEASUREMENTS

The grain size and distribution were determined according to the mean linear intercept method, sometimes referred to as the Heyn intercept, which defines the average chord length intersected by the grains on a random straight line in the planar polished and etched section.[100]

A specimen of 3mm thickness was cut from the radius of the forged disc, half of this then being mounted for examination as representative of the whole specimen, followed by polishing and etching.

Table 7.IV. Experimental Conditions Used In Measuring Various Parameters On The QTM.

Details	Conditions
Specimen	Unetched
Illumination	Incident
Objective	x 63
Prism lens	x 6.3
Area of the television screen	500,000 $(p.p)^2$
Calibration factor at the used magnification	1 p.p. = 0.24μm
Resolution	0.24μm
Field area	0.0288 mm^2
Electronic resolution setting	Maximum
No. of fields per specimen	~ 70
Parameters measured on each field:	
(1) Area of inclusions	In $(p.p)^2$
(2) Total number of inclusions	>1 p.p. across the forging direction
(3) Size distribution:	
(a) No. of inclusions greater than	1 p.p. across the F.D.
(b) " " " " "	2 " " "
(c) " " " " "	5 " " "
(d) " " " " "	10 " " "
(e) " " " " "	15 " " "
(f) " " " " "	25 " " "
(g) " " " " "	40 " " "
(h) " " " " "	60 " " "

QTM was used to measure the grain size and distribution. The equipment was capable of moving the specimen step by step in two directions and the step size was adjusted as required. The point which was to be investigated could be rotated on its own axis.

The specimen surface was divided into 65 imaginary sections. Three points were taken from each section and also each point was rotated four times (45^{o}) on its axis. More than 20 intercepts were counted manually on the television screen from each reading and the data were statistically analysed.

7.6.6. SURFACE QUALITY

The last forged materials were visually and macroscopically examined for edge and surface cracks. Surface finish and the oxidation on the surface were also noted.

CHAPTER 8

PELLETISATION, THE COMBINED REDUCTION AND SINTERING, AND PREFORMING BEHAVIOUR OF SPONGE IRON PELLETS

The first step in making pellet forgings from magnetite superconcentrate is to make the oxide pellets. This was done by mixing oxide concentrate with a suitable organic binder and pelletising this mixture in a disc pelletiser. The second step is to reduce the oxide pellets to metal. This was done by H_2 reduction at a high temperature which allowed the necessary sintering to take place. The final step is to cold compact the pellets to various preform densities and sizes.

In this chapter the results obtained are discussed under the following headings:

(i) pelletisation of organically bonded magnetite superconcentrate pellets

(ii) the combined reduction/sintering of magnetite superconcentrate pellets, and

(iii) preforming behaviour of sponge iron pellets.

8.1. PELLETISATION OF ORGANICALLY BONDED MAGNETITE SUPERCONCENTRATE PELLETS

Organically bonded magnetite superconcentrate pellets were made according to the procedure outlined in Section 7.2. The materials, equipment and process parameters were chosen following the experience gained during the development work on pellet production for the Pellet Rolling Project at this University.

The composition of the pellet is important. It has been suggested[101] that only high purity superconcentrate iron oxide (>99%) should be used and that no impurities should be introduced during pelletisation. The analysis of the magnetite superconcentrate used in the present case is given in Table 7.1.

A binder is essential as the pellets are then more easily handled, but it is advantageous to use organic binders as these decompose during the reduction stage leaving no residue or only a carbonaceous one. For the highest ductility, organic binders should be free from sulphur, although for machining purposes some sulphur may be advantageous in some cases. Typical binders are phenolic resins, furfural derivatives, molasses, cellulose and starch derivatives and large numbers of gums and adhesives. Siliceous binders such as bentonite are generally to be avoided because of the introduction of undesirable impurities. In the experimental work on the direct route from iron oxide superconcentrate to strip by Singer[82] it was shown that cellulose-based binder, Celacol M450, could successfully be used. On the other hand, recent development work on pelletisation of iron ore superconcentrates[80] has shown that pellets bonded with cellulose, phenol or sugar performed poorly during direct-reduction in a rotary kiln. Bitumen bonded pellets, after baking treatment, were successfully reduced in the rotary kiln, avoiding breakdown to powder and sticking to the retort walls.

In the present work, however, Celacol M450 was used as a binder. This has a very low ash content and a charring temperature of 330°C approximately. As a result, using the

equipment and materials mentioned above, pelletisation was successfully carried out and gave strong enough oxide pellets to carry out the next processing step, in which the oxide pellets were reduced in a static base furnace.

8.2. THE COMBINED REDUCTION AND SINTERING OF MAGNETITE SUPERCONCENTRATE PELLETS

Having demonstrated the possibility of obtaining organically bonded iron ore pellets with enough strength to handle, the next step was to reduce the oxide pellets to metal. This was most conveniently done by chemical reduction with H_2 at a high temperature. At the temperature employed sintering between the reduced particles also takes place.

It was not the main object of the present investigation to make an extensive study of the reduction and sintering behaviour of magnetite superconcentrate pellets. However, during the course of the pellet reduction stage close observations were made to ensure that the oxide pellets were reduced completely. A fixed temperature, H_2 flow rate and time were selected and used because of the following reasons.

It was shown that the rate of reduction increases sharply with temperature.[2] Dube[2] investigated the reduction rate of a magnetite superconcentrate strip of similar chemical composition to the magnetite superconcentrate pellets used at various temperatures using H_2 as a reductant. The reduction rate at 1000°C was found to be very low as compared with that at 1100°C and 1200°C, and the results indicated that the magnetite superconcentrate strip was completely reduced in 30 min. at 1200°C and in about 75 min. at 1100°C. The special

furnace used in this work was capable of attaining temperatures in excess of 1100°C, but in order to reduce the possibility of damage to the elements all reduction and sintering was done at 1100°C.

The reduction of magnetite superconcentrate pellets by hydrogen requires the transfer of hydrogen through the boundary layer towards the core and the transfer of an equal number of moles of water vapour away from the surface. The thickness of the boundary layer surrounding the pellets depends mainly upon the temperature, the flow rate of gas and on the geometry of the material to be reduced. It was found that the reduction rate of iron ore increased with an increase in gas flow rate, keeping all other variables constant.[102-104] Eventually, a situation was reached where any further increase in gas flow rate did not have any effect on the rate of reduction. The lowest flow rate above which there is no significant increase in the rate of reduction at a given temperature is called the 'critical flow rate'. The establishment of a critical flow rate ensures that the bulk gas stream is not deficient in the reducing gas as a result of the concentration of the reaction product, approaching equilibrium in the gas boundary layer.

Pocovi et al[105] established that the critical flow rate for hydrogen reducing a magnetite superconcentrate disc, having a similar composition to the one used in this work, at 1200°C was about 2 l/min. which was equivalent to a linear flow velocity past the specimen of 0.067 m/s, as against a flow rate of 3.77 l/min. giving a flow velocity of 0.008 m/s used in the present case. It was not possible to pass the reducing gas over the pellets at a velocity approaching the

critical flow rate in the experimental equipment used. Therefore, the time necessary for complete reduction in the present investigation was longer than would be necessary using a furnace specially designed for the purpose. Moreover, the reducing gas was not evenly distributed over the pellets in the apparatus used.(see Fig. 7.13.) The pellets were contained on a tray and the reducing gas was delivered near to one end; therefore, the reducing gas did not flow readily over the bottom layer of the pellets. However, it was found that a combined reduction and sintering time of 120 min. was sufficient to reduce the magnetite superconcentrate pellets completely. These pellets are called sponge iron pellets. It is probable that the time required for complete reduction could be further reduced by designing a special pellet container and gas delivery system, which would allow the gas to be distributed uniformly all over the pellets.

For the iron powder pellets, which were partly oxidised during pelletisation, a temperature of 1100°C, a gas flow rate of 3.77 l/min., and reduction and sintering time of 60 mins. were used. It was found that the iron powder pellets were completely reduced and sintered.

Fig. 8.1 shows the iron powder as received and sponge iron pellets and iron powder pellets after combined reduction and sintering. It can be seen that some cracking of the pellet surfaces has occurred. This was probably due to swelling of the pellets during the process.

Microscopic examination of the cross-section of the sponge iron pellets and the iron powder pellets showed that there was

Fig. 8.1. (a) NC 100.24 iron powder,
(b) sponge iron pellets and
(c) NC 100.24 iron powder pellets.

a b c

no magnetite present in the reduced pellets, (see Figs. 8.2 and 8.3.) It can be seen in Fig. 8.2 that the sponge iron pellets are highly porous and contain two types of porosity. These are small porosity inside the particles (internal) and larger porosity between the particles (interparticle). It must be noted that the 'particles' mentioned above are, in fact, not individual particles; the particles are already sintered and interconnected during the reduction/sintering operation.

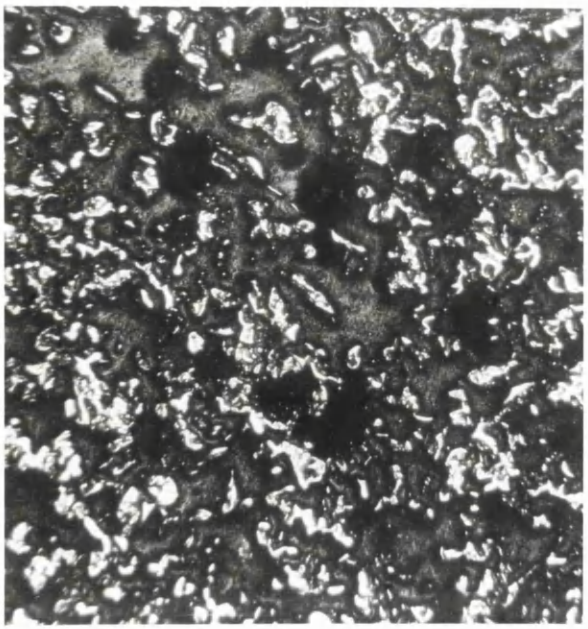
Dube[2] observed that the internal pores in sponge iron particles were small in size and large in number. These pores were generated principally during the wüstite $\longrightarrow$ iron reduction due to the specific volume difference between wüstite and iron. Although the iron powder pellet showed a similar structure to that of the sponge iron pellet, the total volume of the pores was less. (see Fig. 8.3.) Density measurements also substantiated this difference; sponge iron pellets averaging 1.85 $g.cm^{-3}$ and iron powder pellets averaging 2.38 $g.cm^{-3}$.

The strength of the pellets was not investigated specifically but completely reduced and sintered pellets were sufficiently strong that no problems were encountered in the later stages of the investigation.

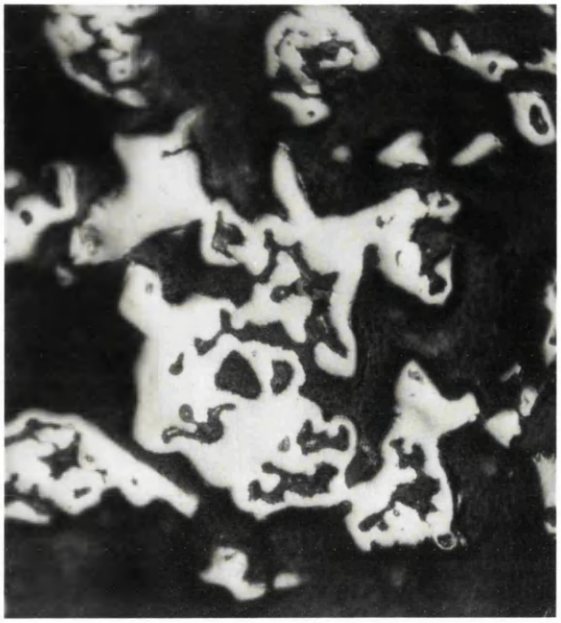
Some problems had been anticipated with pellets sticking together or to the tray and although both occurred in the majority of cases, parting was effected with ease. In a small number of cases the bond was so strong that the pellets were damaged when separated and these were discarded.

Fig. 8.2. Microstructure of a sponge iron pellet.
(a) X60
(b) X300

Fig. 8.3. Microstructure of a NC 100.24 iron powder pellet
(a) X60,
(b) X300



a



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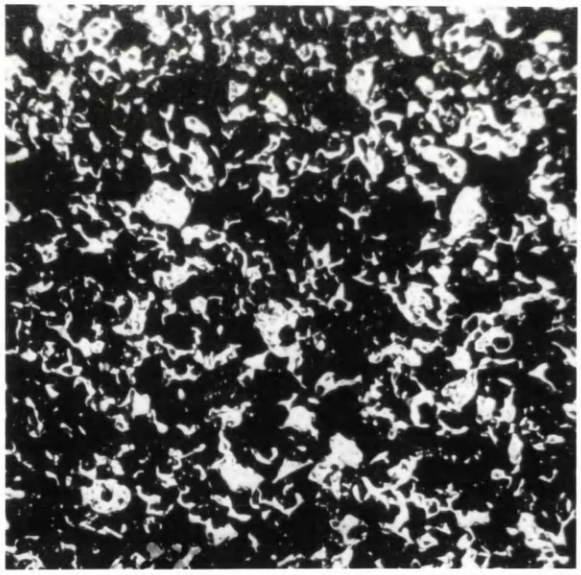

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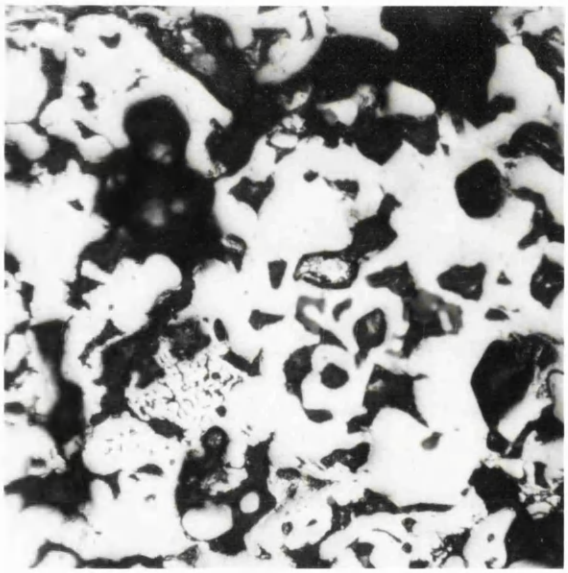


b

8.3. PREFORMING BEHAVIOUR OF SPONGE IRON PELLETS

The manufacture of the forging preform is based on powder metallurgy technology. The principal techniques used for making iron and steel P/M forging preforms are cold die-compacting and isostatic compacting. In this investigation cold die-compacting in a closed die was used. The preforms were designed to give final forgingsof cylindrical discs and the same amount of pellets or powder was used for each forging in each set of experiments. Preforms for closed upsetting (limited flow) and for repressing (with no flow) were pressed without lubricant.

As reviewed in Section 3.3, preform design was based on a consideration of preform shape and size (having decided on the type of deformation) with the aim of ensuring that final hot forging is crack-free. The most suitable shape for the preform, similar to the final forging, was a disc. Since there are no well established data for designing preform dimensions, it was thought that the height to diameter ratio, H/D, should be kept as small as possible to give as uniform a stress distribution as possible during hot forging. Height to diameter ratio was the only dimensional parameter to be considered for the preforms during repressing because there was no lateral flow involved in this case. As mentioned above, an equal weight of material was used for each preform in each set of experiments and various preform densities were used. Height to diameter ratio was also varied from 0.56 for lowest density preformsto 0.24 for highest density preforms.

The height to diameter ratio for the closed upsetting (limited flow) preforms was varied between 0.95 for the

Fig. 8.4. (a) and (b) NC 100.24 iron powder pellet preforms and (c) and (d) sponge iron pellet preforms. XO.6.

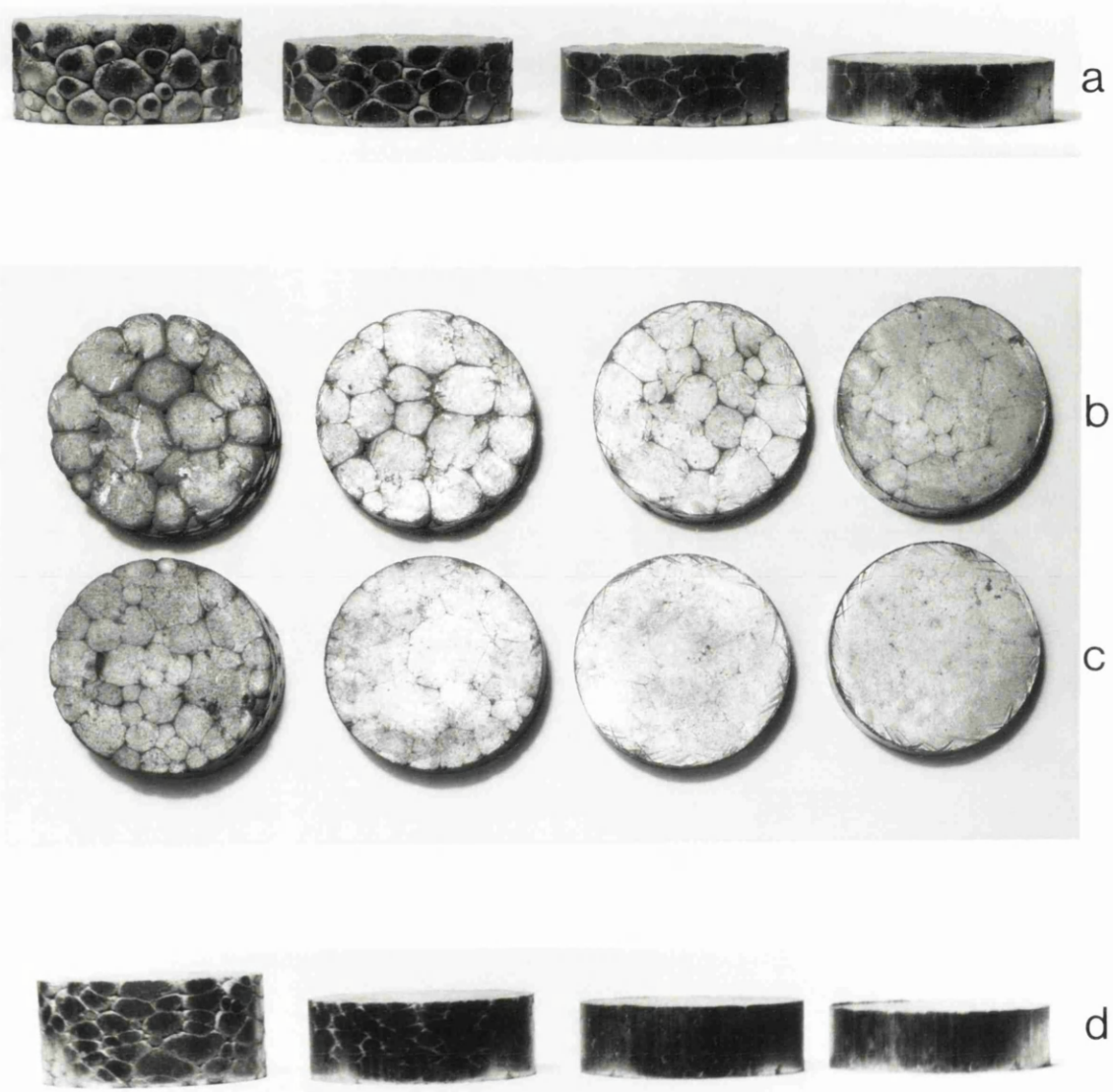
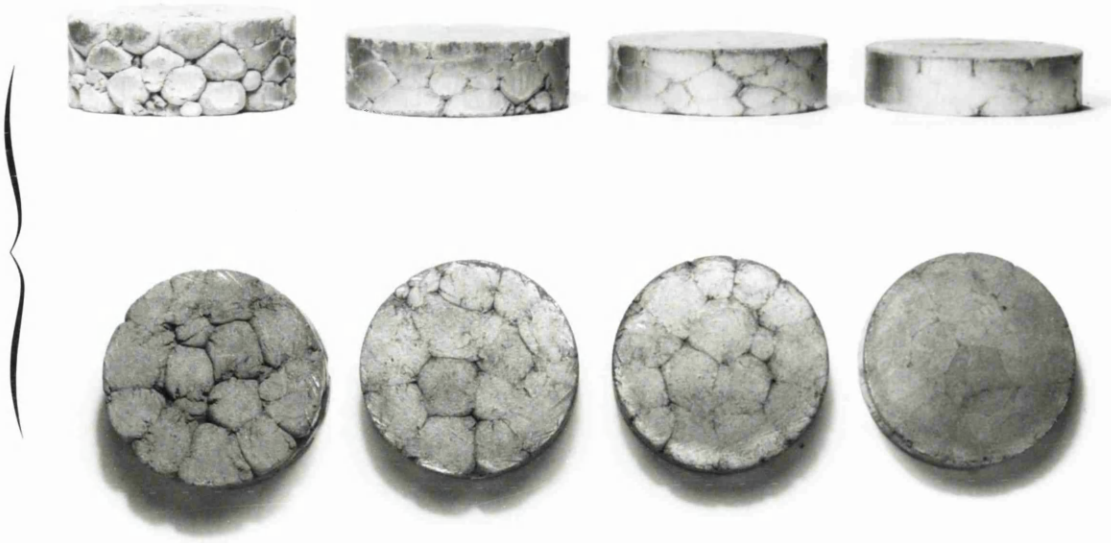
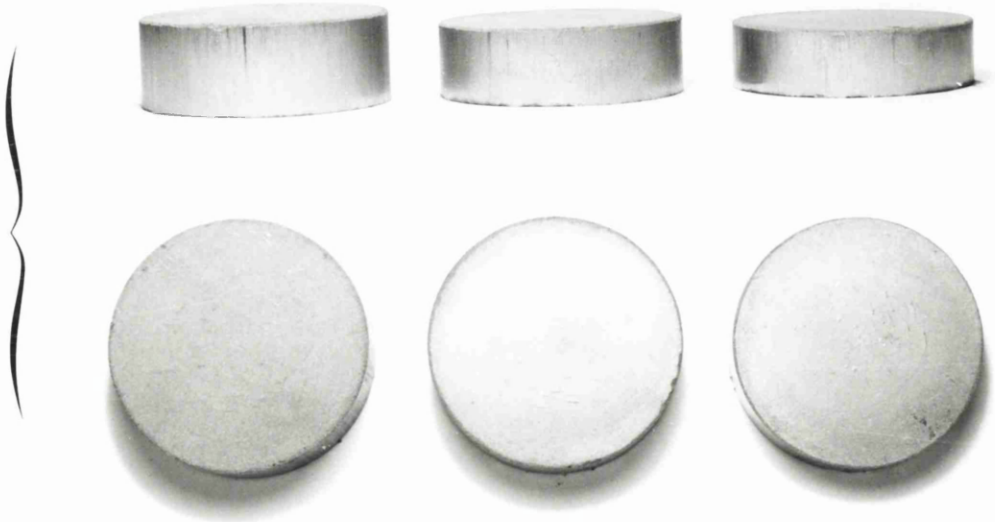

a
b
c
d

Fig. 8.5. ASC 100.29 atomised iron powder pellet preforms. X0.55

Fig. 8.6. NC 100.24 iron powder preforms. X0.6





lowest density preforms to 0.53 for the highest density preforms. In addition to height to diameter ratio for the closed upsetting preforms, the diameter ratio between the preform and the final forging, Do/D, was also considered. According to Guest et al[52] the critical dia. ratio, Do/D, is $\sim$ 1.4. When this ratio increases radial cracks start to form. However, a Do/D ratio of 1.34 was used in the present investigation and no radial cracks were observed in the final hot-forged discs.

An important feature of the present process is the large amount of plastic deformation carried out during the hot forging operation, which reduces the overall volume of the pellet preform. As a result, porosity level is reduced, especially between the pellets. By ensuring that the pellet preform is of low density and retains a spongy structure, large amounts of plastic deformation can be incorporated during closure of the porosity. High porosity sponge iron pellets resulted in preforms with as low a density as 50% of theoretical and yet which were still strong enough to handle and machine. Preforms were then prepared with densities varying between 50% to 96% of theoretical. Fig. 8.4 shows the NC 100.24 iron powder pellet preforms and sponge iron pellet preforms of densities 50%, 60%, 70% and 85% of theoretical. Figs. 8.5 and 8.6 show the ASC 100.29 atomised iron powder pellet preforms of densities 50%, 60%, 70% and 85% of theoretical and NC 100.24 iron powder preforms of densities 60%, 70% and 85% of theoretical.

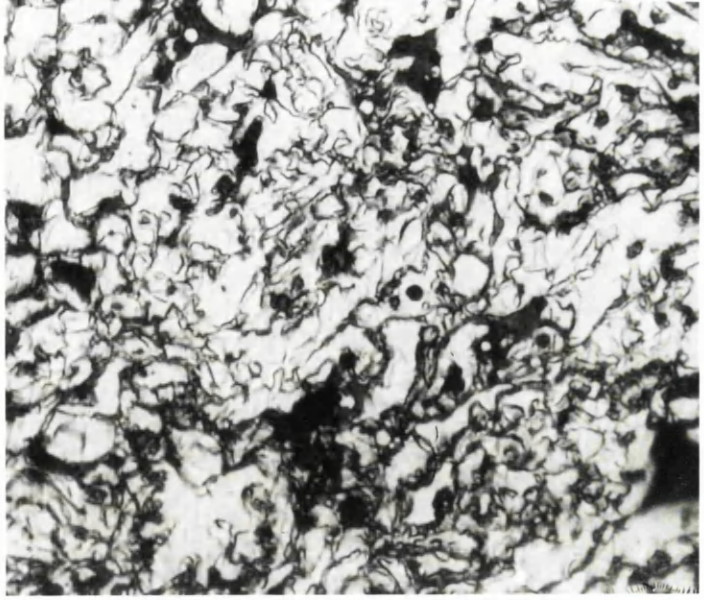
Fig. 8.4 shows that the sponge iron pellet preforms are deformed more than iron powder pellet preforms. As the

preform density increased for the sponge iron pellet preforms the pellet boundaries disappeared while the iron powder pellet preforms revealed the pellet boundaries even at 85% density. This latter effect can be seen also with atomised iron powder pellet preforms, (see Fig. 8.5.). This shows the high porosity level of the sponge iron pellets and demonstrates that even for the lowest density preforms the sponge iron pellets are strongly bonded.

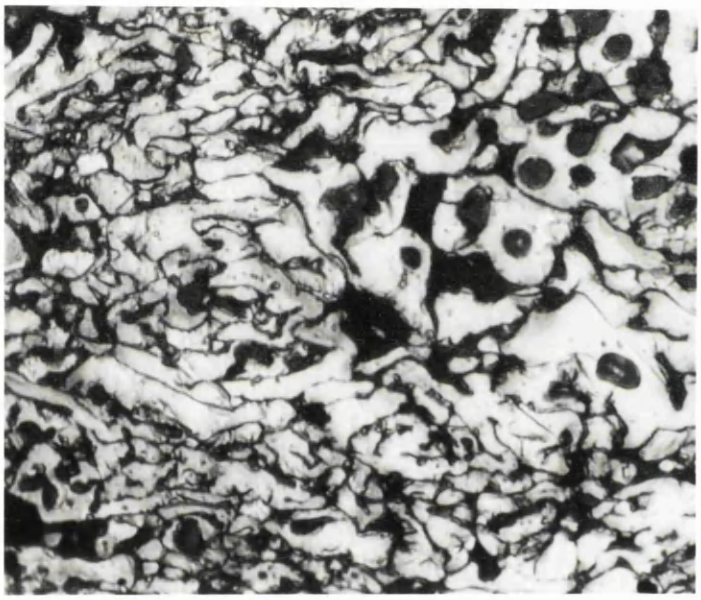
During machining, the sponge iron pellet preforms performed without any significant difficulties, but both iron powder pellet preforms, especially those of 50% density, started to break at the edges and in some cases they disintegrated. Figs. 8.4 and 8.5 show that 50% density preforms in general have weak bonds, with some pellet preforms even splitting when ejected from the die. Iron powder preforms (Fig. 8.6) also showed similar behaviour during machining. The lowest density iron powder preform, 60%, was not machined easily unless very thin passes were used. From these observations it is clear that significantly low density preforms can be produced from porous sponge iron pellets.

Microscopic examinationsof cross-sections of preforms were made to study the effect of deformation on porosity. Fig. 8.7 shows the sponge iron pellet preforms at 51%, 72% and 83% of theoretical density. 51% dense preforms show no apparent spreading of particles, although the pellets are deformed substantially, also can be seen in Fig. 8.4. Inter-particle porosity is reduced as well as interpellet porosity. This would be explained by the fact that very little plastic deformation occurs and the pellets and particles are merely

Fig. 8.7. Microstructures of sponge iron pellet preforms of
(a) 50%,
(b) 70%,
(c) 85% theoretical density.
Centre, X560



a



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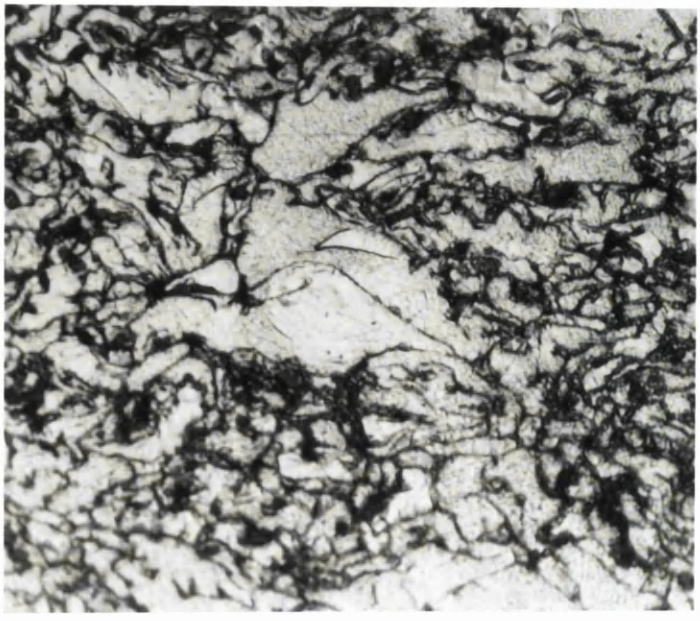


c

rearranged. As the preform density is increased,(see Fig. 8.7 (b)) the particles start to flatten and undergo plastic deformation together with the closure of pores. But still some large voids are to be seen in the micrograph, which shows the centre of the preform where deformation is low. As the preform density increases further deformation continues and all the large voids disappear. (see Fig. 4.7(c)) Iron powder pellets behave in a similar manner but apparent deformation starts at a higher preform density compared to sponge iron pellet preforms. (see Fig. 8.8.) The iron powder preform of 86% theoretical density (Fig. 8.9) still does not show any significant particle flattening.

The above metallographic examination, however, indicates that the high porosity sponge iron pellets undergo a high degree of deformation during cold compacting even in the low pressure centre region. Also, sponge iron pellets indicated better die filling than the one from iron powder pellets.

It has been shown by several investigators[106-109] that the densification process of metal powders involves three steps:

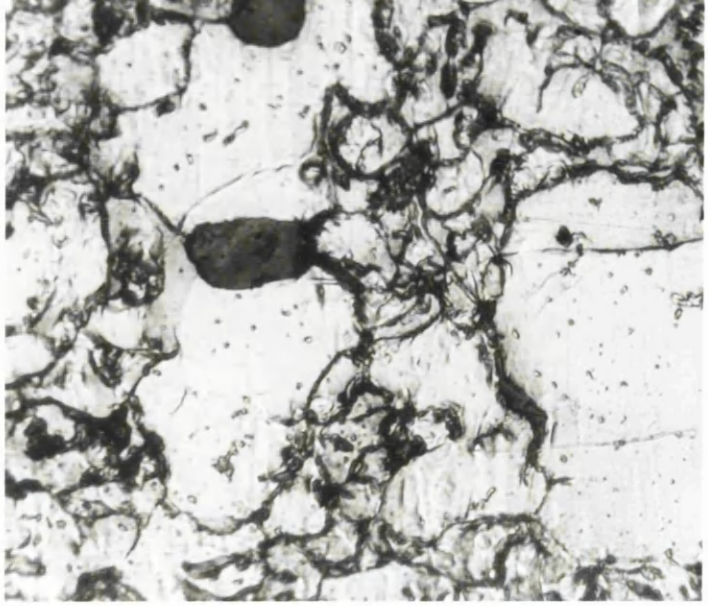
(i) Bulk movement of individual particles at relatively low pressures.

(ii) Rapid densification with the accompanying extensive plastic deformation of individual particles.

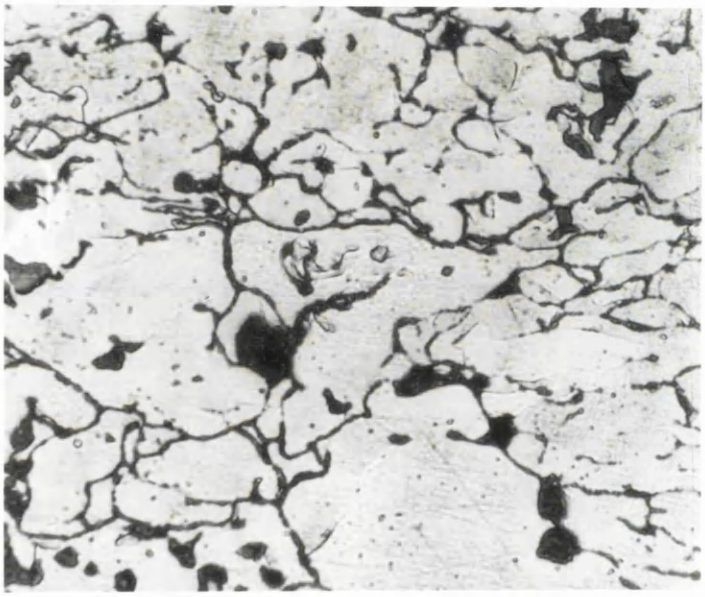
(iii) Higher levels of deformation producing little further increase in density.

Generally increasing the compaction pressure will result in increased plastic deformation and concomitant work hardening

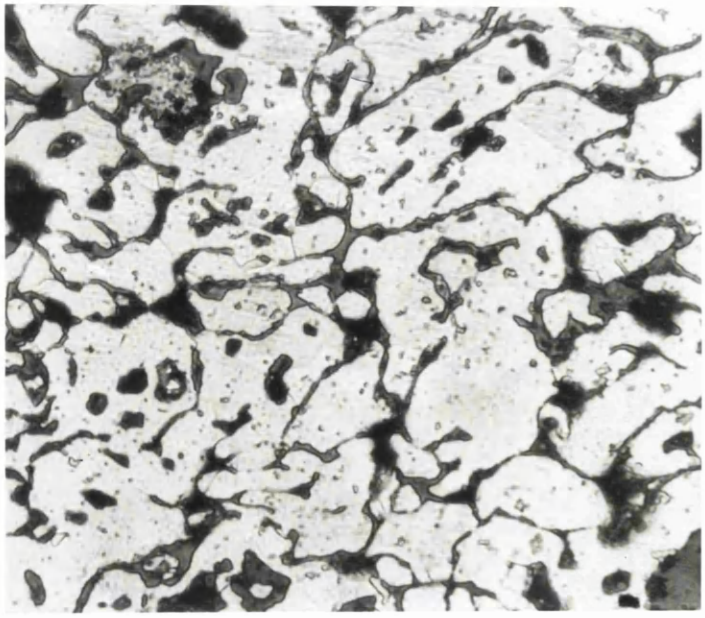
Fig. 8.8. Microstructures of NC 100.24 iron powder pellet preforms of
(a) 50%,
(b) 70%,
(c) 85% theoretical density.
Centre, X560



a

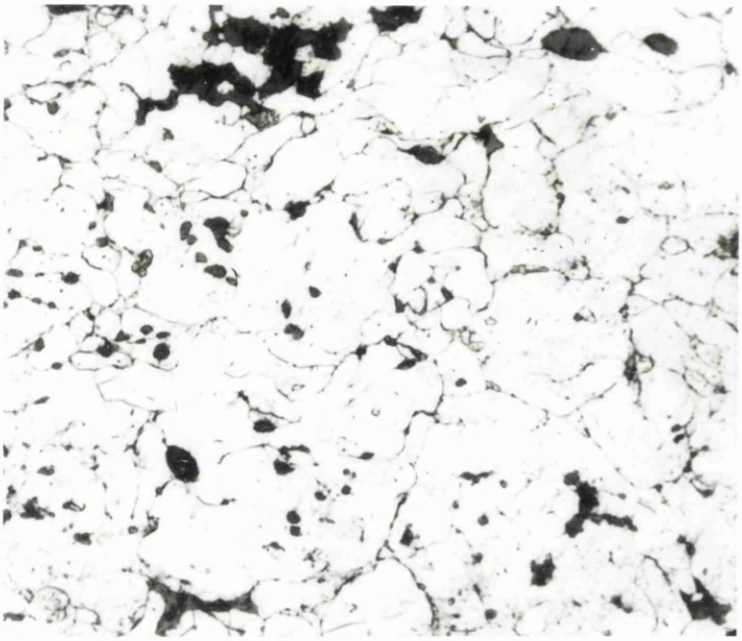


b



c

Fig. 8.9. Microstructure of NC 100.24 iron powder preform of 86% theoretical density. Centre, X560



of the individual particles. Hirschhorn and Garey[106] determined the plasticity by measuring the microhardness of individual particles resulting from work hardening during plastic deformation of several types of iron powder, and found that plastic deformation starts very early in the compaction process. In addition, the metallurgical structure of individual particles, such as impurities and grain size, will also influence their plastic deformation. According to Hirschhorn and Garey,[106] electrolytic iron powders exhibited the greatest densification, while reduced powders showed the least. They pointed out that there is a general trend for increased hardness and percent increase in hardness after compaction with increasing impurity content.

Fig. 8.10 shows graphically the densification behaviour with preforming pressure for the various pellets and powders used in this investigation, showing that, for a given compacting pressure, atomised iron powder pellets achieve the highest densification and sponge iron powder pellets the lowest.

Bockstiegel[110] showed that the large pores between the particles of the iron powder compact shrank much faster with increasing compacting pressure than the small pores inside the particles. The change of interparticle porosity with increasing compacting pressure is the same whether the powder particles are porous or not when reduced iron powder and practically pore-free electrolytic iron powders are compared. Bockstiegel[106] also advanced the possible mechanism that the large pores between the particles and the small pores inside the particles both shrink due to deformation only, but

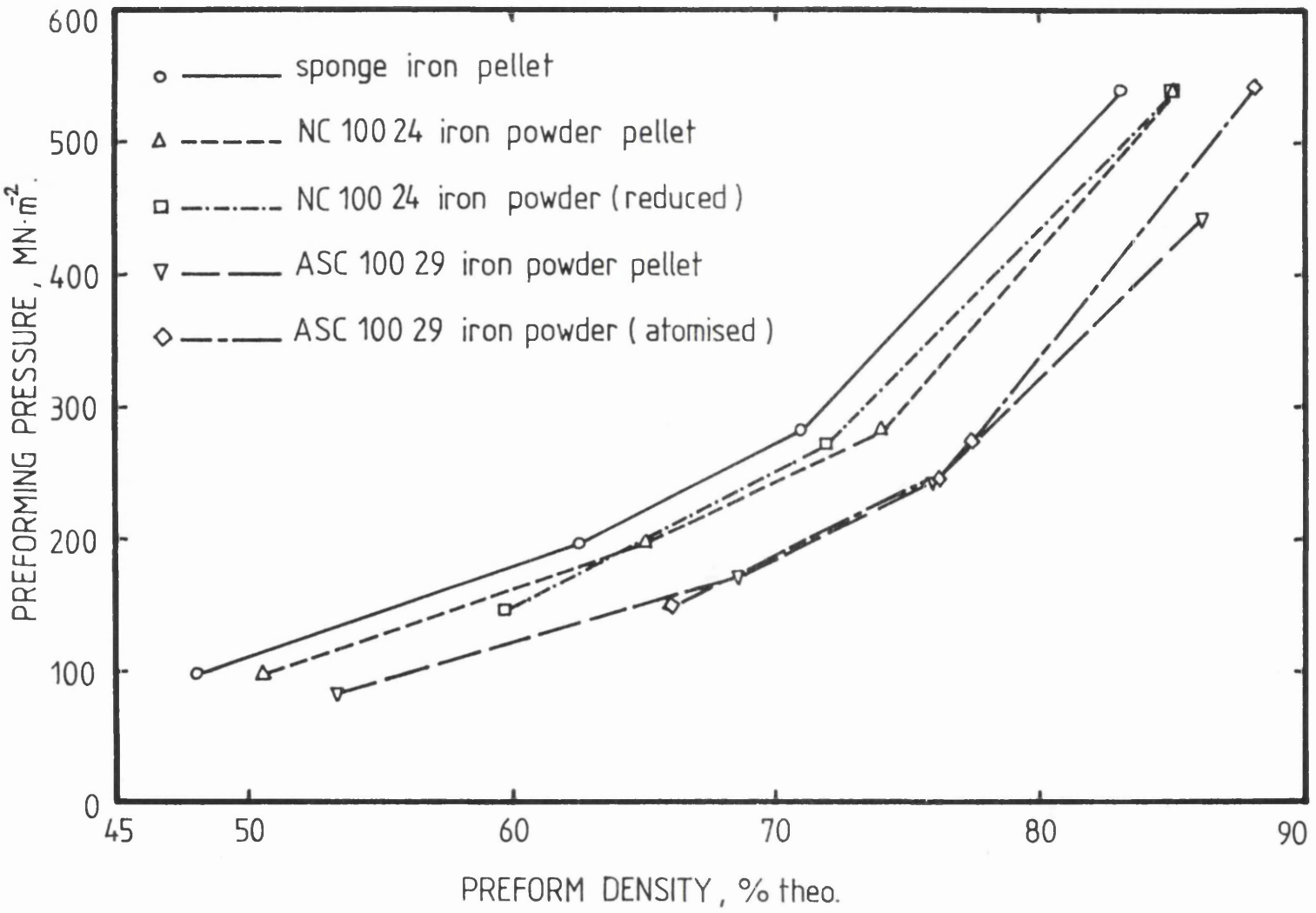


Fig. 8.10. Densification behaviour with preforming pressure for the various pellets and powders used.

added that large pores close at a very early stage of the deformation process, not only through deformation but also through particle rearrangement, while the small pores inside the particles shrink through deformation only.

In the present case, the sponge iron pellets, iron powders and their pellets contain different levels of porosity and impurities. In particular the sponge iron pellets and the NC 100.24 iron powder pellets already contain two types of porosity before compaction, as mentioned earlier. In addition, these pellet compacts contain a third type of porosity which consists of very large pores between the pellets. Therefore, high porosity sponge iron pellets deform earlier than less porous iron powder pellets, and this early start of deformation results in higher strain hardening. Because of this and the large amount of inclusions present in the sponge iron pellets, a higher pressure is required for a given density as compared to the other materials used. (see Fig. 8.10.)

In brief, however, sponge iron pellets require higher compacting pressure than iron powders or iron powder pellets for a given preform density since:

(i) More deformation is necessary because of the very low apparent density.

(ii) There is a higher impurity content.

CHAPTER 9

THE HOT FORGING OF SPONGE IRON PELLET PREFORMS

As the process is novel, little was known of the effect of the process variables on the properties of the forged product. Therefore a preliminary set of experiments was carried out to determine whether the new route gave a product which had mechanical properties at least equal to the equivalent powder forged product. Evaluation of the preliminary experiments showed that the following process variables should be studied in depth to obtain an adequate evaluation of the process:

(i) Deformation type

(ii) Preform density

(iii) Pellet size

(iv) Annealing treatment

(v) Annealing atmosphere

(vi) Hot-forging pressure.

The results were interpreted by reference to the mechanical properties and the micro-structures. In this chapter the results obtained are interpreted in terms of mechanical properties under the appropriate headings, and the micro-structure will be dealt with in the next chapter.

9.1. PRELIMINARY INVESTIGATION

In this initial work, the preforms were produced from:

(i) sponge iron pellets

(ii) NC 100.24 iron powder pellets

(iii) NC 100.24 iron powder.

The preforms were cold compacted to a range of initial

densities, then preheated for 20 minutes and subsequently hot forged at 1100°C to produce forged discs. Each forged disc was cut in half and one half was annealed for 1 hour at 700°C in H_2; the procedure is outlined in detail in Section 7.4.

Tensile test pieces were then machined from each disc (see Fig. 9.1) and tested in both the as-forged and the annealed conditions. The results obtained are shown in table 9.I and Figs. 9.2-9.5.

Generally no cracks developed in the hot forged discs from either the repressed (51 mm dia.) preforms or the limited flow (38 mm dia.) preforms. Cracks did develop when a 38 mm dia. preform was placed off-centre in the 51 mm dia. final hot forging die cavity. Cracks occurred along the side of the preform furthest from the die wall. In open die forging the basic process is the compression of a workpiece between flat dies. As the die closes, barrelling of the workpiece occurs because of friction between the die surfaces and the workpiece ends, and chilling of the workpiece ends. This inhibits the free lateral spread of the material under deformation and causes bulging in the middle. As the process continues tensile stresses build up on the bulging surface until rupturing begins, resulting in edge cracks. This is the mechanism which caused cracking of the 38 mm dia. preform hot forged off-centre in the die. During hot deformation the preform started to flow radially, but before the side of the preform, furthest from the die wall, reached the die wall the deformation was completed. Therefore the cracks formed in this side of the forging were present in

Fig. 9.1. A typical hot forged disc and mechanical test pieces machined from a hot forged disc. X 1.55

Table 9.I. Mechanical Properties Of Forgings.

Material	Preform Dia. (mm)	Preform Density (% theo.)	Condition*	Elongation (%)	Y.S. ($MN.m^{-2}$)	U.T.S. ($MN.m^{-2}$)
Sponge Iron Pellets	38	52	F	19	353	398
			A	20	369	468
		75	F	23	334	392
			A	29	356	419
		84	F	22	237	348
			A	24	249	350
	51	53	F	21	356	409
			A	28	349	457
		72	F	20	307	357
			A	23	327	431
		81	F	21	270	331
			A	29	286	333
NC 100.24 Iron Powder Pellets	38	52	F	18	414	461
			A	19	424	487
		70	F	21	348	433
			A	23	330	445
		82	F	30	254	321
			A	30	239	318
	51	52	F	17	396	449
			A	17	382	477
		76	F	19	313	366
			A	22	299	380
		85	F	26	236	299
			A	30	236	309
NC 100.24 Iron Powder	38	70	F	23	212	297
			A	29	214	304
		86	F	26	207	278
			A	33	208	290
	51	74	F	23	297	371
			A	24	260	358
		86	F	30	223	286
			A	37	237	304

* F: as-forged, A: annealed at 700°C for 1 hour.

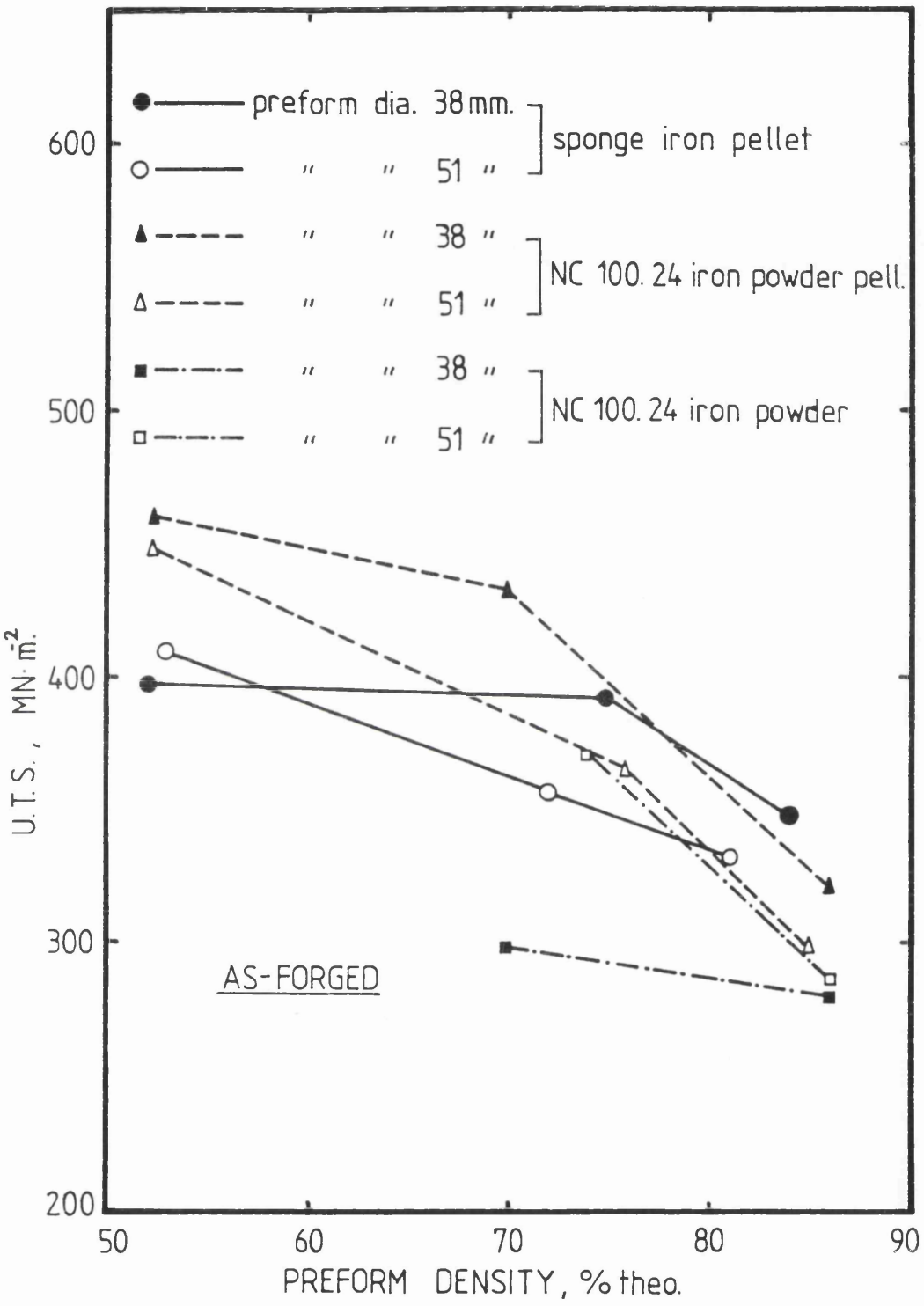


Fig. 9.2. Effect of preform density and preform diameter on the U.T.S. of the forgings in as-forged condition.

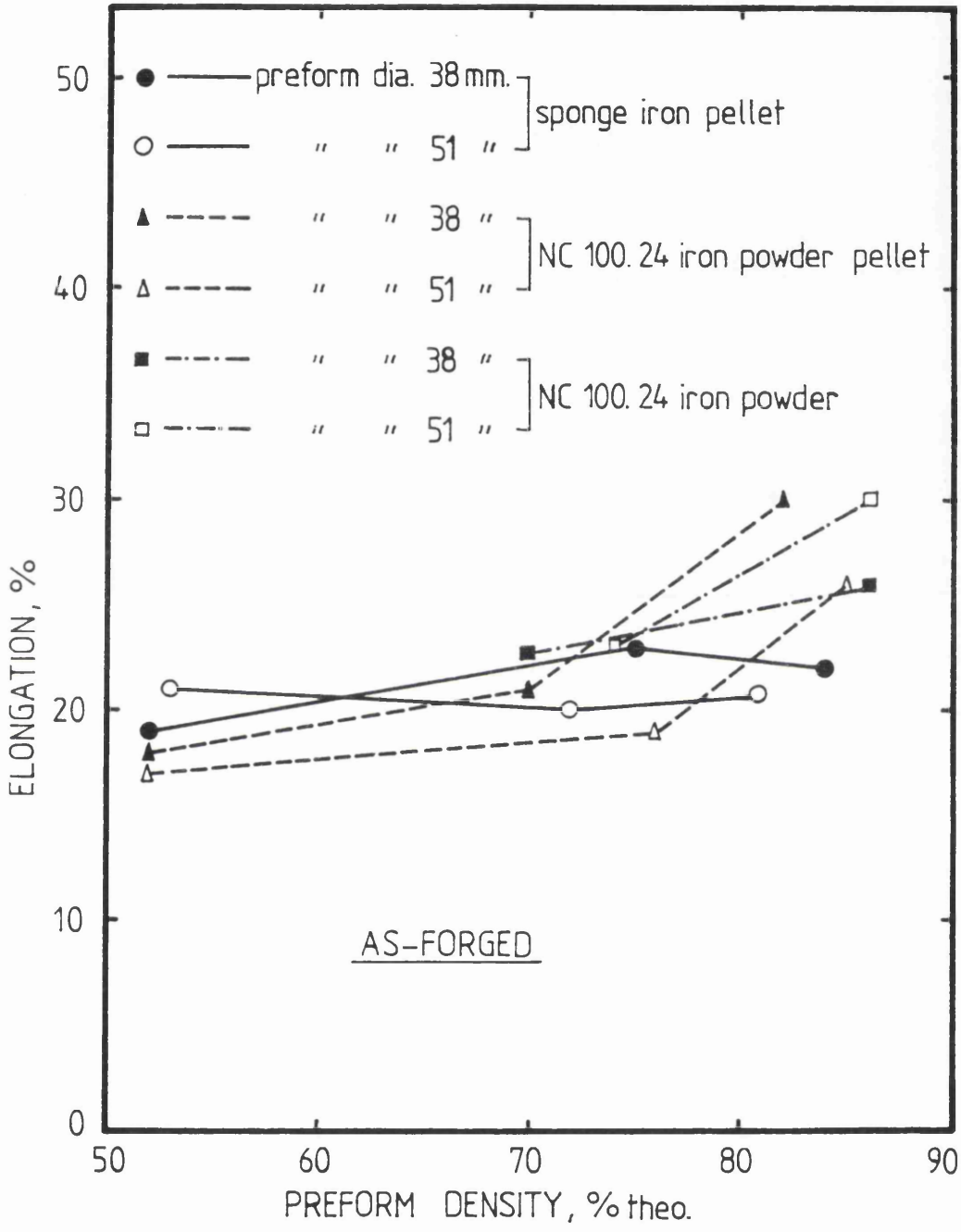


Fig. 9.3. Effect of preform density and preform diameter on the ductility of the forgings in as-forged condition.

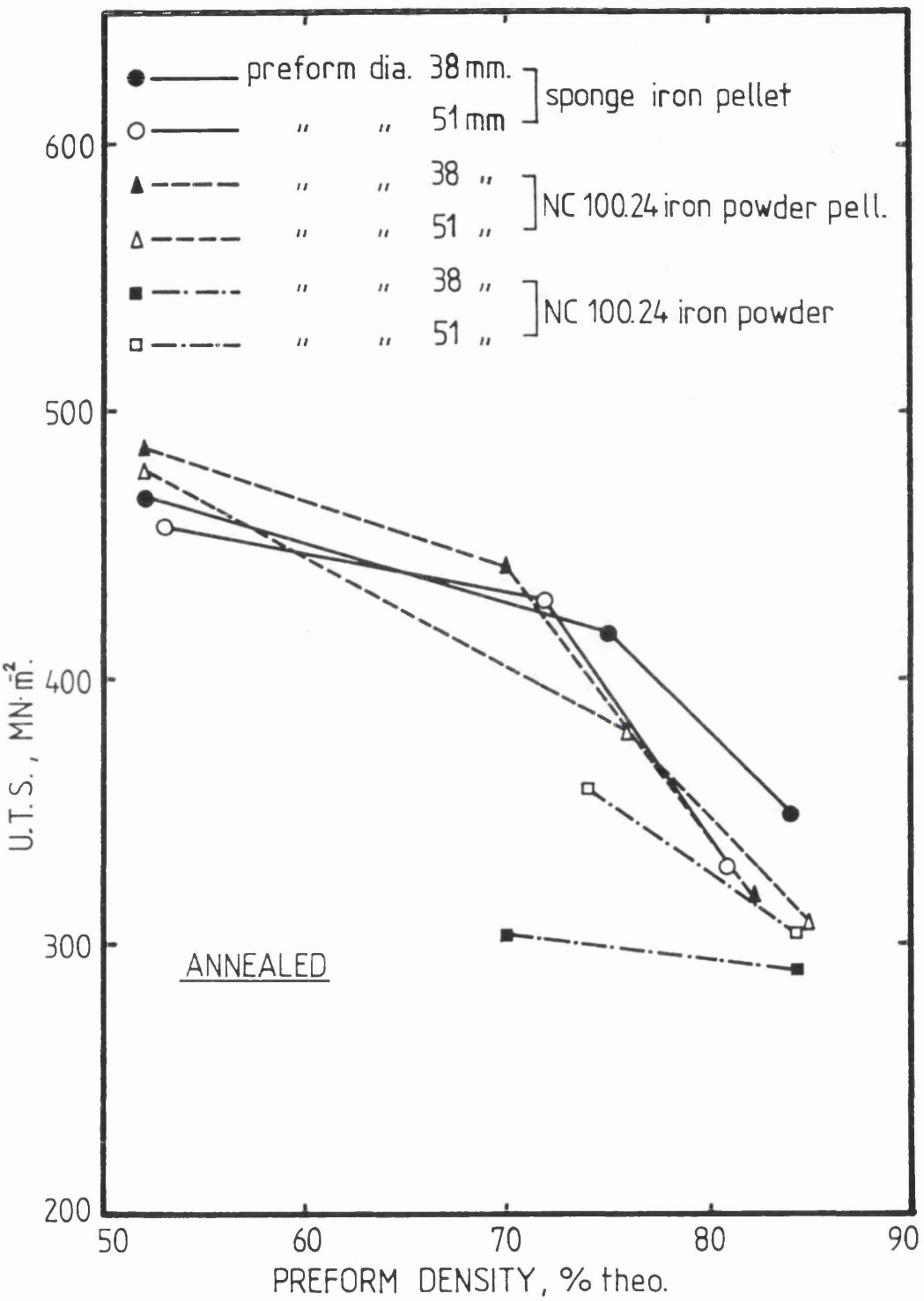


Fig. 9.4. Effect of preform density and preform diameter on the U.T.S. of the forgings in annealed condition.

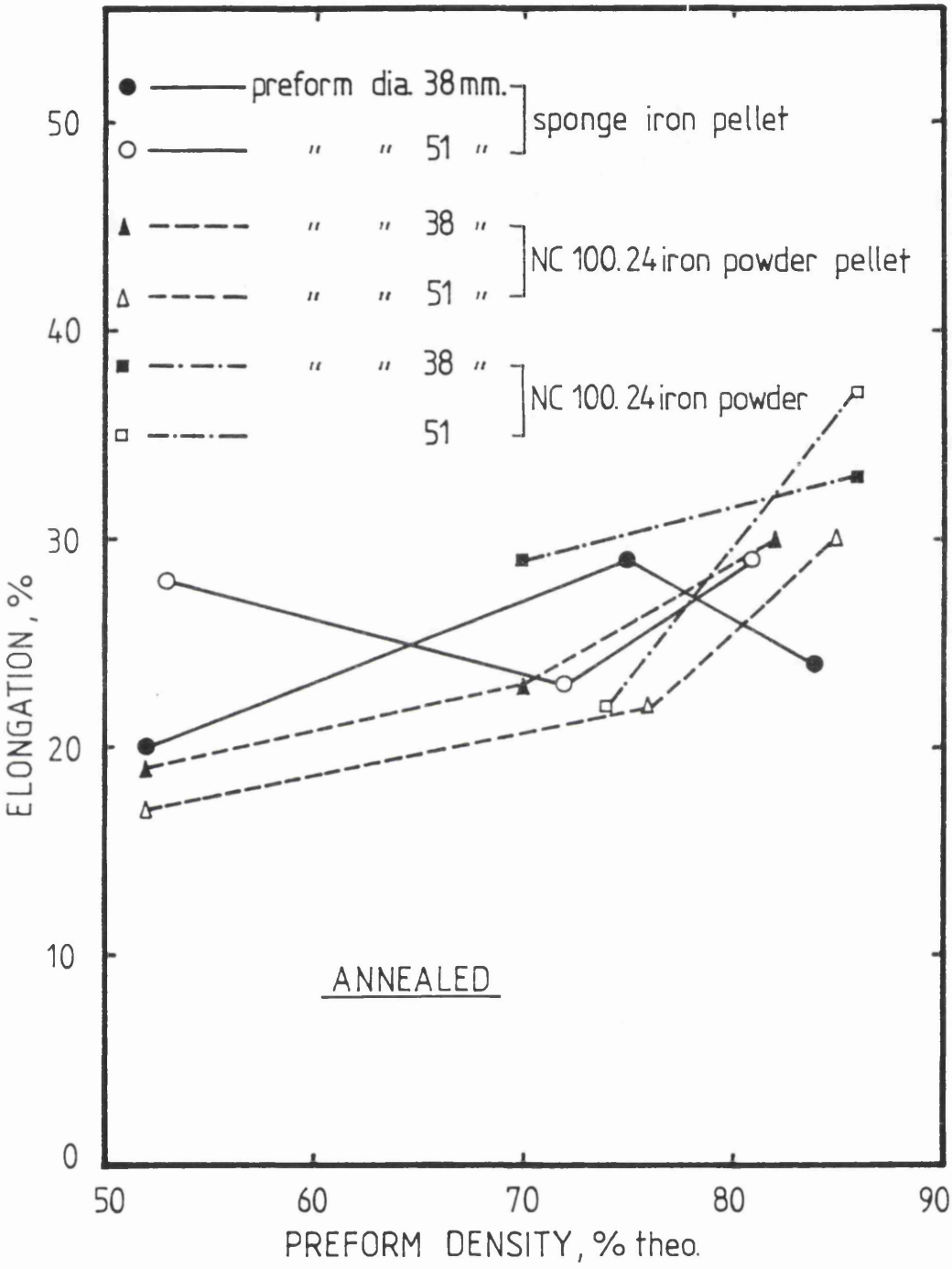


Fig. 9.5. Effect of preform density and preform diameter on the ductility of the forgings in annealed condition.

the final forging. However, when the preform was carefully placed centrally in the die cavity, any cracks formed in the earlier stage of the deformation started to close up when the die wall was reached. As the deformation progressed further the cracks disappeared completely because of rewelding, the workpiece temperature being still high.

As a result the sponge iron pellet preforms behaved well during hot forging at 1100°C at all preform densities and with both deformation types of repressing and with limited lateral flow. This clearly demonstrates that the sponge iron pellet preforms had sufficient strength and ductility at 1100°C to enable them to withstand all the stresses involved during hot forging and shows that the preform design was successful.

The relationship between the preform density and the U.T.S. of the three materials in the as-forged condition is shown graphically in Fig. 9.2, demonstrating that the U.T.S. of low density preforms is significantly higher than for high density preforms for all materials.

For all materials the elongation values of the forged product were similar for the 50% and 70% preform density samples. At 85% preform density no increase in ductility was found for the sponge iron pellet forgings, but for the other two materials a sharp increase occurred. (see Fig. 9.3 and Table 9.I.)

In the annealed condition the U.T.S. values were slightly higher than for the as-forged condition, but the same trend was present as in the results of the as-forged condition. (see Fig. 9.4.)

Elongation results in the annealed condition are shown graphically in Fig. 9.5 demonstrating an increase of elongation with increase in preform density for both iron powder products. For sponge iron pellets there was no clear trend.

Examination of the structure of the forgings showed that sponge iron pellet forgings gave a small grain size. Iron powder pellet forgings gave a slightly coarser grain size and comparable powder forged products had a much larger grain size. Also, inclusions and residual porosity were more uniformly distributed in the sponge iron pellet product than with either of the iron powder products. The effect of inclusions and grain size on the properties of forgings will be dealt with in the next chapter. Three typical microstructures are shown in Figs.9.6 and 9.7.

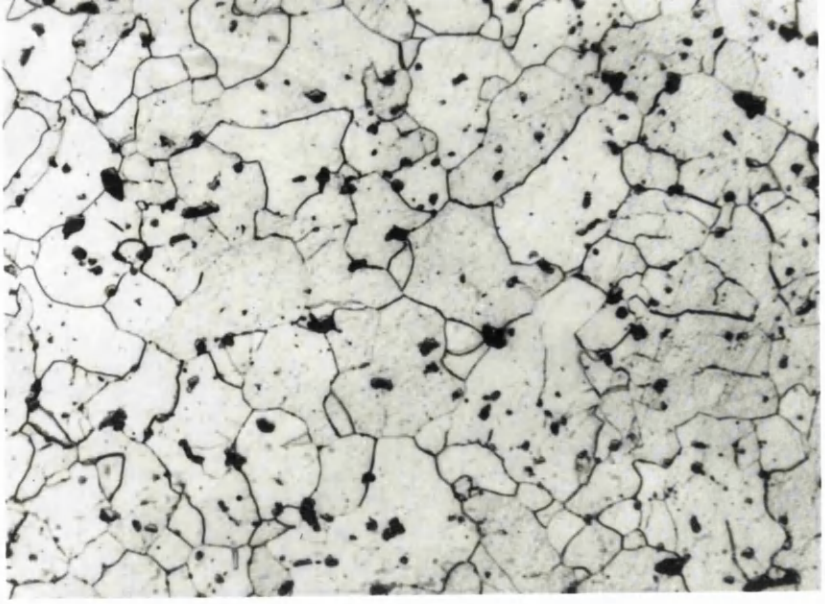
In summary, generally these results show that there is a significant effect of using low density pellet preforms, i.e., that under certain conditions pellet forgings can produce products having a higher U.T.S. than conventional powder forgings, both before and after annealing. Microscopic examination gives support to these results.

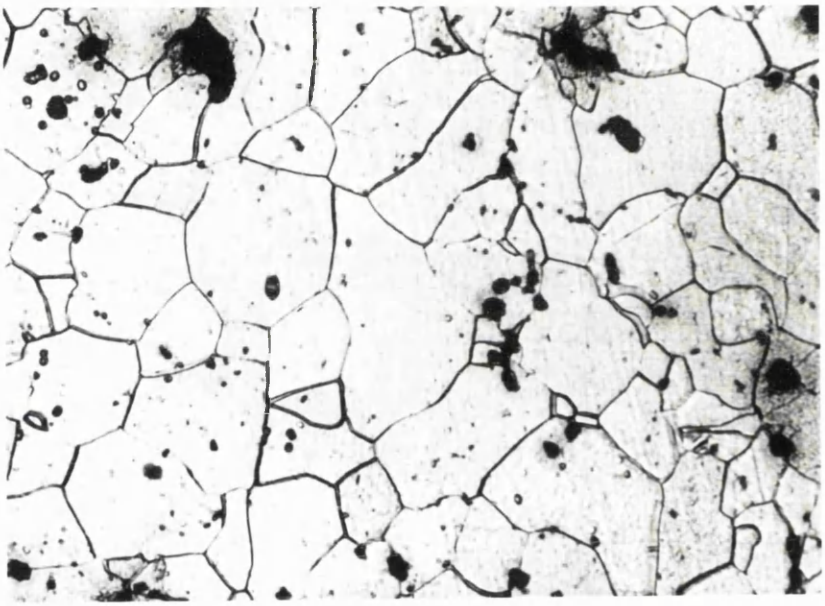
9.2. EFFECT OF PELLET SIZE, TYPE OF DEFORMATION, PREFORM DENSITY, AND ANNEALING ON THE MECHANICAL PROPERTIES OF FORGINGS

The initial experimentation had shown that a product with properties similar to those of conventional powder forging could be produced by the new route. However, the effect on the properties of the product of variations in

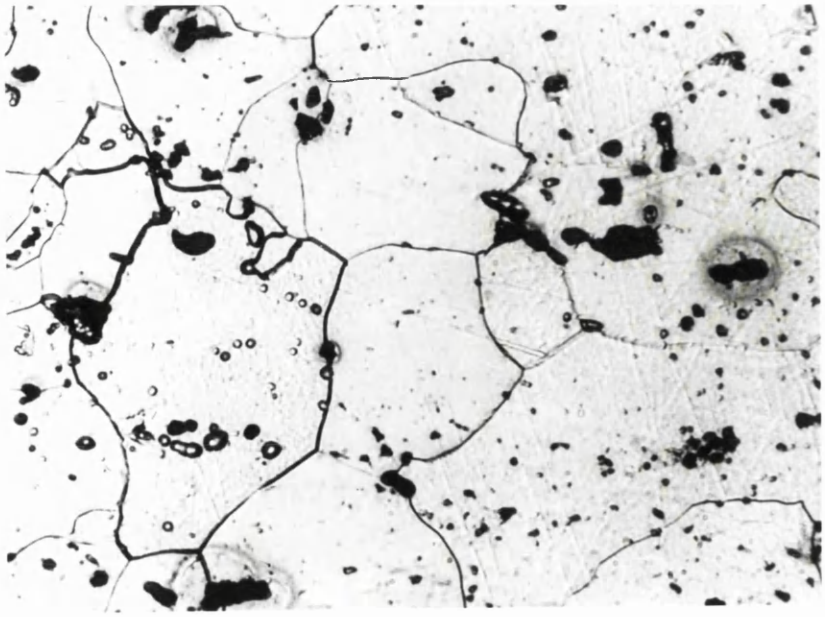
Fig. 9.6. Typical structure of sponge iron pellet forging
X530

Fig. 9.7. Typical structure of forgings produced from
(a) NC 100.24 iron powder pellet forging
(b) NC 100.24 iron powder forging
X530





a



b

the production parameters was unknown and a statistically designed full factorial experiment was undertaken to determine these effects. In Fig. 7.12 the experimental details are illustrated schematically, temperature being maintained constant using three materials and four variables, as follows:

Materials	Sponge iron pellets
	NC 100.24 iron powder pellets
	NC 100.24 iron powder
Variables	Preform density (50%, 70% and 85% theo.)
	Preform diameter (31 mm and 58 mm)
	Pellet size (< 8 mm, 8-14 mm and 14-20 mm)
	As-forged or annealed
Properties	Y.S.
	U.T.S.
	Elongation

The results obtained are shown in Tables 9.II, 9.III and 9.IV. In view of the large number of variables involved in producing final forgings, it was decided to design the experiment in order to quickly determine those variables which had major effects and the way in which they interacted with each other. A full 2^4 factorial design was used and the measured properties of the forgings were analysed by a Yates[111] procedure to determine the effects.

In the case of sponge iron pellets and iron powder pellets, four variables were investigated. The variables were coded in the following way:

A Preform density

B Preform diameter

Table 9.II. Mechanical Properties Of Sponge Iron Pellet Forgings.

Pellet size (mm)	Preform Diameter (mm)	Preform Density (% theo.)	Condition*	Y.S. ($MN.m^{-2}$)	U.T.S. ($MN.m^{-2}$)	Elongation (%)
< 8	38	50	F	405	459	12
			A	430	456	16
		71	F	312	356	19
			A	279	346	24
		84	F	252	308	24
			A	211	304	30
	51	50	F	310	410	18
			A	336	399	20
		72	F	329	415	17
			A	363	393	22
		83	F	283	337	21
			A	281	324	22
8 - 14	38	51	F	363	470	19
			A	391	463	22
		72	F	314	410	20
			A	339	421	22
		83	F	227	333	21
			A	233	321	26
	51	51	F	296	391	22
			A	289	373	29
		70	F	316	413	17
			A	331	406	18
		83	F	321	418	16
			A	278	278	22
14 - 20	38	50	F	391	442	16
			A	354	385	23
		71	F	301	404	22
			A	331	404	23
		83	F	258	340	19
			A	220	317	25
	51	52	F	451	502	11
			A	456	512	14
		71	F	304	405	11
			A	285	347	21
		84	F	291	363	22
			A	296	347	26

* F: as-forged, A: as-forged and annealed.

Table 9.III. Mechanical Properties Of Iron Powder Pellet Forgings

Pellet size (mm)	Preform Diameter (mm)	Preform Density (% theo)	Condi-* tion	Y.S. ($MN.m^{-2}$)	U.T.S. ($MN.m^{-2}$)	Elongation (%)
< 8	38	50	F	301	386	16
			A	292	365	22
		68	F	350	454	14
			A	357	454	16
		84	F	257	334	16
			A	213	309	21
	51	50	F	259	370	17
			A	319	363	23
		72	F	343	446	19
			A	432	471	19
		84	F	223	327	25
			A	225	315	30
8 - 14	38	53	F	360	456	14
			A	384	432	18
		72	F	358	461	14
			A	403	480	18
		84	F	220	304	25
			A	212	303	28
	51	51	F	278	377	20
			A	309	347	26
		72	F	276	391	15
			A	328	379	20
		86	F	266	334	25
			A	257	329	30
14 - 20	38	51	F	320	386	21
			A	291	339	22
		71	F	280	380	22
			A	255	336	24
		83	F	223	303	25
			A	200	292	27
	51	51	F	352	467	12
			A	465	485	14
		72	F	349	450	14
			A	450	470	17
		85	F	293	366	20
			A	289	352	22

* F: as-forged, A: as-forged and annealed.

Table 9.IV. Mechanical Properties Of Iron Powder Forgings

Material	Preform Diameter (mm)	Preform Density (% theo.)	Condition *	Y.S. ($MN.m^{-2}$)	U.T.S. ($MN.m^{-2}$)	Elongation (%)
NC 100.24 Iron Powder	38	70	F	245	349	18
			A	252	315	29
		86	F	242	343	18
			A	184	278	29
	51	73	F	228	309	11
			A	280	315	31
		85	F	265	331	18
			A	226	315	26

* F: as-forged, A: as-forged and annealed.

C Pellet size

D Annealing time after forging

Two levels of each variable were chosen as follows:

	Level 1	Level 2
A	50%	85%
B	38 mm	51 mm
C	< 8 mm	14 - 20 mm
D	0 s	3600s at 700°C

For forgings made directly from iron powder, only variables A, B and D were investigated and preforms of 70% and 85% at Level 1 and Level 2 of variable A were used. Thus two 2^4 factorial experiments and one 2^3 factorial experiment were conducted. In each case the yield stress, U.T.S. and elongation to fracture of the forgings were determined. No replication was attempted and since there was insufficient background information to form an external estimate, an estimate of error variance was obtained from the experiments themselves. Interactions of order greater than two were assumed to be insignificant and these were used to produce an estimate of error variance. The significance of effects was then judged using a standard F-test.

The results were analysed using a standard Yates[111] technique and the effects, together with their levels of significance, are given in Tables 9.V, 9.VI and 9.VII. In these tables the presence of a lower case letter in the treatment column indicates that the related variable was set at level 2. It should be noted that the sign of the

Table 9.V. Statistical Evaluation Of Results From Sponge Iron Pellet Forgings.

Treatment	Y.S. ($MN.m^{-2}$)			U.T.S. ($MN.m^{-2}$)			Elongation (%)			Effect Code
	Result	Effect	Significance	Result	Effect	Significance	Result	Effect	Significance	
(1)	405	-	-	459	-	-	12	-	-	TOTAL
a	252	-130	< 0.1%	308	-115	< 0.1%	24	7.4	< 0.5%	A
b	310	23	< 10%	410	23	< 5%	18	-1.4	Not sig.	B
ab	283	30	< 5%	337	3	Not sig.	21	-0.4	Not sig.	AB
c	391	26	< 10%	442	26	< 5%	16	-0.9	Not sig.	C
ac	258	-17	Not sig.	340	- 3	Not sig.	19	-0.4	Not sig.	AC
bc	451	45	< 2.5%	502	37	< 1%	11	-1.1	Not sig.	BC
abc	291	-43	Not sig.	363	-36	Not sig.	22	4.9	Not sig.	ABC
d	430	- 7	Not sig.	456	-15	Not sig.	16	4.1	< 2.5%	D
ad	211	-12	Not sig.	304	1	Not sig.	30	0.1	Not sig.	AD
bd	336	16	Not sig.	399	7	Not sig.	20	-1.6	Not sig.	BD
abd	281	5	Not sig.	324	- 8	Not sig.	22	-0.1	Not sig.	ABD
cd	354	- 9	Not sig.	385	- 7	Not sig.	23	0.9	Not sig.	CD
acd	220	12	Not sig.	317	1	Not sig.	25	-0.1	Not sig.	ACD
bcd	456	6	Not sig.	512	11	Not sig.	14	0.1	Not sig.	BCD
abcd	296	- 5	Not sig.	347	- 7	Not sig.	26	0.6	Not sig.	ABCD

Table 9.VI. Statistical Evaluation Of Results From Iron Powder Pellet Forgings.

Treatment	Y.S. ($MN.m^{-2}$)			U.T.S. ($MN.m^{-2}$)			Elongation (%)			Effect Code
	Result	Effect	Significance	Result	Effect	Significance	Result	Effect	Significance	
(1)	301	-	-	386	-	-	16	-	-	TOTAL
a	257	-85	< 0.1%	334	-68	< 0.1%	16	4.9	< 0.1%	A
b	259	41	< 0.1%	370	44	< 0.1%	17	-0.9	< 2.5%	B
ab	223	- 7	Not sig.	327	-13	< 2.5%	25	2.9	< 0.1%	AB
c	320	43	< 0.1%	368	25	< 0.5%	21	-0.9	< 2.5%	C
ac	223	-21	< 1%	303	-18	0.5%	25	1.4	0.5%	AC
bc	235	50	< 0.1%	467	48	< 0.1%	12	-5.9	< 0.1%	BC
abc	239	- 5	Not sig.	366	-17	Not sig.	20	-1.1	Not sig.	ABC
d	292	8	Not sig.	365	-13	< 2.5%	22	3.6	< 0.1%	D
ad	213	-26	< 0.5%	309	- 3	Not sig.	21	-0.1	Not sig.	AD
bd	319	35	< 0.5%	363	9	5%	23	0.1	Not sig.	BD
abd	225	-18	Not sig.	315	- 6	Not sig.	30	-0.1	Not sig.	ABD
cd	291	6	Not sig.	339	4	Not sig.	22	-1.9	< 0.5%	CD
acd	200	- 2	Not sig.	292	- 1	Not sig.	27	0.4	Not sig.	ACD
bcd	465	6	Not sig.	485	2	Not sig.	14	0.1	Not sig.	BCD
abcd	289	-13	Not sig.	352	- 6	Not sig.	22	-0.1	Not sig.	ABCD

Table 9.VII. Statistical Evaluation Of Results From Iron Powder Forgings.

Treatment	Y.S. ($MN.m^{-2}$)			U.T.S. ($MN.m^{-2}$)			Elongation (%)			Effect Code
	Result	Effect	Significance	Result	Effect	Significance	Result	Effect	Significance	
(1)	245	-	-	349	-	-	18	-	-	TOTAL
a	242	-22	Not sig.	343	-5.3	Not sig.	18	0.5	Not sig.	A
b	228	19	Not sig.	309	-3.8	Not sig.	11	-2	Not sig.	B
ab	265	13.5	Not sig.	331	16.3	10%	18	0.5	Not sig.	AB
d	252	-9.5	Not sig.	315	-27.3	< 5%	29	12.5	< 0.5%	D
ad	184	-39	< 10%	278	-13.3	Not sig.	29	-3	< 10%	AD
bd	280	16	Not sig.	315	22.3	5%	31	1.5	Not sig.	BD
abd	226	-6.5	Not sig.	315	2.3	Not sig.	26	-3	Not sig.	ABD

effect is also given in the effect column. Where a minus sign exists before an effect this indicates that increasing the level of the relevant factor decreases the result.

Table 9.V shows that for sponge iron pellet forgings, preform density has the most significant effect on the properties. Preform diameter and pellet size have much less significant effects on the Y.S. and U.T.S. Although the degree of annealing has some effect on the elongation, this property is mainly dependent on the preform density. These results are shown graphically in Figs. 9.8 - 9.11. Despite the scatter of results, generally U.T.S. values increased with decrease in preform density and elongation values increased with increase in preform density. Annealing of the forged product gave an increase in elongation. (see Figs. 9.10 and 9.11.)

Table 9.VI shows that the Y.S. of the iron powder pellet forgings are significantly affected by preform density, preform diameter and pellet size. But it is apparent from the value of the effects that the preform density is the most significant variable. The effectsof all the variables on U.T.S. are significant at different orders of magnitude, the highest being for preform density. The other variables showed little significant effect, also shown graphically in Figs. 9.12 and 9.13. As with the sponge iron pellet forgings, preform density has the highest significant effect on elongation. Annealing was also found to be significant. (see Figs. 9.14 and 9.15.)

Table 9.VII shows that the U.T.S. and elongation of the iron powder forgings are only significantly affected

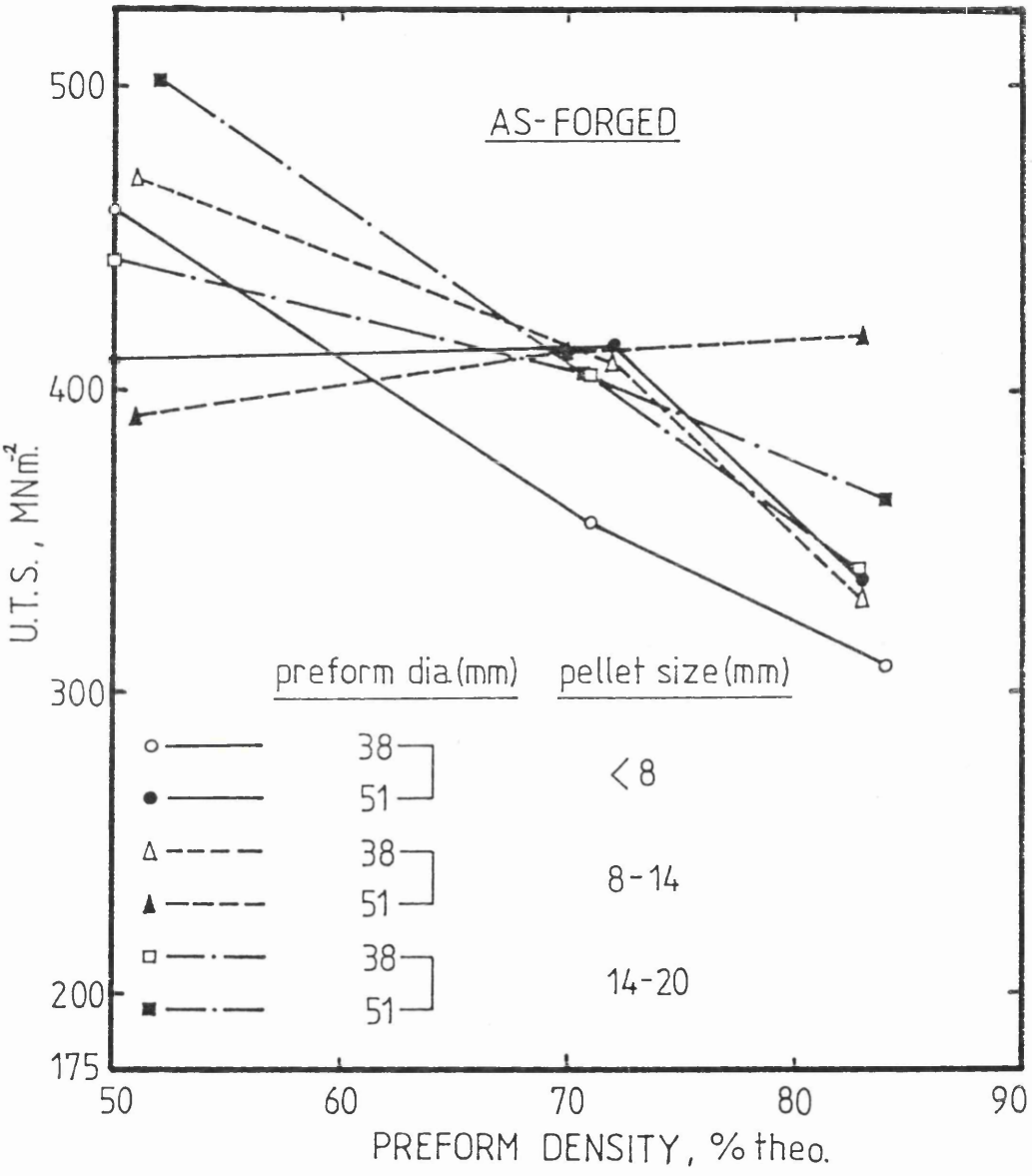


Fig. 9.8. U.T.S. of sponge iron pellet forgings.

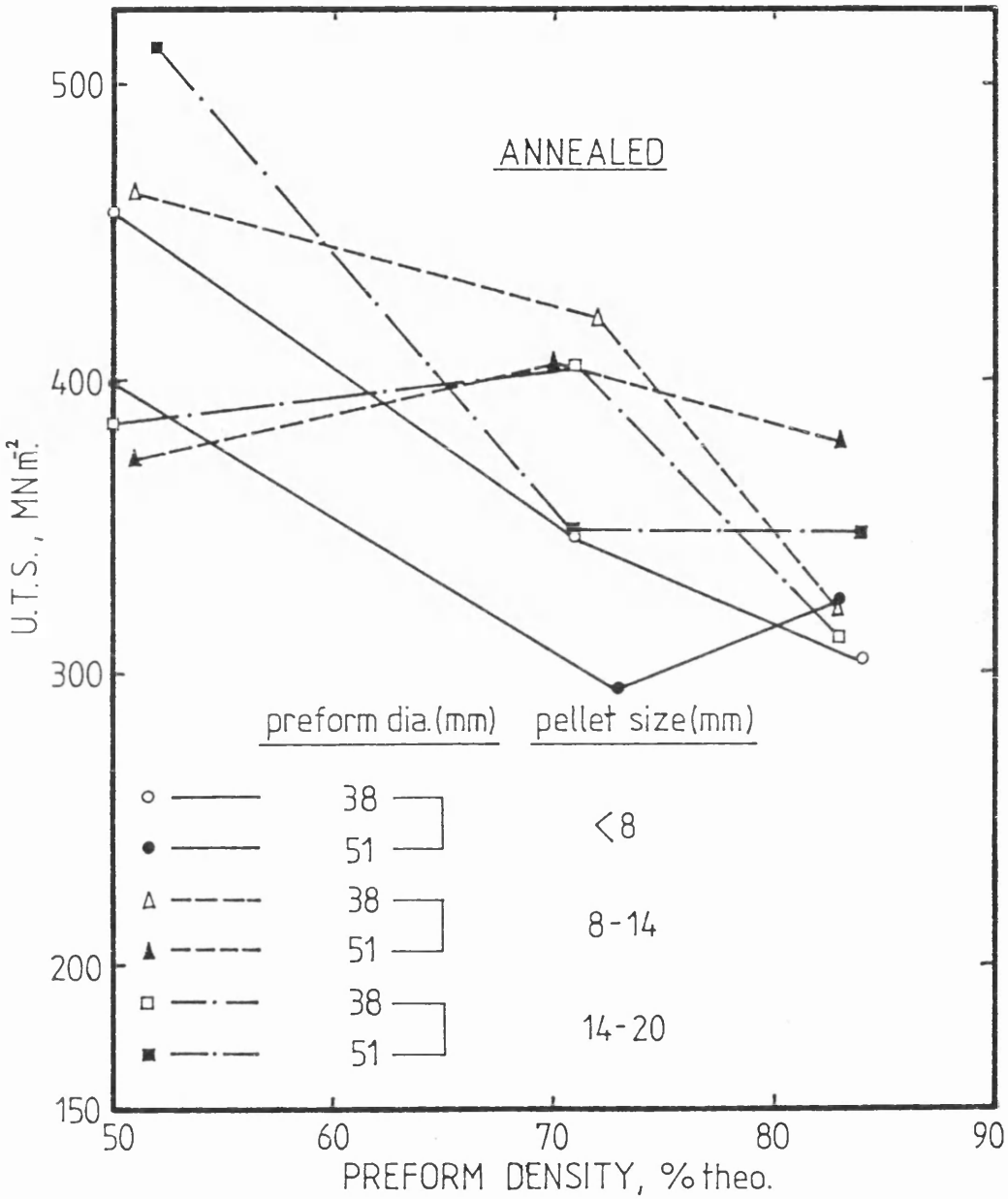


Fig. 9.9. U.T.S. of sponge iron pellet forgings.

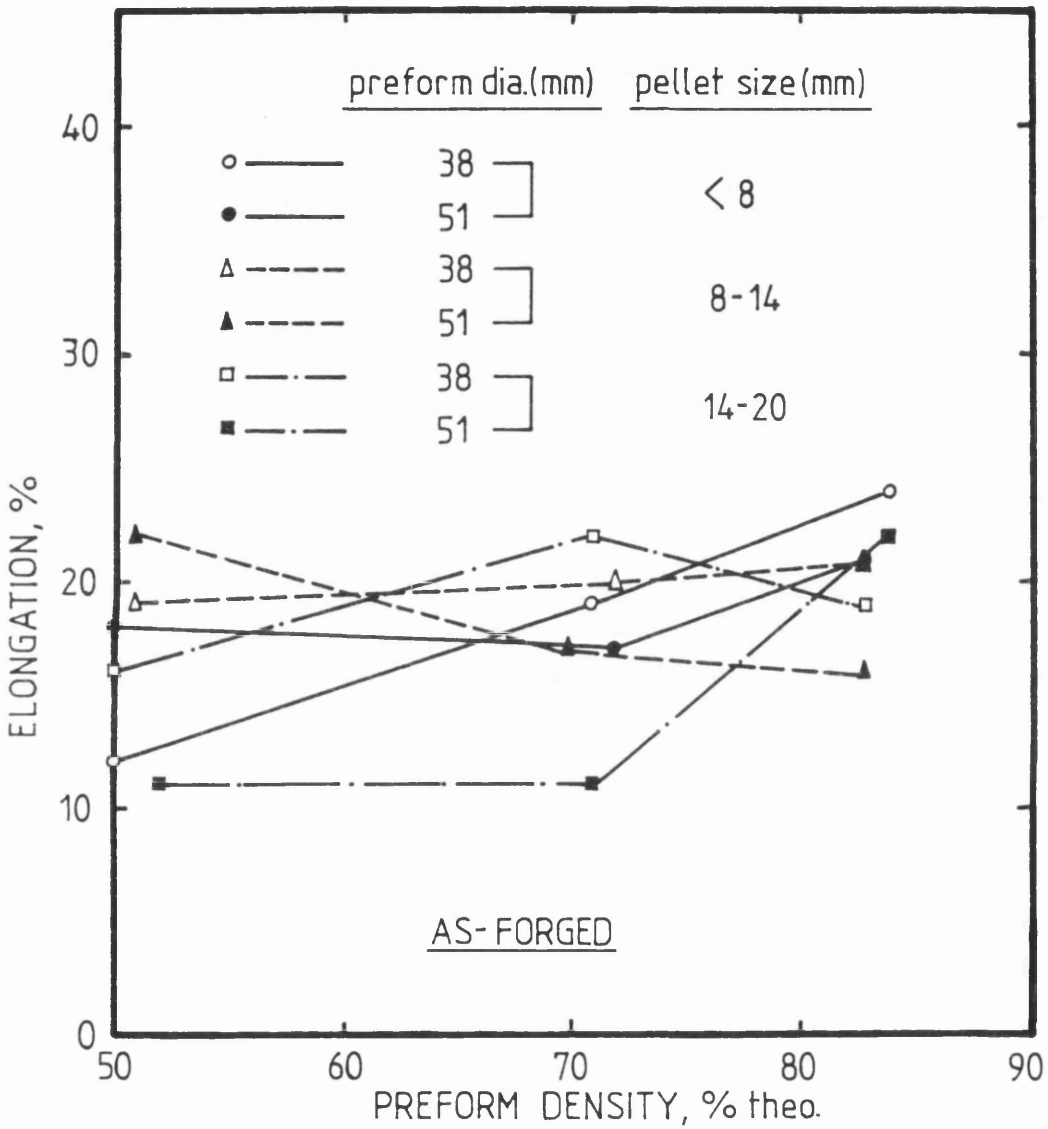


Fig. 9.10. Ductility of sponge iron pellet forgings.

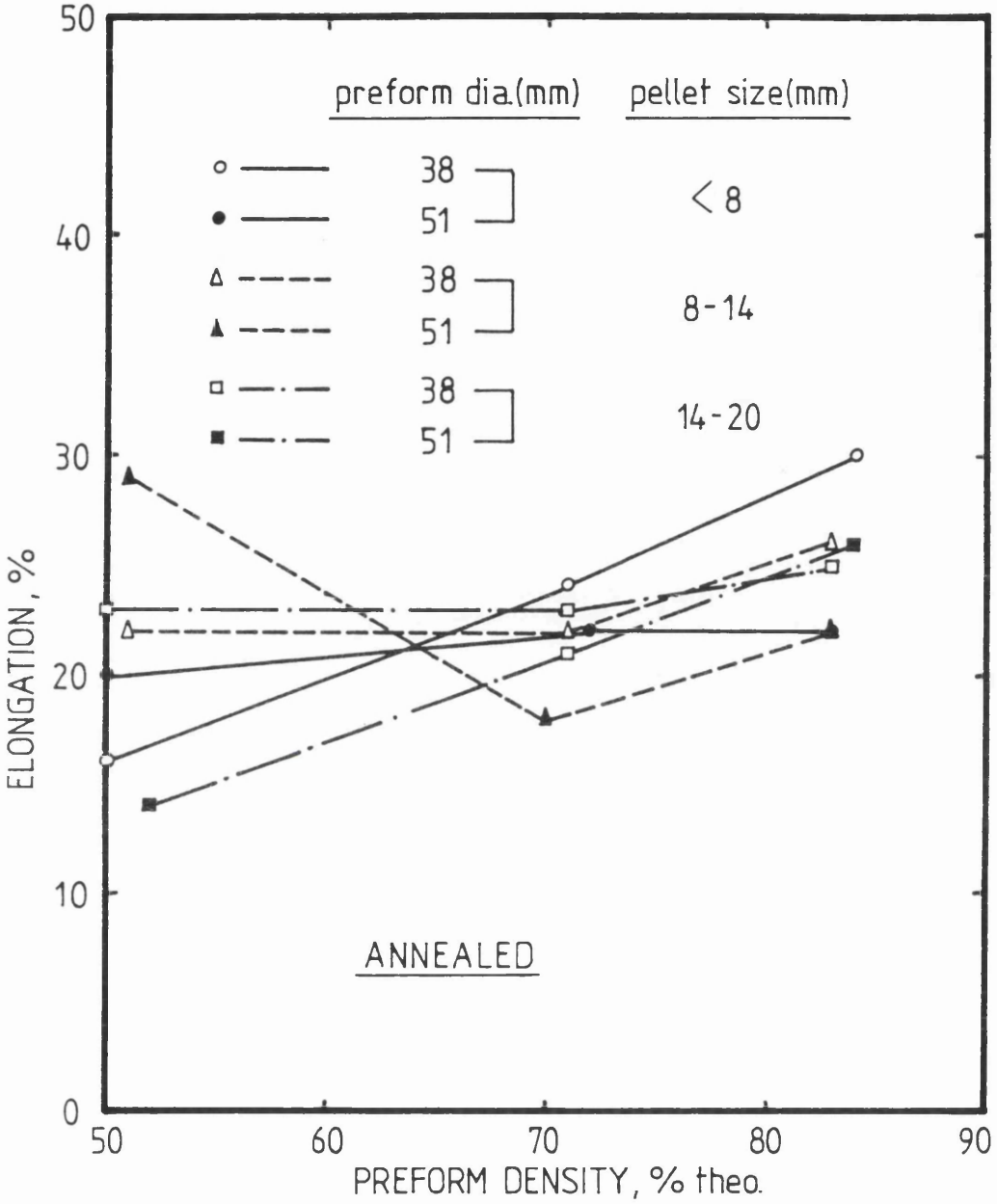


Fig. 9.11. Ductility of sponge iron pellet forgings.

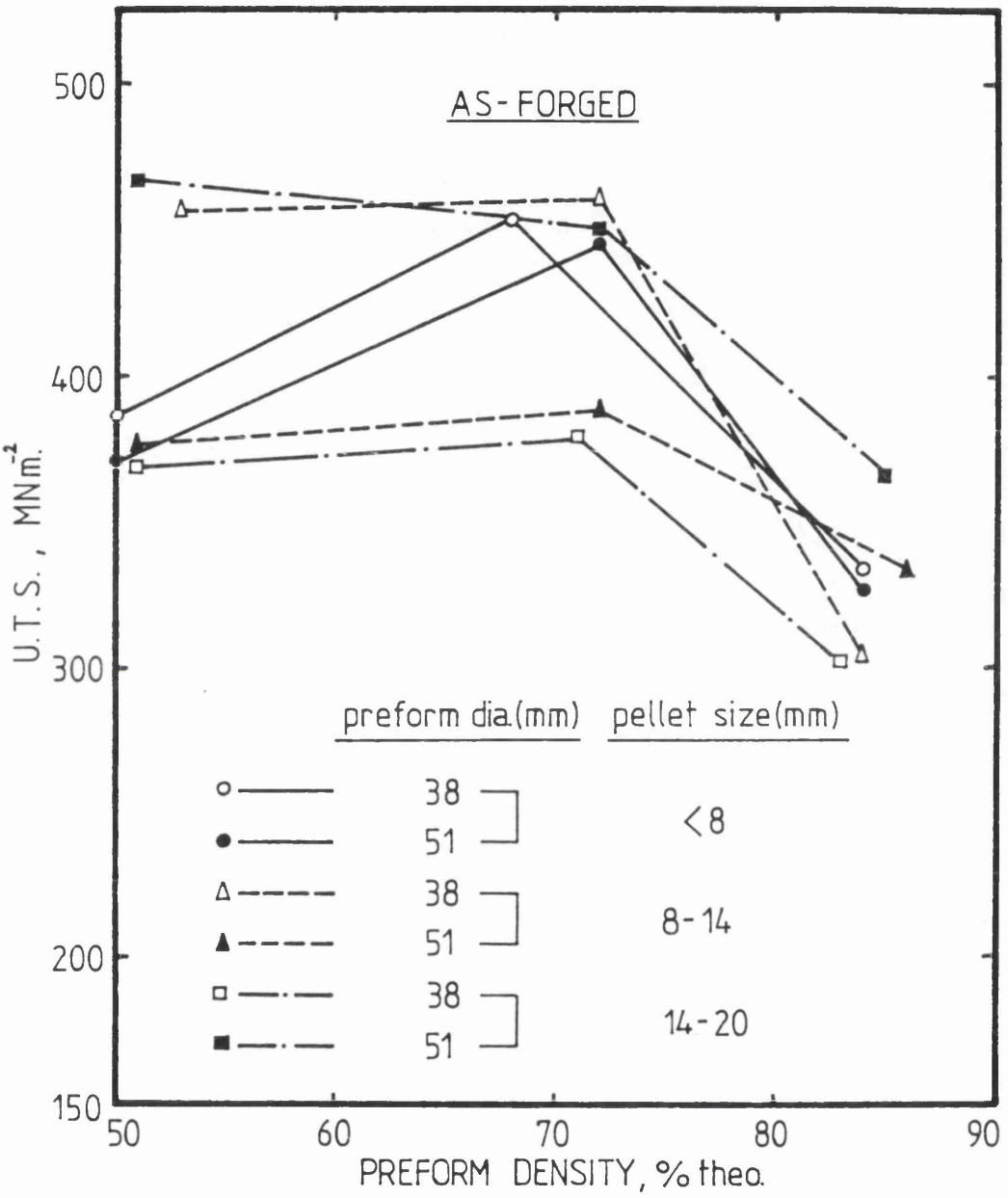


Fig. 9.12. U.T.S. of iron powder pellet forgings.

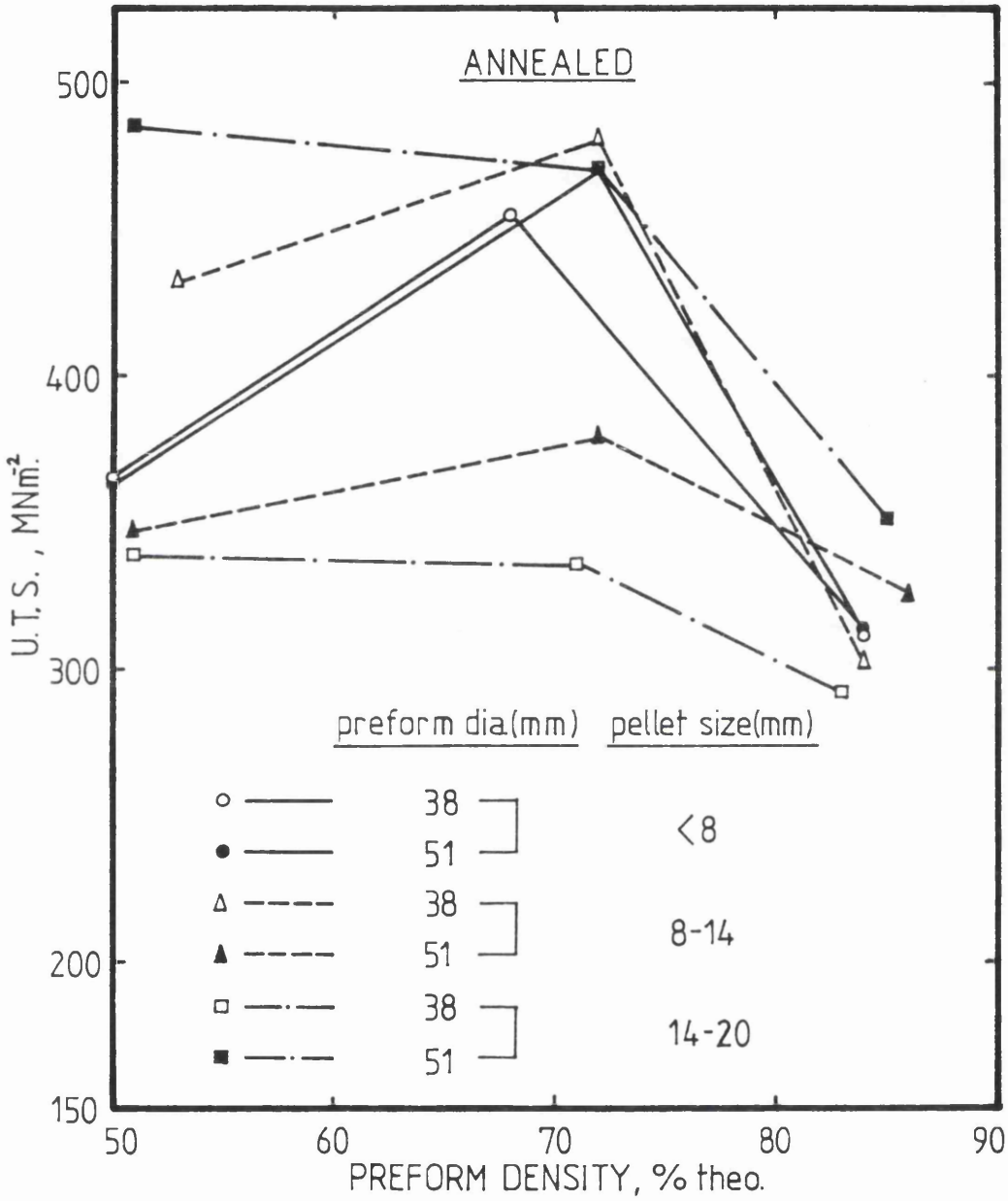


Fig. 9.13. U.T.S. of iron powder pellet forgings.

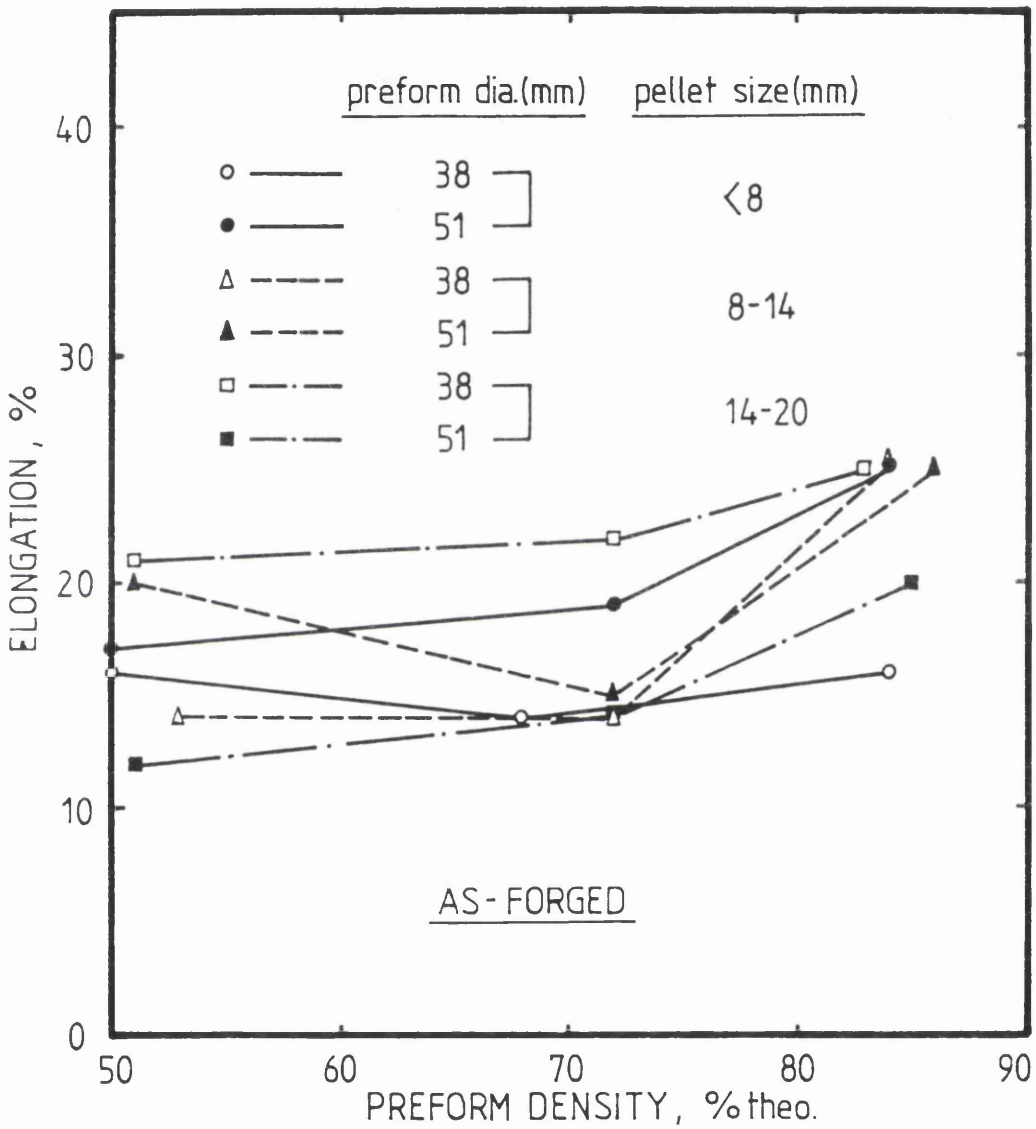


Fig. 9.14. Ductility of iron powder pellet forgings.

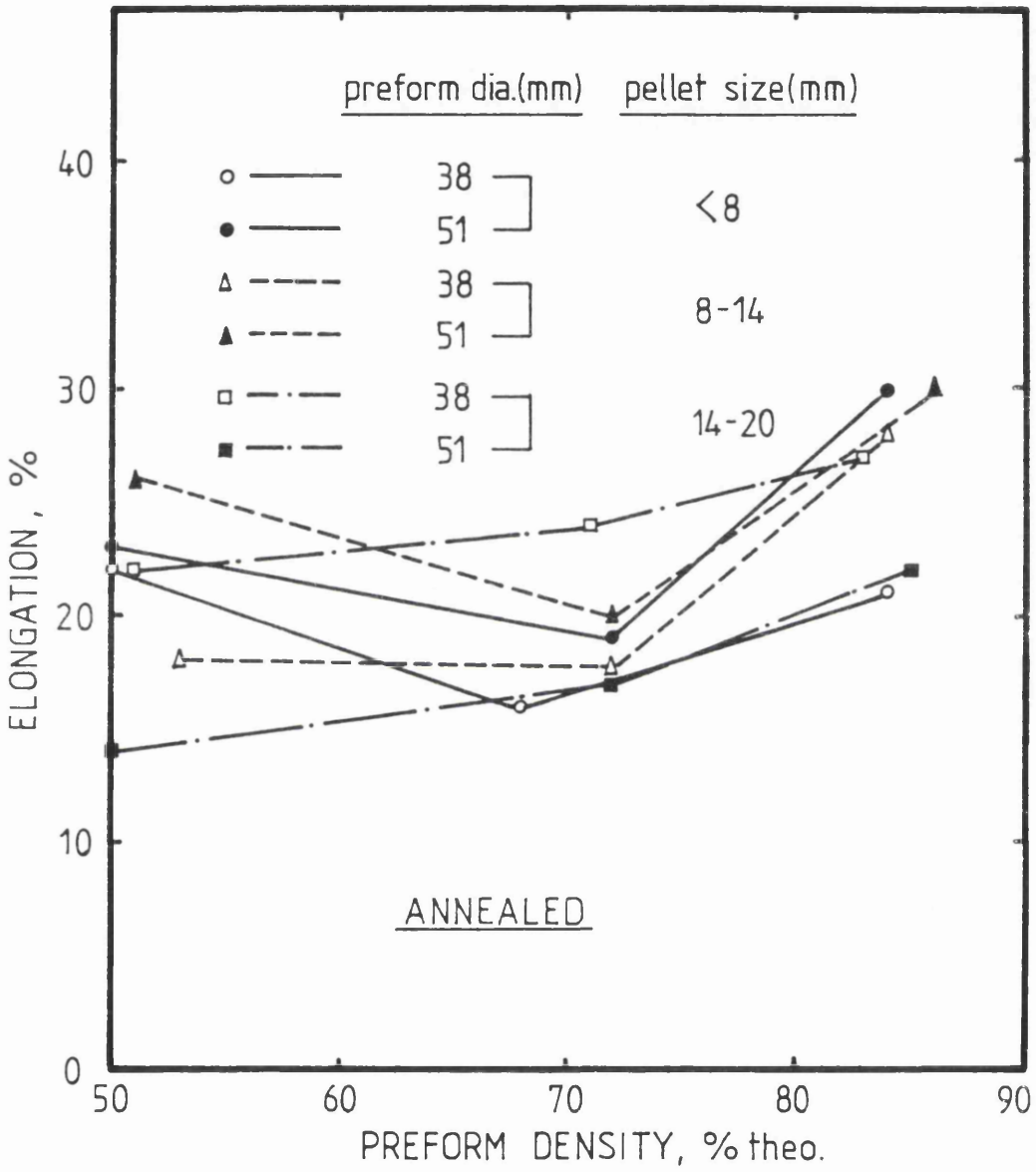


Fig. 9.15. Ductility of iron powder pellet forgings.

by the annealing treatment. Figs. 9.16 and 9.17 show that annealing has decreased the U.T.S. values and increased the elongation values of the forgings.

To summarise, for sponge iron pellet forgings and iron powder pellet forgings only preform density has a significant effect on strength and ductility. The effect of other variables and their interactions are small in comparison. However, this variable has no significant effect on iron powder forgings, but the properties are very dependent on the degree of annealing treatment. In Table 9.VIII the U.T.S. and elongation values are listed against those variables which produced a significant effect, for the various materials.

The three materials thus show significant differences in the response of their properties to the manufacturing variables. The strongest material can be produced from sponge iron pellets whilst the most ductile from iron powder forging. Iron powder pellet forgingsproduces a material with intermediate properties. The structural reasons for the above variations in properties will be discussed in the next chapter. It appears that sponge iron pellet forgings have a small grain size whereas iron powder pellet forgings have an intermediate grain size and iron powder forgings have a coarser grain size. In addition, the inclusions are smaller and more uniformly distributed in sponge iron pellet forgings. (see Figs.9.6 and 9.7.)

9.3. EFFECT OF SINTERING OF PREFORMS PRIOR TO FORGING ON THE FINAL PROPERTIES OF FORGING

The results on the effect of sintering prior to hot

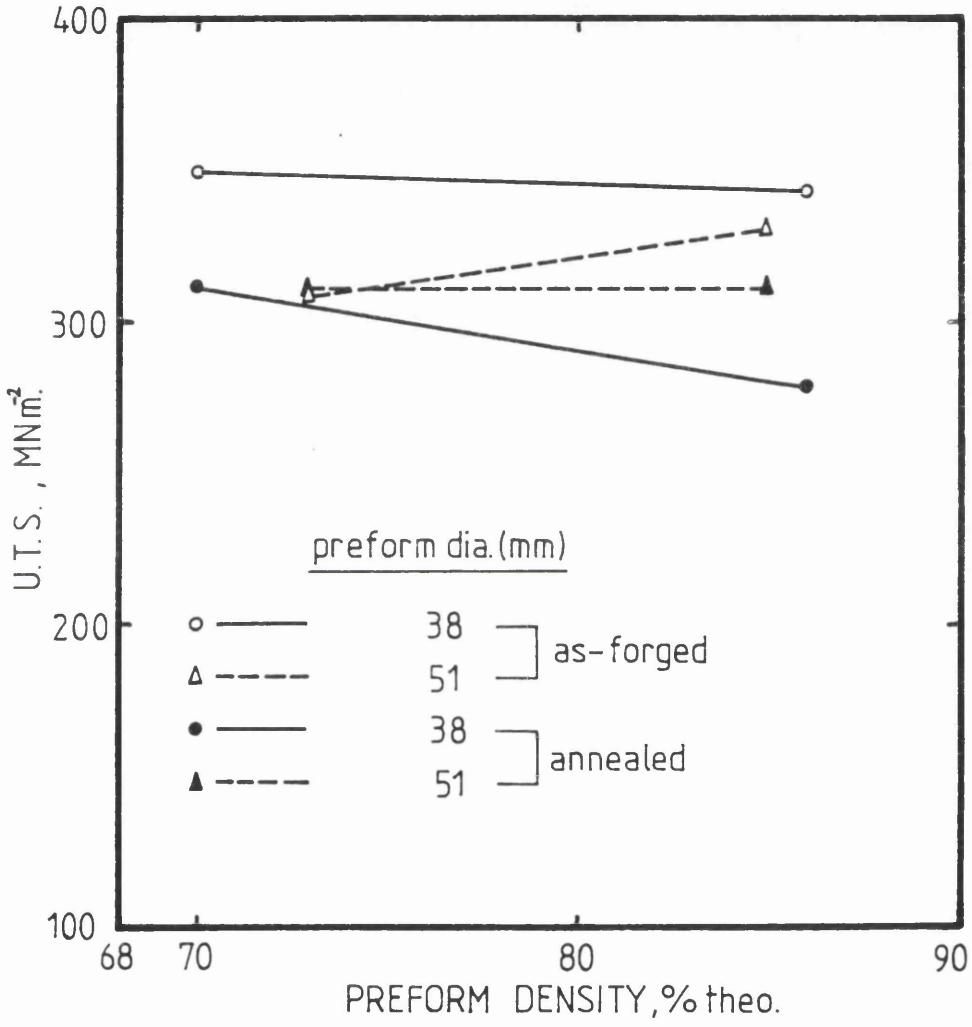


Fig. 9.16. U.T.S. of iron powder forgings.

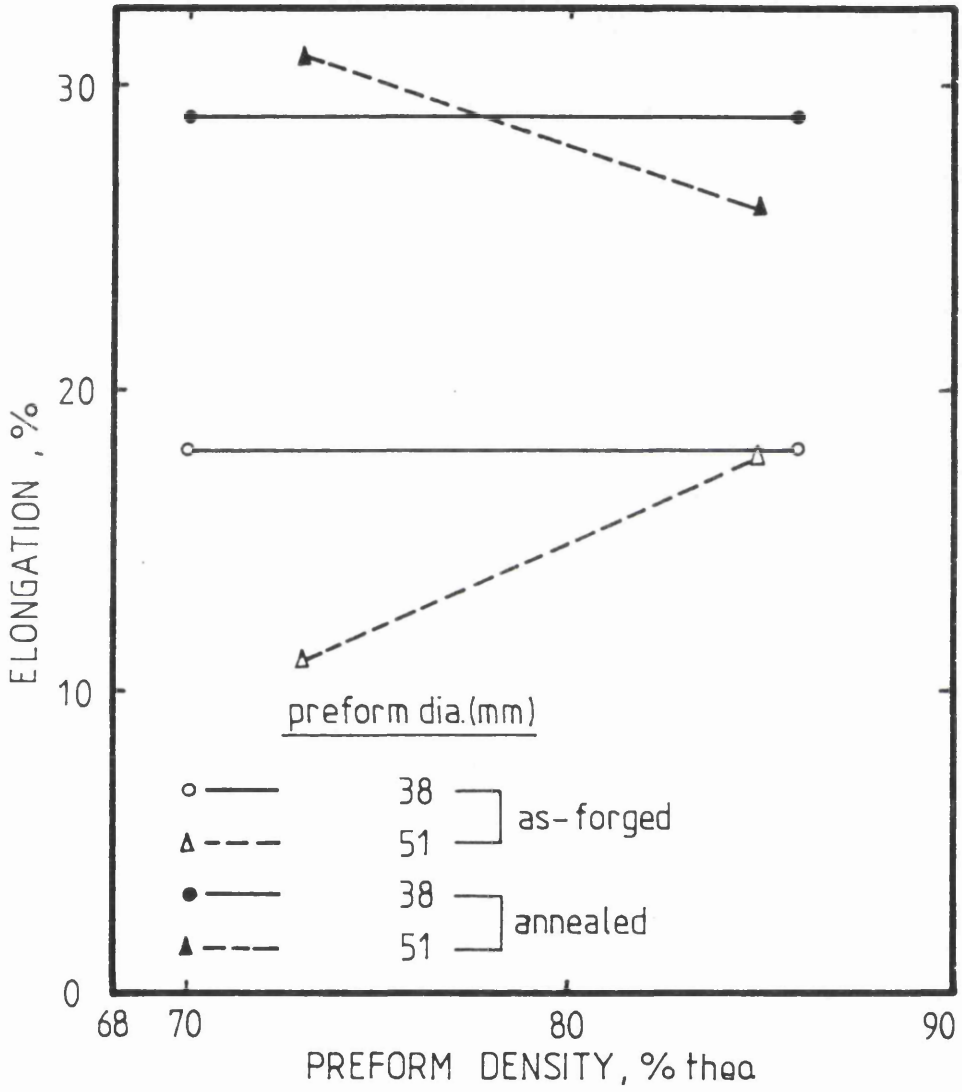


Fig. 9.17. Ductility of iron powder forgings.

Table 9.VIII. Variation Of Properties With The Significant Variables.

Material	Variable	U.T.S. ($MN.m^{-2}$)	Elongation (%)
Sponge Iron Pellet Forgings	Low Preform Density	446	16.3
	High Preform Density	330	23.6
Iron Powder Pellet Forgings	Low Preform Density	393	18.4
	High Preform Density	325	23.3
Iron Powder Forgings	As-Forged	333	16.3
	Forged and Annealed	306	28.8

forging on the mechanical properties of sponge iron pellet forgings and NC 100.24 iron powder forgings are shown in Table 9.IX and Figs. 9.18(a) and 9.18(b). All preforms were prepared in a 38 mm dia. die to 70% theoretical density. Preforms were sintered at 1100°C for 1, 2, 3 hours in H_2 and one preform was used without sintering. Sintered preforms and the unsintered preform were preheated at 1200°C for 30 min. in H_2 and hot forged in a 51 mm dia. die by applying 525 $MN.m^{-2}$ pressure. All mechanical properties were measured in the as-forged condition and after annealing at 700°C for 1 hour in H_2.

Different processing routes of powder preform forging have been investigated throughout the development of powder preform forging. Skelly[74] reported that, following six different sequences, the best properties were obtained from the material which had followed a sintering and hot forging sequence. It was also pointed out that combining the sintering and hot forging preheat step in one operation, followed by forging,was the most promising procedure with respect to economy, simplicity, and properties. Maclean et al[112] studied the mechanical properties of powder metal forgings as a function of processing route and showed that the mechanical properties of the sinter-forged material (preform-sinter-cool-preheat-forge) were superior to those of the powder forged material (preform-preheat-forge), especially where impact strength was concerned.

Both materials investigated in this present work showed similar effects. Fig. 9.18(a) shows that the tensile properties were markedly increased with increasing sintering

Table 9.IX. The Effect Of Preform Sintering Time Prior To Hot Forging On Mechanical Properties Of Forgings.

Material	Sintering Time (hrs)	*Condition	Elong-ation (%)	Y.S. ($MN.m^{-2}$)	U.T.S. ($MN.m^{-2}$)
Sponge Iron Pellets	-	F	22	333	405
		A	26	325	391
	1	F	20	317	416
		A	21	372	441
	2	F	23	324	433
		A	18	429	494
	3	F	20	357	476
		A	15	428	501
Höganäs NC 100.24 Iron Powder	-	F	23	261	321
		A	27	237	313
	1	F	25	278	370
		A	25	290	358
	2	F	21	304	403
		A	19	316	419
	3	F	20	341	428
		A	18	381	445

* F: as-forged, A: as-forged and annealed.

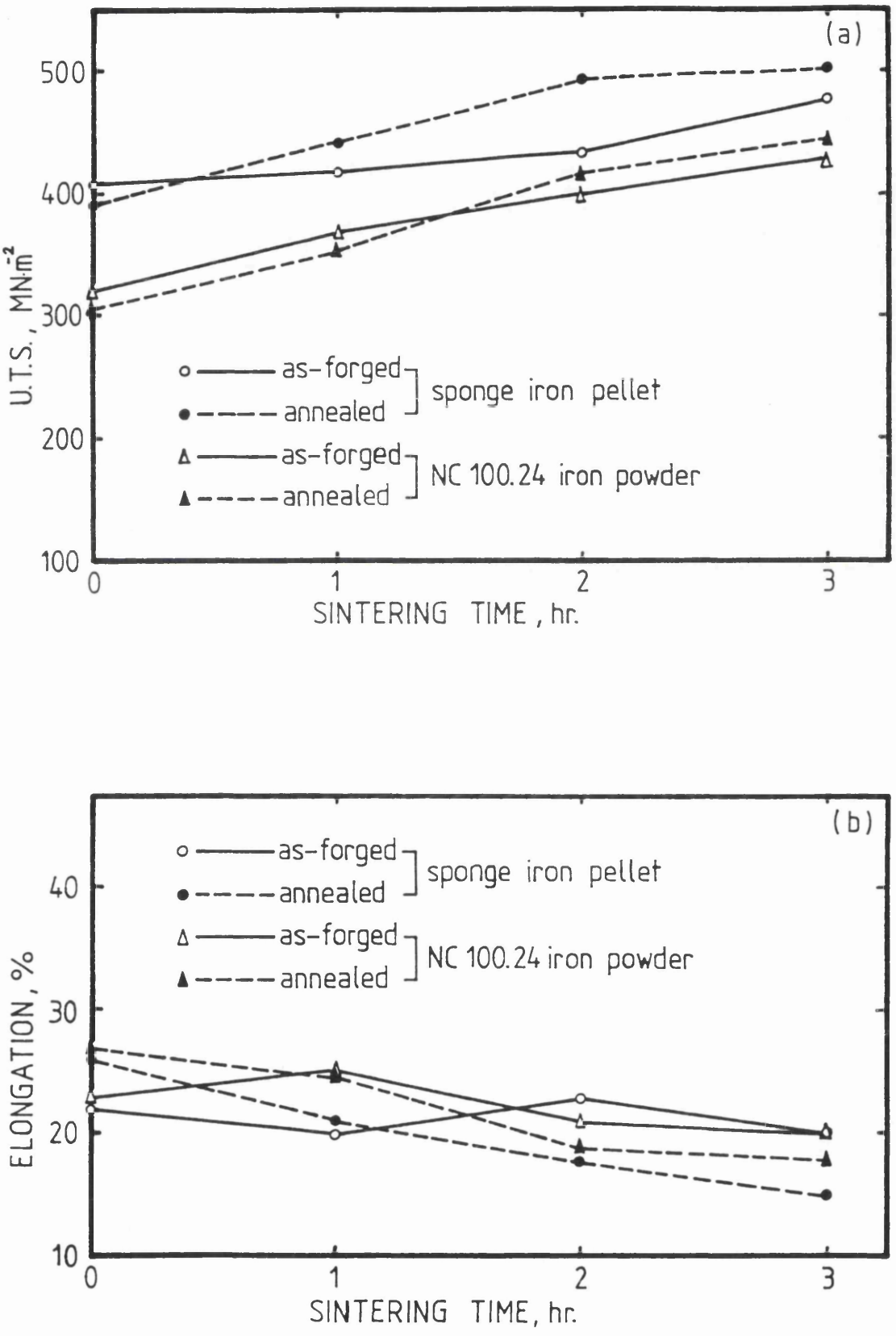


Fig. 9.18. Effect of sintering on the mechanical properties of forgings.

time, whereas (see Fig. 9.18(b)) the elongation values were decreased slightly - all forgings in the as-forged condition. As was explained by Maclean,[112] this effect could be due to the retained oxygen content of the forgings. When the preforms were sintered, the oxide film in the porous pellets was reduced by the active sintering atmosphere of H_2. As the sintering time increased, the oxide film in the pellet was decreased while the unreducible oxides, which were already present in the pellets as impurities, were retained in the forgings. However, overall oxygen content was decreased with increasing sintering time. This can be seen in Fig. 9.18(a) where after two hours sintering the relative increase in U.T.S. was decreased. This could mean that the impurities in the forgings had become the dominant effect. Another possible explanation was that even though all the forgings were of the same density, greater deformation had occurred with the increase in sintering time. This is because increasing sintering time promotes better metal-to-metal contact, causing greater fragmentation of the surface oxide which is then finely distributed in the final forging.

9.4. EFFECT OF ANNEALING ATMOSPHERE ON THE MECHANICAL PROPERTIES OF FORGINGS

The results obtained from density measurements show that hot forged products contained a small number of residual pores (see next section). In addition to this, it was found that the mechanical properties of forgings increased after annealing in H_2 for 1 hour. Therefore, some experiments were carried out to investigate the effect of annealing atmosphere on these properties. Additional annealing

treatments were as follows:

(i) in Argon for 1 hour at 700°C.

(ii) in vacuum at 0.075 torr (10 Nm^{-2}) for 1 hour at 700°C.

The results are given in Table 9.X and for comparison the results for the as-forged condition after annealing in H_2 for 1 hour at 700°C are included.

The results show the general trend that U.T.S. and elongation both increase with annealing for all conditions used. Vacuum annealing gave higher U.T.S. values than the other two annealing treatments in most of the results. Annealing in argon gave higher U.T.S. values than those obtained by annealing in hydrogen in about 70% of the results. The effect on elongation of these treatments was compared to the as-forged results. The elongation values for H_2 annealed specimens are equal to or higher than those of specimens in as-forged condition. An increase in elongation occurred in most of the specimens annealed in argon and in about half of the specimens annealed in vacuum.

The reason for these results is not obvious, but the effect of annealing (in H_2) is dealt with in some detail in the next chapter.

9.5. EFFECT OF HOT-COMPACTION PRESSURE ON THE PROPERTIES OF FORGINGS

Porosity is undesirable in powder metallurgy structural components since it reduces the load-bearing area and causes stress concentrations leading to early failure. Powder forging is one of the techniques used to minimise or eliminate

Table 9.X. The Effect Of Annealing Atmosphere On The Mechanical Properties Of Forgings.

Material	Preform Dia.(mm)	Preform Density (% theo.)	Y.S. ($MN.m^{-2}$)				U.T.S. ($MN.m^{-2}$)				Elongation (%)			
			As-forged.	Annealed In			As-forged.	Annealed In			As-forged.	Annealed In		
				H_2	Ar_2	Vacuum		H_2	Ar_2	Vacuum		H_2	Ar_2	Vacuum
Sponge Iron Pellets	38	50	378	382	378	482	460	471	463	517	17	22	19	15
		71	321	325	347	331	397	401	413	395	21	25	21	19
		85	247	225	239	253	333	327	337	344	22	26	21	25
	51	50	352	359	348	373	440	453	463	487	18	23	19	17
		70	319	322	341	384	416	441	447	451	16	19	18	13
		84	295	281	277	282	363	355	321	343	19	25	27	22
NC 100.24 Iron Powder Pellets	38	51	351	345	422	445	424	413	474	495	17	20	21	14
		70	330	341	333	358	435	447	437	451	18	20	19	18
		83	235	220	224	213	315	310	321	303	24	27	23	28
	51	49	318	372	361	459	423	411	457	501	17	20	20	15
		69	317	381	323	377	417	422	426	451	17	20	19	17
		85	255	252	261	283	334	324	317	378	24	28	20	18
NC 100.24 Iron Powder	38	70	245	252	247	237	349	317	351	334	18	28	18	21
		86	242	197	215	195	343	303	317	288	17	29	21	27
	51	72	269	271	293	307	309	315	303	377	15	31	20	17
		84	265	226	256	262	331	315	315	347	18	26	24	22

this porosity. In powder forging the porosity of the forging is usually controlled by the compaction pressure and forging temperature.

In the present work a single forging temperature of 1100°C was used as mentioned earlier. Giving due consideration to the other parameters involved an experimental procedure was determined to maximise the use of the 1.072 MN hydraulic press, and a series of experiments carried out to evaluate the effect of the variables involved on the porosity of the final forgings. The results obtained are given in Table 9.XI.

The density of pure iron, or that of low carbon mild steel, was not used in the calculations because of the presence of unreducible oxides. The maximum density attainable in the solid forging was calculated from the chemical analysis of the superconcentrate, assuming that all elements except iron are present as unreducible pure oxides, and all the iron oxides present in the superconcentrate had been reduced. The value thus obtained was 7.769 $g.cm^{-3}$ as shown in Table 9.XII.

From the results in Table 9.XI it was found that the final densities of forgings varied between 99.25 - 99.97% of theoretical. Therefore, very high densities were achieved in this experiment. Although all the materials showed similar final densities for a given set of conditions, what difference in porosity existed in each material was mainly affected by the preform density. Generally the porosity content of each material tended to decrease with increasing preform density. Porosity level was less effected by the

Table 9.XI Density And Porosity Of Forgings.

Material	Preform Diameter (mm)	Preform Density (% theo.)	Final Density		Porosity (% vol.)
			($g.cm^{-3}$)	(% theo.)	
Sponge Iron Pellets	38	50	7.734	99.55	0.45
		71	7.746	99.70	0.30
		86	7.767	99.97	0.30
	51	50	7.748	99.73	0.27
		73	7.759	99.87	0.13
		86	7.761	99.90	0.10
NC 100.24 Iron Powder Pellets	38	51	7.751	99.73	0.27
		73	7.714	99.25	0.75
		85	7.762	99.87	0.13
	51	50	7.736	99.54	0.46
		71	7.740	99.59	0.41
		84	7.769	99.96	0.04
NC 100.24 Iron Powder	38	69	7.749	99.70	0.30
		85	7.764	99.90	0.10
	51	70	7.765	99.91	0.09
		86	7.758	99.82	0.18

Table 9.XII. Chemical And Size Analysis Of The Magnetite Super-Concentrate And Calculation Of The Theoretical Volume % Of Non-Metallic Inclusions Which Would Remain In The Forged Disc.

Constituent	Chemical analysis of the magnetite super-concentrate wt%	Size analysis of the magnetite super-concentrate: Size μm	Size analysis of the magnetite super-concentrate: Cumulative wt%	Anal. of redcd sp. iron on basis of 100% chem. reaction wt%	Density $g.cm^{-3}$	Vol. of constituents per 100 g of sponge iron c.c.
Fe	71.900	+ 74	0.6	98.972	7.87	12.576
Fe_3O_4	97.000	+ 62	1.1	-	-	-
Fe_2O_3	2.300	+ 44	9.7	-	-	-
SiO_2	0.060	+ 25	57.2	0.083	2.65	0.031
MgO	0.150	+ 20	69.0	0.206	3.58	0.058
Al_2O_3	0.150	+ 16	75.2	0.206	3.97	0.052
TiO_2	0.140	+ 11	84.1	0.193	4.26	0.045
V_2O_5	0.200	+ 5	94.3	0.275	3.36	0.082
Na_2O	0.023	+ 1	99.1	0.032	2.27	0.014
K_2O	0.015	< 1	0.9	0.021	2.32	0.009
P	0.004	-	-	0.006	2.34	0.003
Cu	0.005	-	-	0.007	8.92	0.001
Σ 72.647				100.001		12.871

Total volume of non-metallic inclusions = Volumes of SiO_2, MgO, Al_2O_3, TiO_2, V_2O_5, Na_2O and K_2O

$$= 0.031 + 0.058 + 0.052 + 0.045 + 0.082 + 0.014 + 0.009 = 0.291$$

Volume percentage of the non-metallic inclusion $= \frac{0.291}{12.871} = 2.26$

Theoretical density of the reduced sponge iron $= \frac{100}{12.871} = 7.769\ g.cm^{-3}$

type of deformation.

Having determined the density of the forgings using the above experimental conditions, a further set of experiments was conducted to investigate the effect of hot forging pressure on the final density and the mechanical properties of the sponge iron pellet forgings. The following parameters were constant: preform density of 70%, preform diameter of 38 mm, hot forging die diameter of 38 mm, final forging height to preform height ratio of H/Ho = 0.71 and preheating time of 30 min. Hot forging was carried out at 1100°C at four different pressures. For each pressure setting five forgings were made and the results are shown in Table 9.XIII.

Figure 9.19(a) shows the relationship between the density and the porosity of the forgings and hot compaction pressure. This shows that the density of the forgings increased and porosity level decreased as the hot forging pressure increased. Full theoretical density was achieved using a forging pressure of 926 MNm^{-2}.

Fig. 9.19(b) shows the relationship between U.T.S. and % elongation of the forgings and hot compaction pressure. This shows that U.T.S. values increased slightly with increased pressure but that ductility was not affected.

The above work indicated that generally tensile properties and elongation were not affected substantially by the residual porosity; therefore it was concluded that the experimental design was successful.

A review of the work done by other investigators[65,67-70] highlighted impact strength as being the most significantly

Table 9.XIII. Effect Of Hot Forging Pressure On Density And Mechanical Properties.

Hot Forging Pressure ($MN.m^{-2}$)	Final Density		Porosity (% vol.)	Condition*	Y.S. ($MN.m^{-2}$)	U.T.S. ($MN.m^{-2}$)	Elongation (%)
	($g.cm^{-3}$)	(% theo.)					
463	7.747	99.72	0.28	F	320	396	24
				A	295	376	25
618	7.759	99.87	0.13	F	333	411	24
				A	384	440	24
772	7.767	99.97	0.03	F	345	424	24
				A	390	440	24
926	7.769	100.00	0.00	F	358	433	24
				A	421	459	24

* F: as-forged, A: as-forged and annealed.

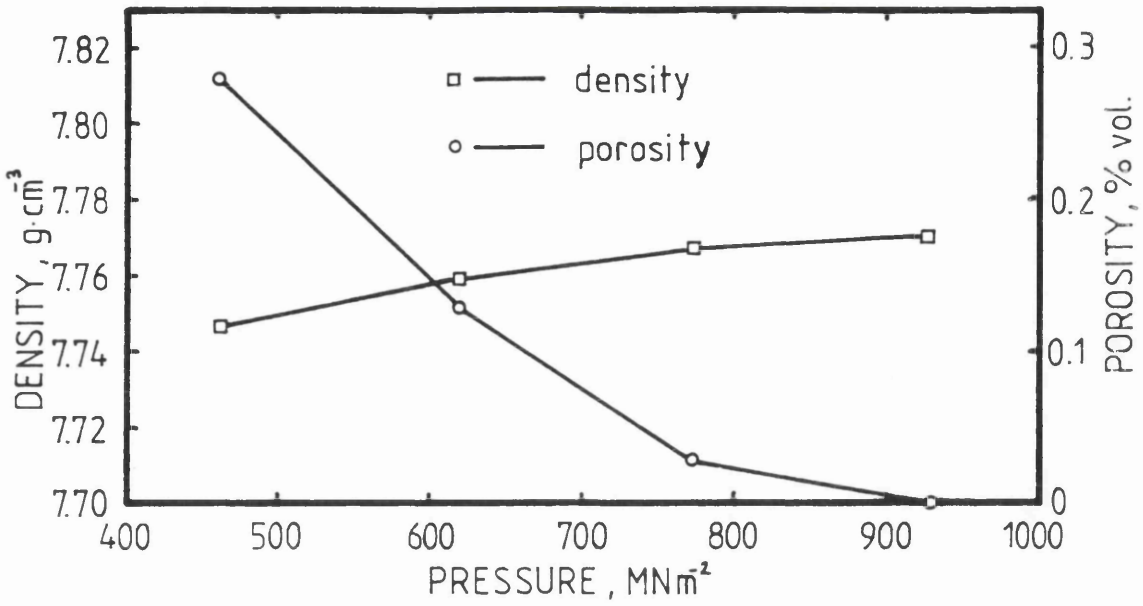


Fig. 9.19(a). Effect of hot-forging pressure on the density of the forgings.

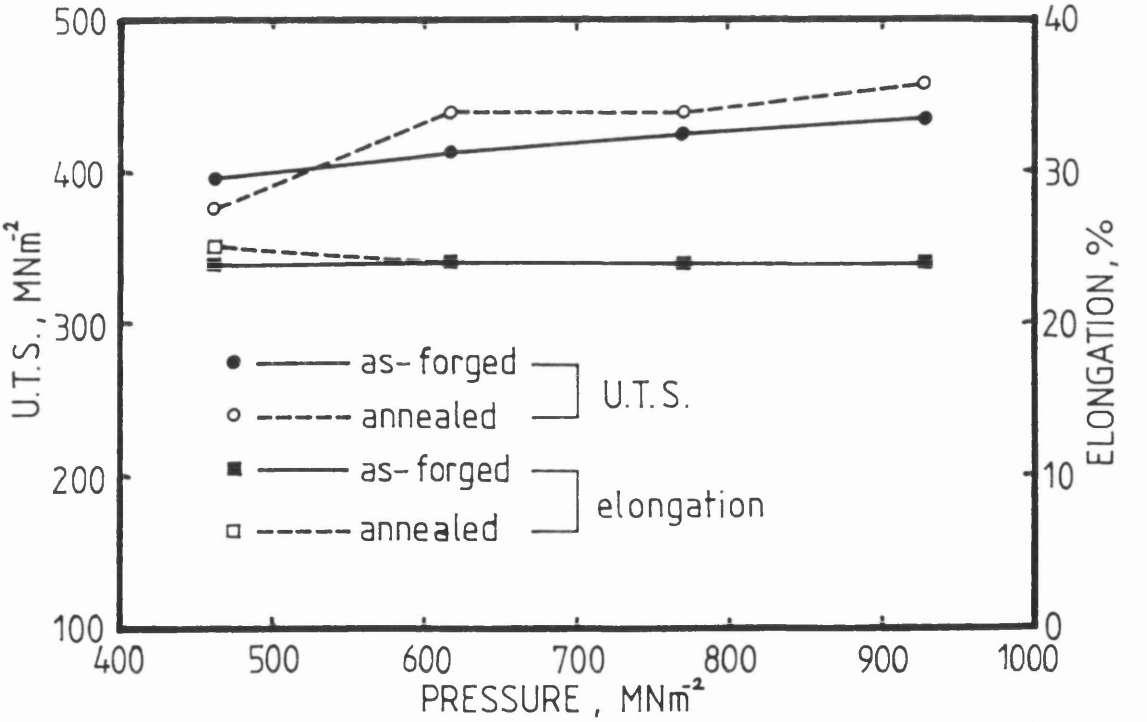


Fig. 9.19(b). Effect of hot-forging pressure on the mechanical properties of forgings.

affected property by residual porosity, with U.T.S. and elongation being less affected particularly at low levels of residual porosity. Hence, when impact resistance is important, forging to full density is essential. A final set of experiments was thus made to examine impact strength, U.T.S. and elongation, taking due note of the results obtained so far. (see Section 7.3.3.4.) A list of the variables investigated and the results are given in Table 9.XIV. The density of the final forgings was determined by the immersion technique and the results showed that full density was achieved with all forgings. In Figs. 9.20 - 9.25 the mechanical properties are plotted against preform density.

Figs. 9.20 and 9.21 show that the U.T.S. of all forgings (as-forged and annealed) increased with decreasing preform density. The highest strength was achieved with sponge iron pellet forgings and the lowest from iron powder forgings. U.T.S. values increased substantially following annealing, especially with low preform density forgings of sponge iron pellets.

Figs. 9.22 and 9.23 show that the ductility of all forgings increased with increasing preform density. Although the strength of forgings increased after annealing, the ductility of forgings was not affected significantly.

It has been shown[51, 113-115] that a certain amount of material flow is beneficial to densification and in many cases even indispensable for reaching optimum properties. Several reasons have been suggested for this. Sliding contact promotes bonding across pore surfaces and can break

Table 9.XIV. Mechanical Properties Of Forgings (Hot-Repressed Preform Forging).

Material	Preform Density (%theo.	Compaction Pressure ($MN.m^{-2}$) Preform	Hot-forging	True Strain (%)	Con-* dition	Y.S. ($MN.m^{-2}$)	U.T.S. ($MN.m^{-2}$)	Elong-ation (%)	Izod Impact Strgth. (J)
Sponge Iron Pellets	49	85	1249	0.78	F	408	521	11	11
					A	589	655	12	8
	60	170	1195	0.59	F	380	465	14	16
					A	539	603	9	6
	69	255	1258	0.44	F	385	452	15	11
					A	514	558	13	6
	75	371	1236	0.35	F	252	378	17	18
					A	331	470	19	16
	79	510	1180	0.30	F	252	368	23	20
					A	362	460	16	19
	92	633	1252	0.16	F	226	311	18	27
					A	245	348	26	25
NC 100.24 Iron Powder Pellets	50	85	1198	0.79	F	326	386	15	14
					A	389	442	20	15
	61	170	1252	0.61	F	331	416	16	20
					A	490	563	13	8
	64	185	1258	0.48	F	300	390	16	20
					A	461	517	19	20
	72	255	1258	0.38	F	303	394	17	29
					A	454	533	13	24
	80	371	1242	0.30	F	225	315	21	23
					A	245	382	25	20
	96	772	1258	0.11	F	199	288	27	29
					A	220	313	31	15
NC 100.24 Iron Powder	61	170	1249	0.59	F	297	383	19	15
					A	353	468	17	15
	70	255	1246	0.46	F	278	341	22	15
					A	339	427	15	18
	76	371	1189	0.37	F	235	330	21	10
					A	262	363	22	11
	89	695	1205	0.18	F	198	284	25	20
					A	219	303	24	22

* F: as-forged, A: as-forged and annealed.

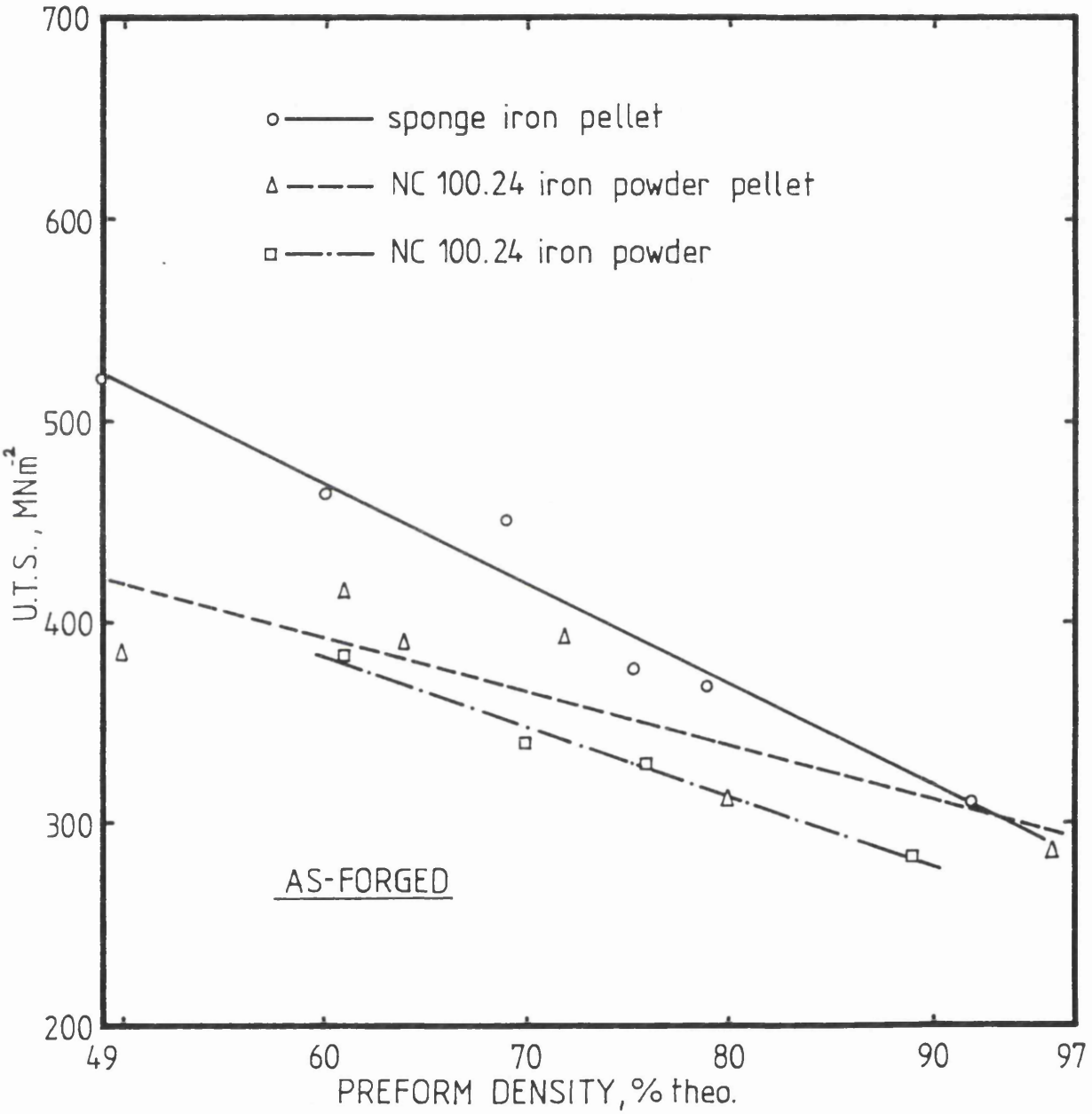


Fig. 9.20. Effect of preform density on the U.T.S. of the forgings.

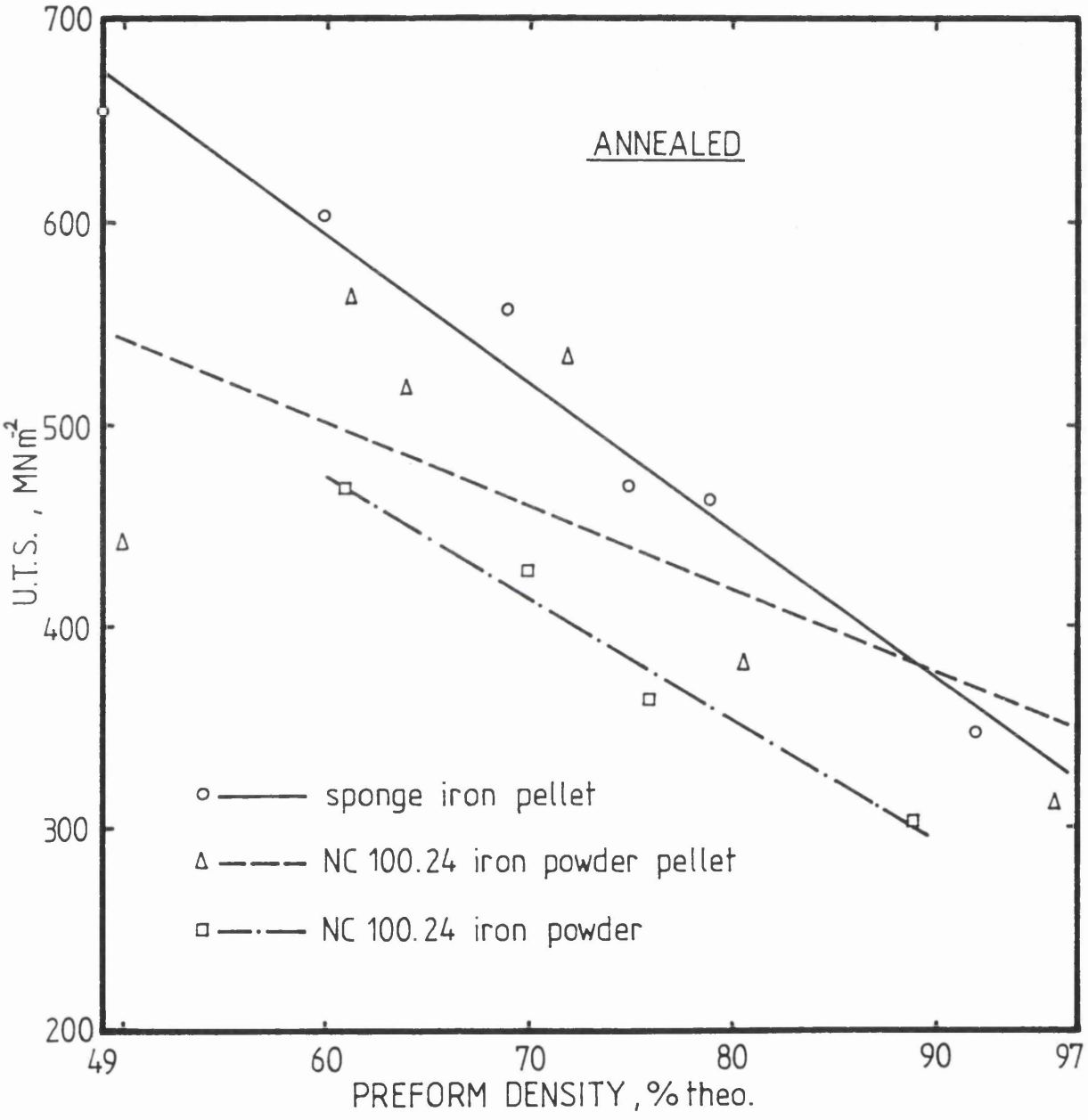


Fig. 9.21. Effect of preform density on the U.T.S. of the forgings.

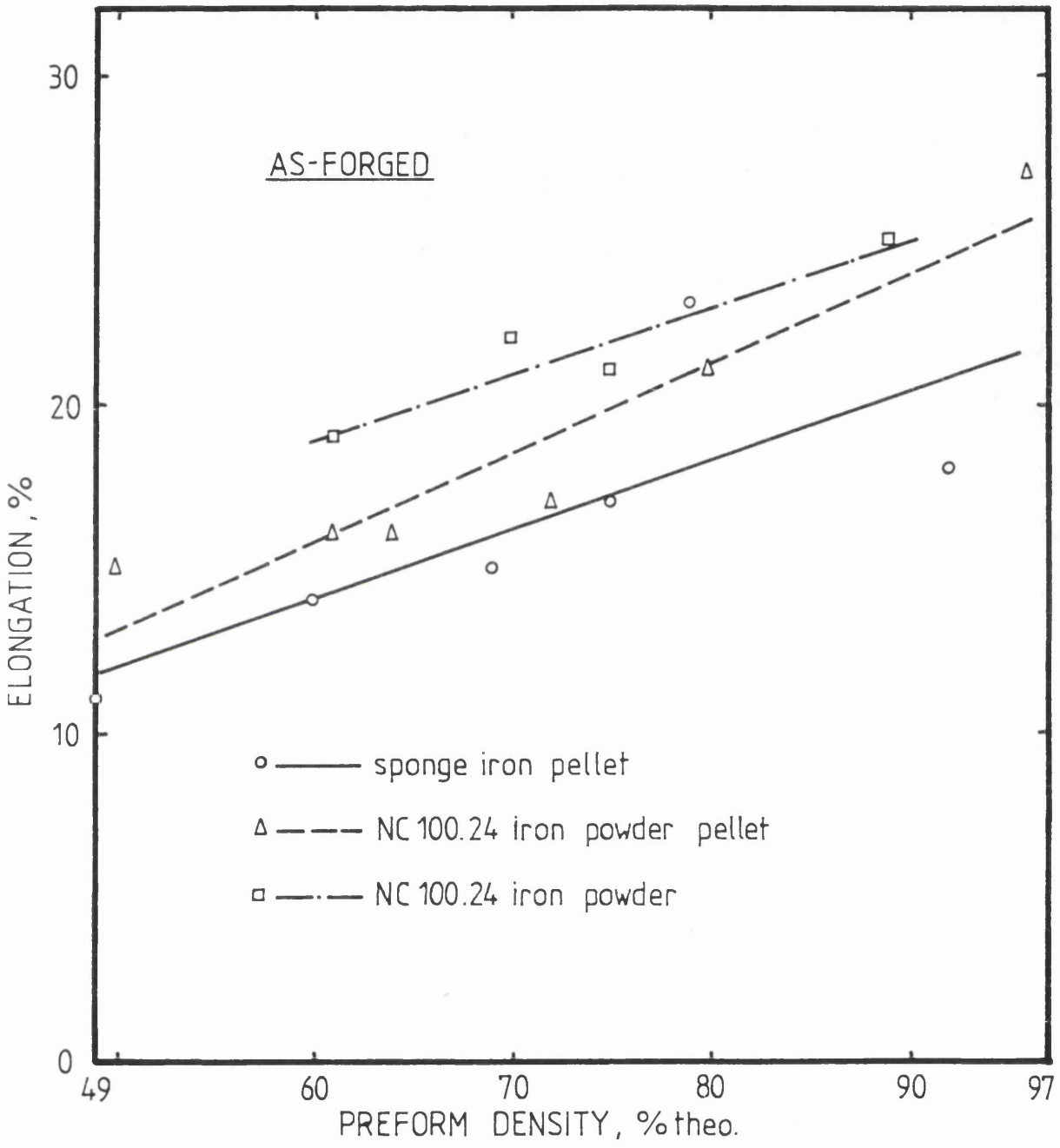


Fig. 9.22. Effect of preform density on the ductility of the forgings.

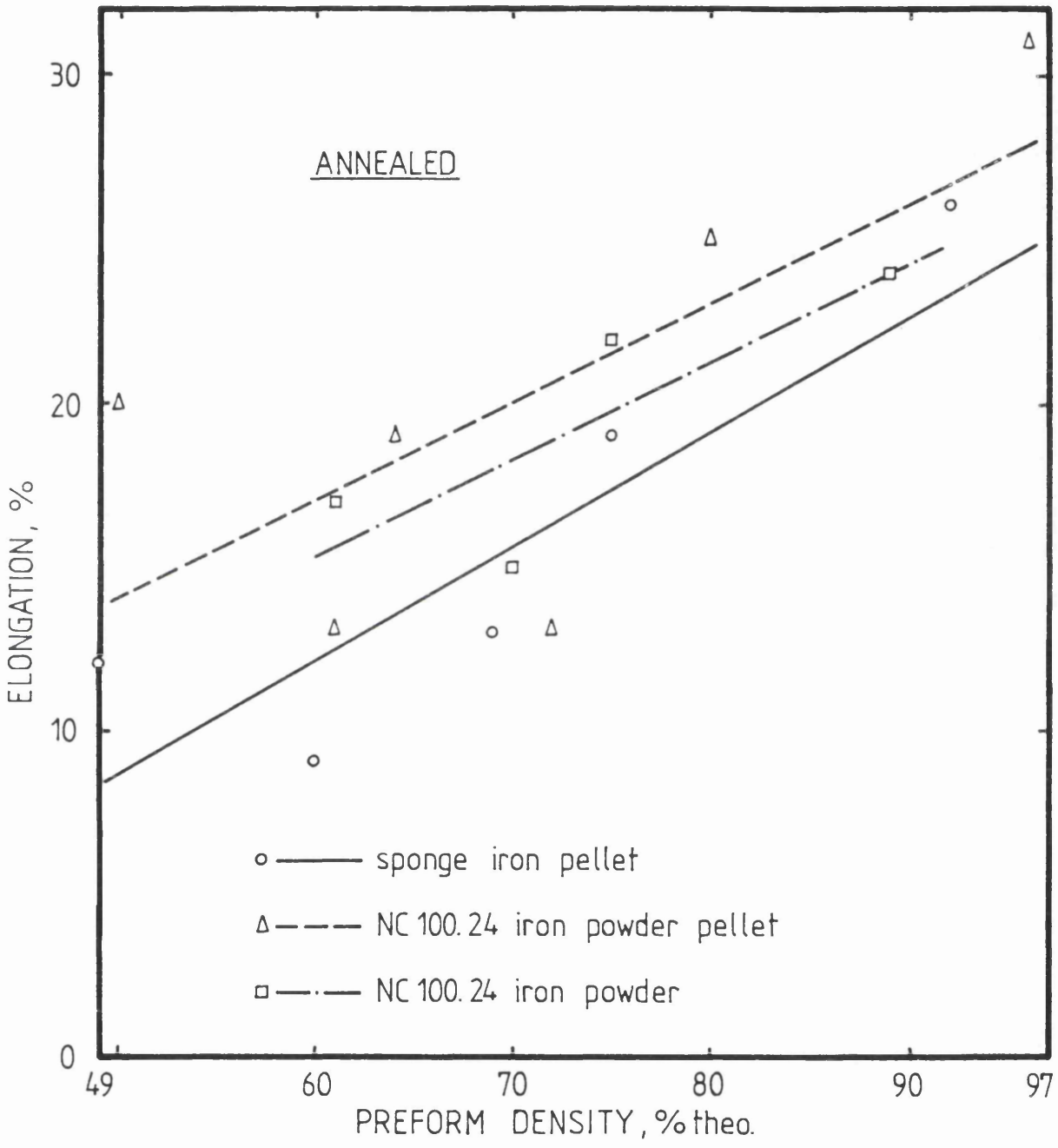


Fig. 9.23. Effect of preform density on the ductility of the forgings.

oxide films. Pore geometry may be changed so that pores collapse more readily under compressive stress. A sponge iron pellet preform consists of a convoluted spongy mass, the constituent parts of which suffer major plastic deformation during forging, even though the overall physical dimensions are not extended in any direction. Thus, this type of deformation was more effective for lower density preform forgings. Structural examinations (see next chapter) however, showed that lower preform density forgings produced finer grain and inclusion sizes, and this was related to the eventual attainment of improved mechanical properties.

Figs. 9.24 and 9.25 show the relation between the impact strength and preform density of forgings in the as-forged and annealed conditions respectively. Although there was a tendency towards improved impact strength with increasing preform density, the results for all materials were scattered between 6-29J. Hirschhorn and Bargainner[116] found similar effects for sponge iron powder forgings, suggesting that the reason for this was the presence of impurities which had the major effect on impact strength since the forgings were free of porosity.

9.6. FORGING OF ATOMISED IRON POWDER PELLET AND ATOMISED IRON POWDER PREFORMS

In order to provide a comparison with the relatively high non-metallic content of the iron powder used in this work, it was decided to produce forgings under identical conditions but using relatively clean ASC 100.29 atomised iron powder (see Table 7.III) as the starting material.

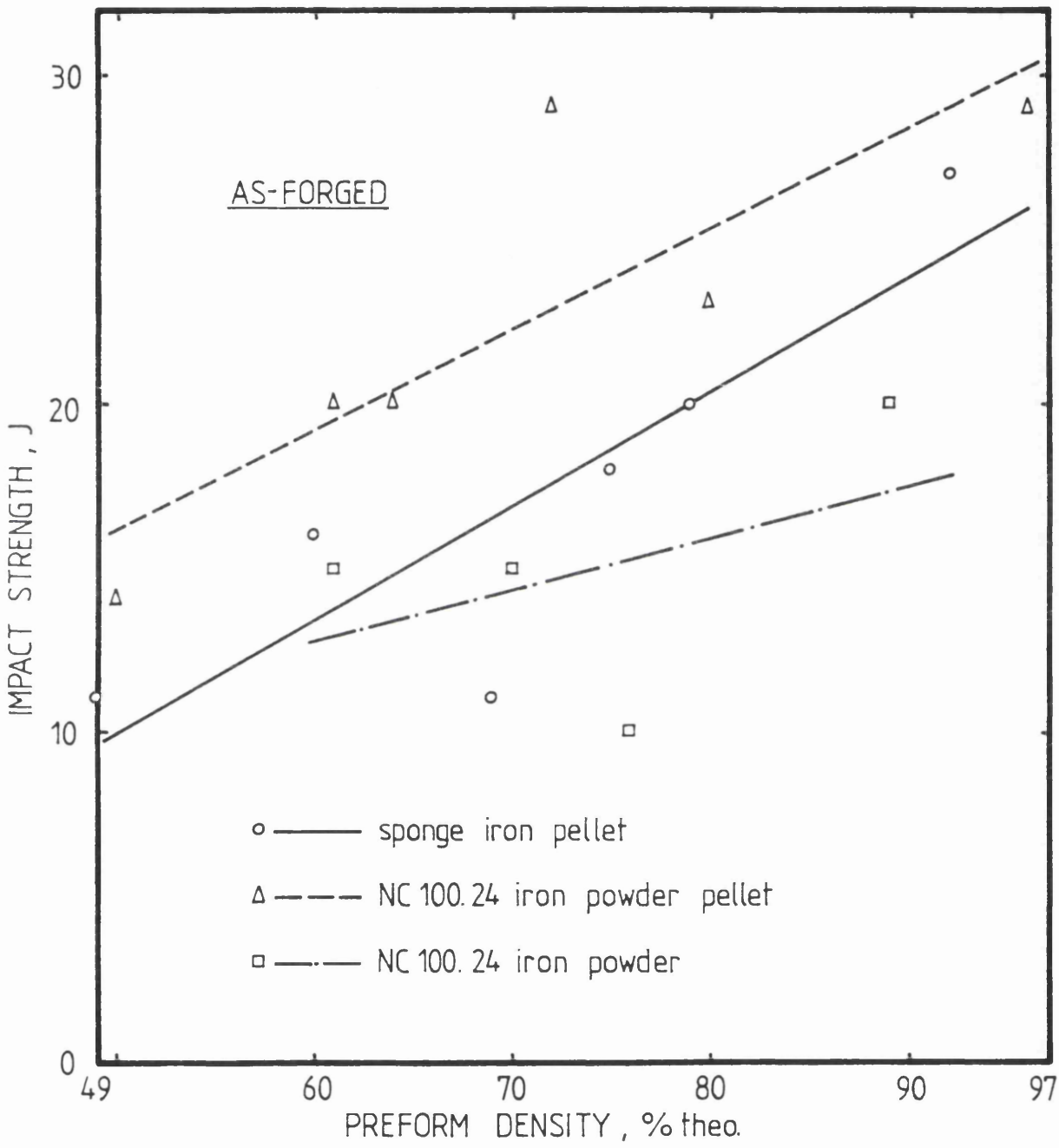


Fig. 9.24. Effect of preform density on the impact strength of the forgings.

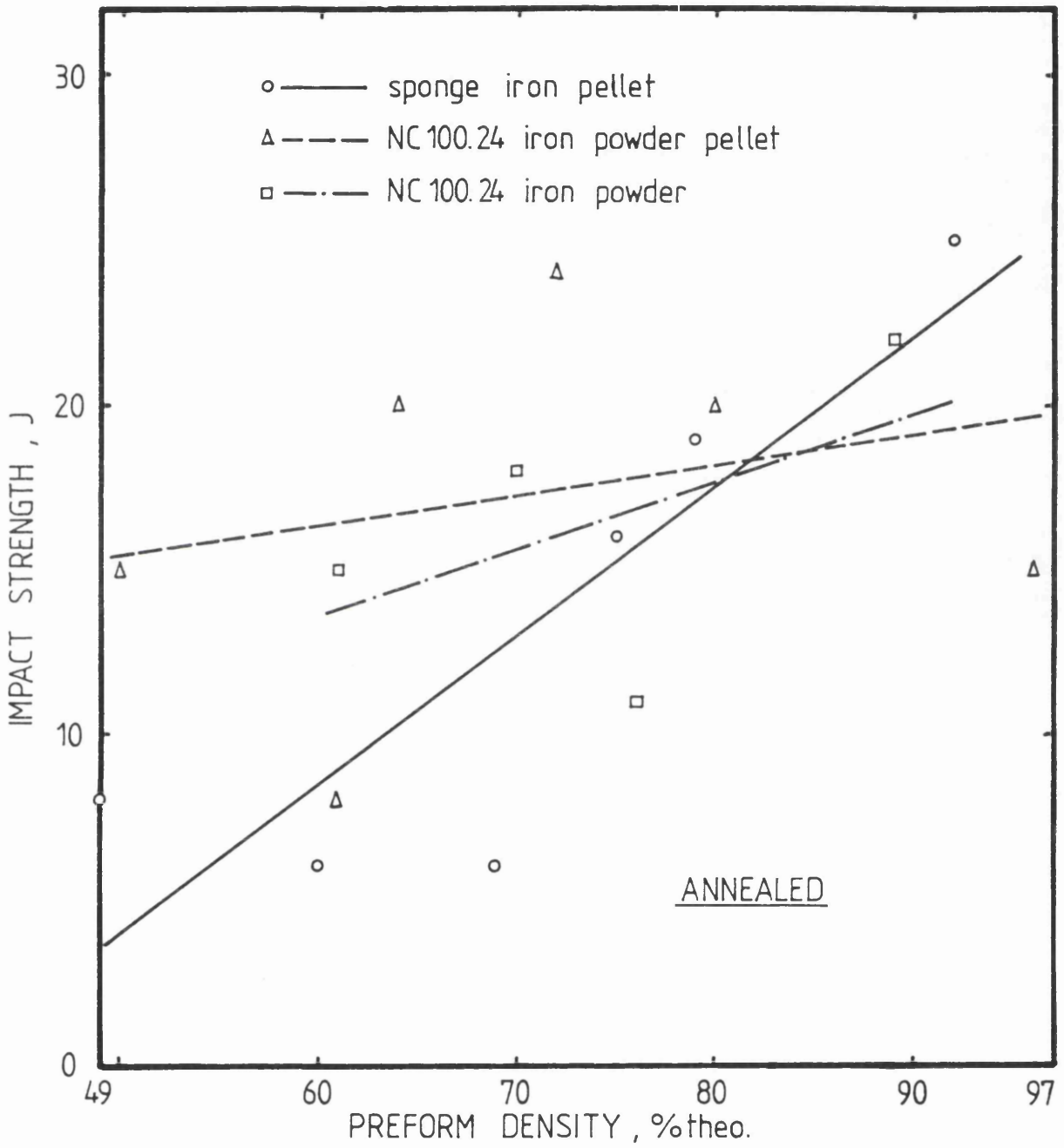


Fig. 9.25. Effect of preform density on the impact strength of the forgings.

The mechanical properties obtained from these forgings are listed in Table 9.XV and shown graphically in Figs. 9.26 and 9.27. These show that for both methods of production, both in the as-forged and annealed conditions, U.T.S. values decreased and the % elongation increased with increasing preform density, showing a similar trend as in the main investigation results. Similar levels of U.T.S. and elongation were obtained for both types of forgings with similar preform densities, and only in the case of the 50% preform density pellet forging did annealing have any significant effect, increasing U.T.S. and lowering elongation.

Microscopic examination of the 50% preform density pellet forgingsshowed residual porosity and iron oxide inclusions. Compared to the sponge iron pellets, these pellets were relatively dense, thus the production of a 50% preform density required only a relatively loosely packed preform, with large voids between the pellets, the pellets themselves bonded weakly. Thus, when the preform was transferred at forging temperature to the die, oxygen penetrated into the pore labyrinth, resulting in an increase in the non-metallic inclusion in the forging. (see Fig.9.28) Presumably, the original large voids between pellets and oxidation of the voids caused the increase in the final forged porosity level. As explained in detail in Chapter 10, the increase in non-metallic content of the 50% preform density pellet forgings is the cause of the high U.T.S. values obtained and the effect of annealing.

Table 9.XV. Mechanical Porperties Of ASC 100.29 Atomised Iron Powder Pellet and ASC 100.29 Atomised Iron Powder Preform Forgings.

Material	Preform Density (% theo.)	Condition.*	Y.S. ($NM.m^{-2}$)	U.T.S. ($MN.m^{-2}$)	Elongation (%)
ASC 100.29 Atomised Iron Powder Pellets	50	F	322	416	25
		A	433	453	17
	68	F	239	330	35
		A	291	325	35
	86	F	184	295	36
		A	246	299	37
ASC 100.29 Atomised Iron Powder	67	F	256	344	35
		A	298	341	35
	88	F	194	291	37
		A	256	312	39

* F : as-forged, A : as-forged and annealed.

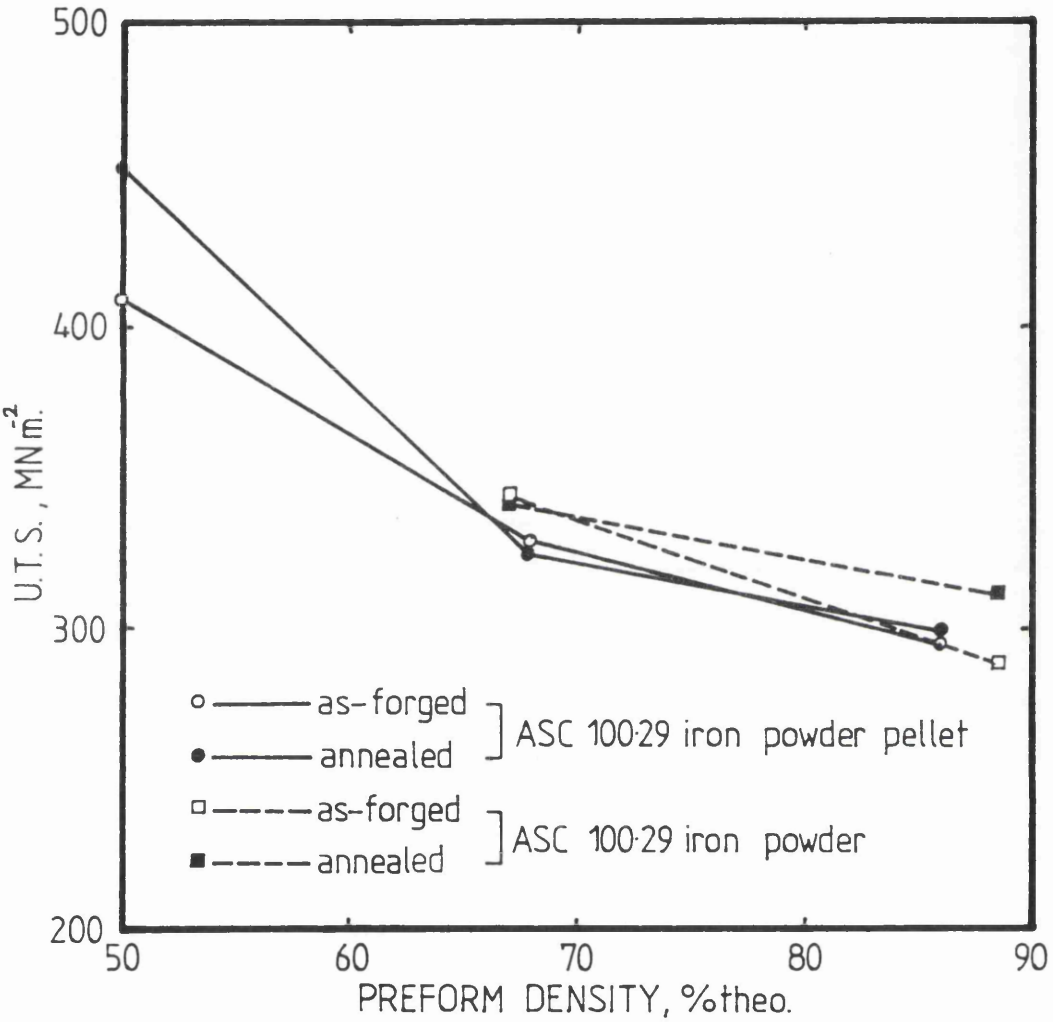


Fig. 9.26. Effect of preform density on the U.T.S. of forgings from atomised iron powder pellets and atomised iron powder.

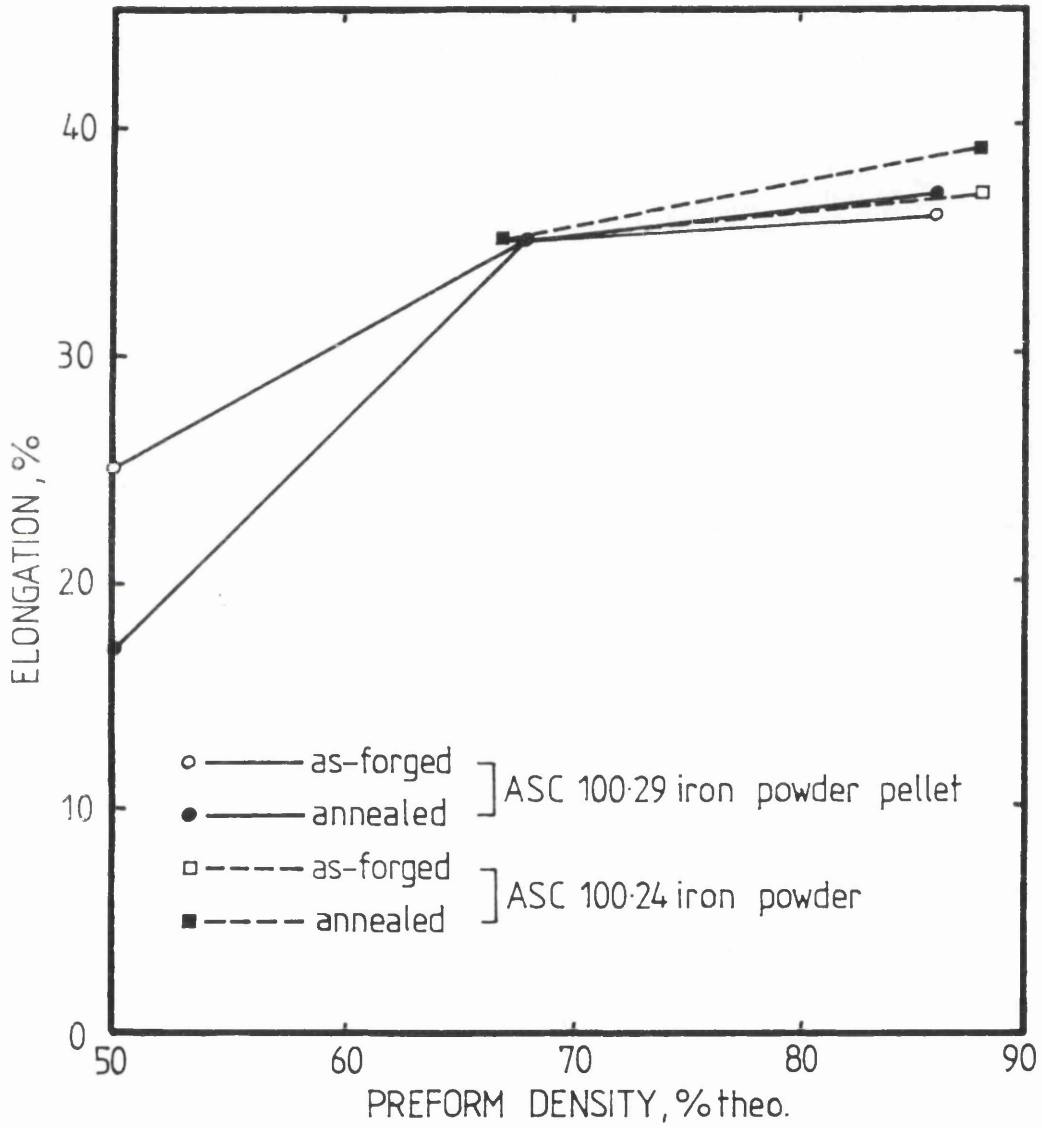
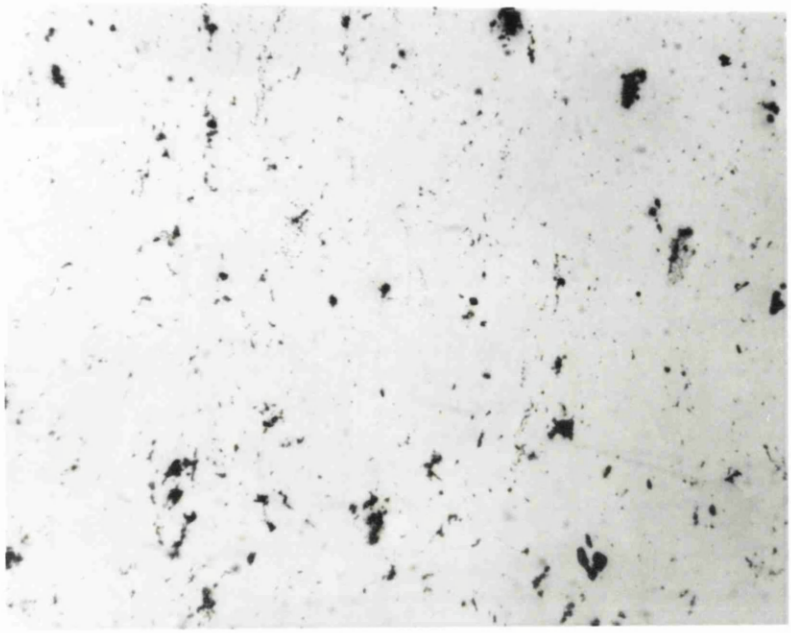
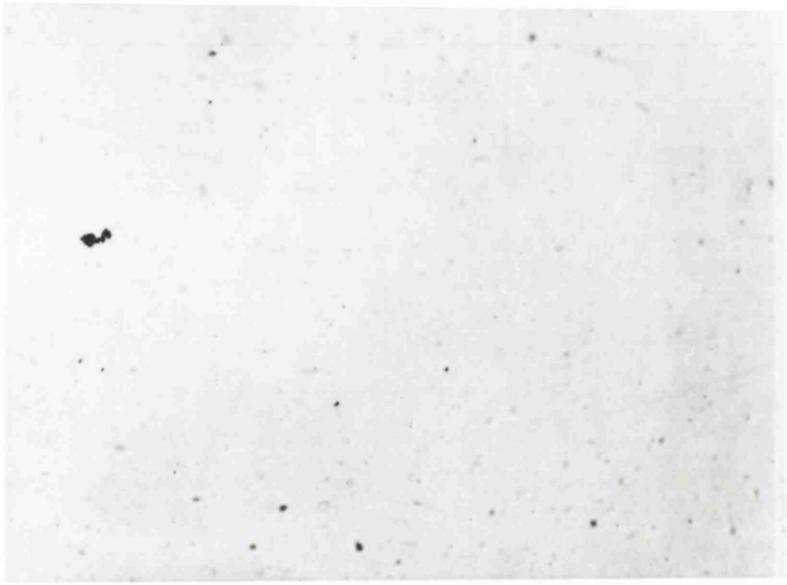


Fig. 9.27. Effect of preform density on the ductility of forgings from atomised iron powder pellets and atomised iron powder.

Fig. 9.28. ASC 100.29 atomised iron powder pellet forgings produced from preforms of
(a) 50% theo.
(b) 68% theo. density
X110



a



b

CHAPTER 10

MICROSTRUCTURAL PROPERTIES OF SPONGE IRON PELLET FORGINGS

In the previous chapters a processing route has been determined for the production of forgings with optimum properties from magnetite superconcentrate powder. Although full density, porous-free, forgings were produced, the density was lower than that for pure iron (or steel) due in the main to the presence of a large number of low density non-metallic inclusions which were in the starting magnetite superconcentrate, and unreducible during the reduction treatment.

Microscopic examination of these forgings showed that the inclusions were small and uniformly distributed; average grain size was also small. Annealing caused grain refinement.

The forgings were, therefore, examined microscopically in some detail to determine the origin, nature, amount, size and size distribution of the non-metallic inclusions, the grain structure and grain size of the forgings. The effect of structure on the mechanical properties was also examined.

10.1. ORIGIN, NATURE, AMOUNT, SIZE AND SIZE DISTRIBUTION OF NON-METALLIC INCLUSIONS

10.1.1. ORIGIN AND NATURE

The main impurities in the magnetite superconcentrate were SiO_2, MgO, Al_2O_3, TiO_2, V_2O_3 and these were present in the following manner.

(i) SiO_2, mainly as free quartz, and also as silicate particles.

(ii) Na_2O, K_2O, associated with mica and feldspar particles.

(iii) MgO, Al_2O_3, TiO_2 and V_2O_3, in the magnetite lattice.

It follows from the Ellingham diagram that these oxides are not reducible under the reduction conditions employed in this investigation. Thus, except for a few inclusions, which might be the result of poor handling or forging conditions, almost all inclusions present in the forgings originate from the above-mentioned unreducible impurities. During the reduction of iron oxide, the impurities separated from the spinel lattice in the form of fine particles. Some chemical reactions between these oxides can occur on heating, without fusion. Table 10.1. shows the temperature at which noticeable reaction will take place between some oxides of interest.[117] The lower temperature of the range shown is for a small particle size, while the higher temperature is for a large particle size. From these values it seems likely that some of the unreducible oxides present in the superconcentrate might react to some extent with each others, forming mixed and complex oxides. Similarly, the possibility of reaction between FeO and some unreducible oxides during the reduction cannot be entirely ruled out. It is not possible to predict the exact nature of the chemical reaction occurring because of the lack of a detailed investigation. However, a qualitative indication can be obtained from the analysis of these inclusions. A preliminary investigation showed that the non-metallic inclusions present in the finished forgings were of complex and mixed oxide types, and therefore it seems that some reaction between the oxides took place.[118]

Table 10.I. Temperature at which noticeable reaction will occur between some oxides of interest.

Reactants	Temperature of noticeable reaction (^{o}C)	Product
SiO_2+MgO	1200-1400	Mg_2SiO_4
SiO_2+Al_2O_3	1300-1500	$3Al_2O_3.2SiO_2$
Al_2O_3+MgO+SiO_2	1400	$2MgO.2Al_2O_3.5SiO_2$

10.1.2. AMOUNT, SIZE, AND SIZE DISTRIBUTION OF INCLUSIONS

The distribution of volume percentage of non-metallic inclusions in sponge iron pellet forgings, NC 100.24 iron power pellet forgings and NC 100.24 iron powder forgings are given in Tables 10.II, 10,III, and 10.IV and shown in Figs. 10.1, 10.2, and 10.3. The mean volume percentage of non-metallic inclusions in the hot forged and annealed forgings are also given in Tables 10.II, 10.III and 10.IV. The relationship between the mean volume percentage of non-metallic inclusions in the forgings and the preform density is given in Fig. 10.4. The number and size distribution of the inclusions present in the forgings are shown in Tables 10.V and 10.VI, and some of these results are represented schematically in Figs. 10.5 - 10.9. It is seen that the number of inclusions greater than 9.6μm in length was relatively small, also that the most commonly occurring length of inclusions in the forgings was 1.2μm. It can be seen from Fig. 10.9 that the size distribution of inclusions was positively skewed, most of the inclusions being of micron and sub-micron size, and relatively few inclusions in the maximum size range.

Table 10.II. Distribution Of Volume % Of Inclusions In The Hot Forged And Annealed Sponge Iron Pellet Preforms.

Preform Density (% theo.)	Inclusion Frequency In These Given Inclusion Content Intervals, %																		Inclusion Content (Vol. %)
	0-	0.4-	0.8-	1.2-	1.6-	2.0-	2.4-	2.8-	3.2-	3.6-	4.0-	4.4-	4.8-	5.2-	5.6-	6.0-	6.4-	6.8-	7.2-	
49	-	5.2	19.0	13.8	25.9	12.1	12.1	6.9	1.7	1.7	-	-	-	1.7	-	-	-	-	-	1.85±0.87
60	-	4.3	12.9	25.7	25.7	14.3	11.4	4.3	-	1.4	-	-	-	-	-	-	-	-	-	1.75±0.60
69	-	8.2	18.0	21.3	23.0	13.1	9.8	3.3	-	-	-	-	3.3	-	-	-	-	-	-	1.73±0.85
75	-	2.0	6.1	10.2	30.6	30.6	14.3	-	-	4.1	-	2.0	-	-	-	-	-	-	-	2.06±0.71
79	-	-	-	15.2	37.9	22.7	6.1	9.1	4.6	3.0	1.5	-	-	-	-	-	-	-	-	2.15±0.64
92	-	-	2.0	2.0	24.5	28.6	16.3	10.2	4.1	2.0	2.0	-	-	-	-	-	-	-	-	2.30±0.71

Table 10.III. Distribution Of Volume % Of Inclusions In The Hot Forged And Annealed Iron Powder Pellet Preforms.

Preform Density (% theo.)	Inclusion Frequency In These Given Inclusion Content Intervals, %																		Inclusion Content (Vol.%)
	0-	0.4-	0.8-	1.2-	1.6-	2.0-	2.4-	2.8-	3.2-	3.6-	4.0-	4.4-	4.8-	5.2-	5.6-6.0	6.4-6.8	7.6 8.0	8.0-8.4	9.6-10.0	
50	-	-	5.4	17.9	14.3	14.3	16.1	17.9	7.1	3.6	3.6	-	-	-	-	-	-	-	-	2.35±0.80
61	-	1.6	12.9	21.0	17.7	19.4	3.2	9.7	-	3.2	1.6	4.8	-	-	-	-	1.6	1.6	1.6	2.37±1.73
64	-	-	19.4	11.3	17.7	14.5	12.9	8.1	4.8	1.6	4.8	-	-	1.6	-	-	1.6	1.6	-	2.37±1.14
72	-	-	3.2	8.1	17.7	27.4	16.1	16.1	8.1	1.6	-	-	-	1.6	-	-	-	-	-	2.36±1.39
80	-	3.9	3.9	11.8	21.6	15.7	21.6	5.9	5.9	2.0	-	2.0	2.0	-	2.0	2.0	-	-	-	2.40±0.76
96	-	1.7	8.6	12.1	13.8	19.0	10.3	12.1	10.3	3.5	5.2	3.5	-	-	-	-	-	-	-	2.42±0.98

Table 10.IV. Distribution Of Volume % Of Inclusions In The Hot Forged And Annealed Iron Powder Preforms.

Preform Density (% theo.)	Inclusion Frequency In These Given Inclusion Content Intervals, %																		Inclusion Content (Vol. %)
	0-	0.4-	0.8-	1.2-	1.6-	2.0-	2.4-	2.8-	3.2-	3.6-	4.0-	4.4-	4.8	5.2-	5.6-	6.0-	6.4-	6.8-	7.2-	
61	-	3.5	6.9	19.0	17.2	13.8	10.3	12.1	3.5	3.5	5.2	1.7	1.7	-	1.7	-	-	-	-	2.36±1.11
70	-	-	12.0	17.3	17.3	20.0	8.0	5.3	8.0	1.3	2.7	2.7	2.7	1.3	-	-	1.3	-	-	2.34±1.15
76	-	-	6.4	17.5	15.9	19.1	15.9	7.9	4.8	4.8	4.8	1.6	-	-	1.6	-	-	-	-	2.37±1.00
89	-	-	7.1	16.7	18.4	21,1	13.6	4.8	5.3	2.6	1.6	2.0	-	1.6	1.6	-	1.6	-	2.0	2.41±1.56

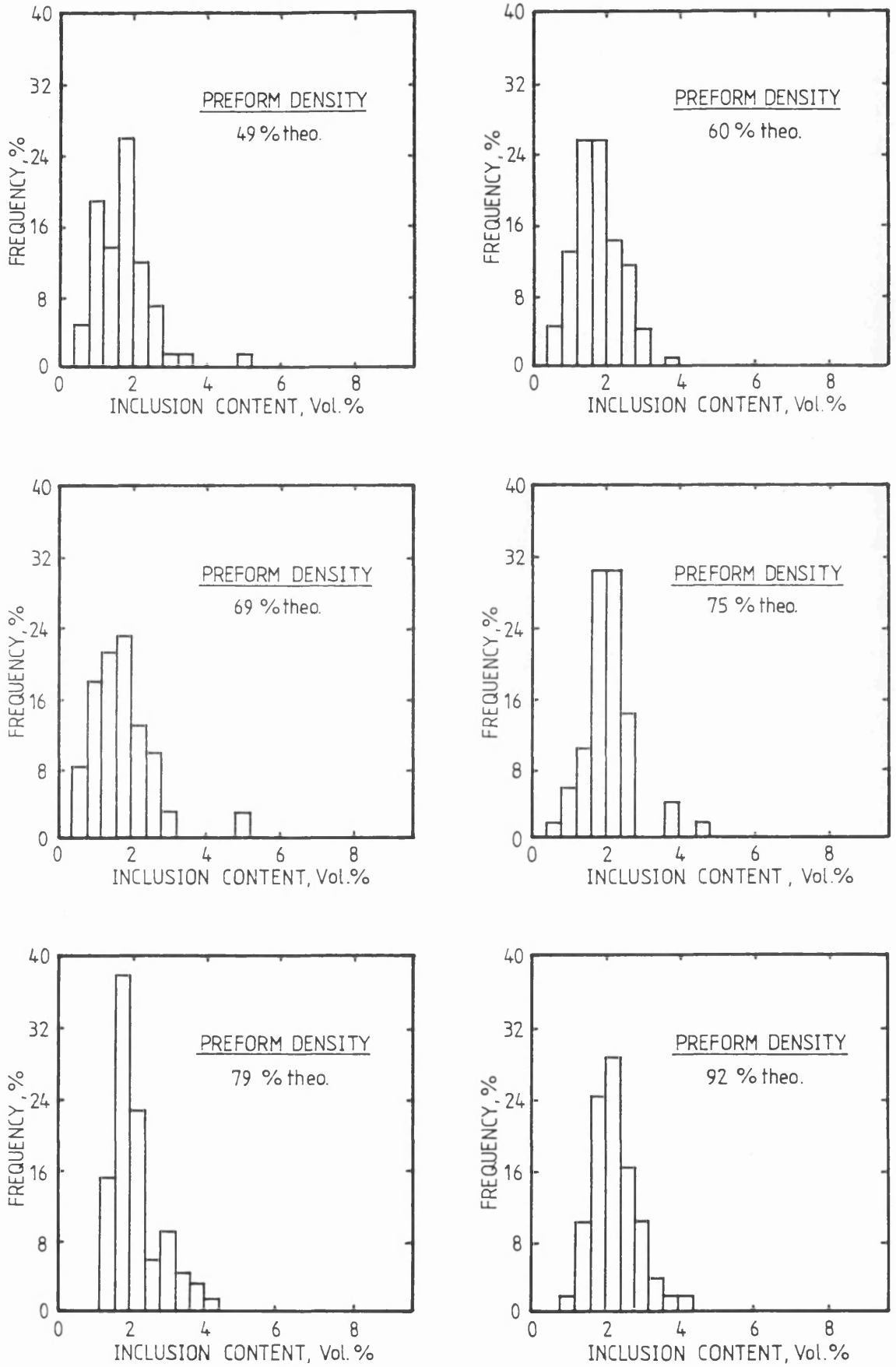


Fig. 10.1. Distribution of volume % of inclusions in the hot forged and annealed sponge iron pellet forgings.

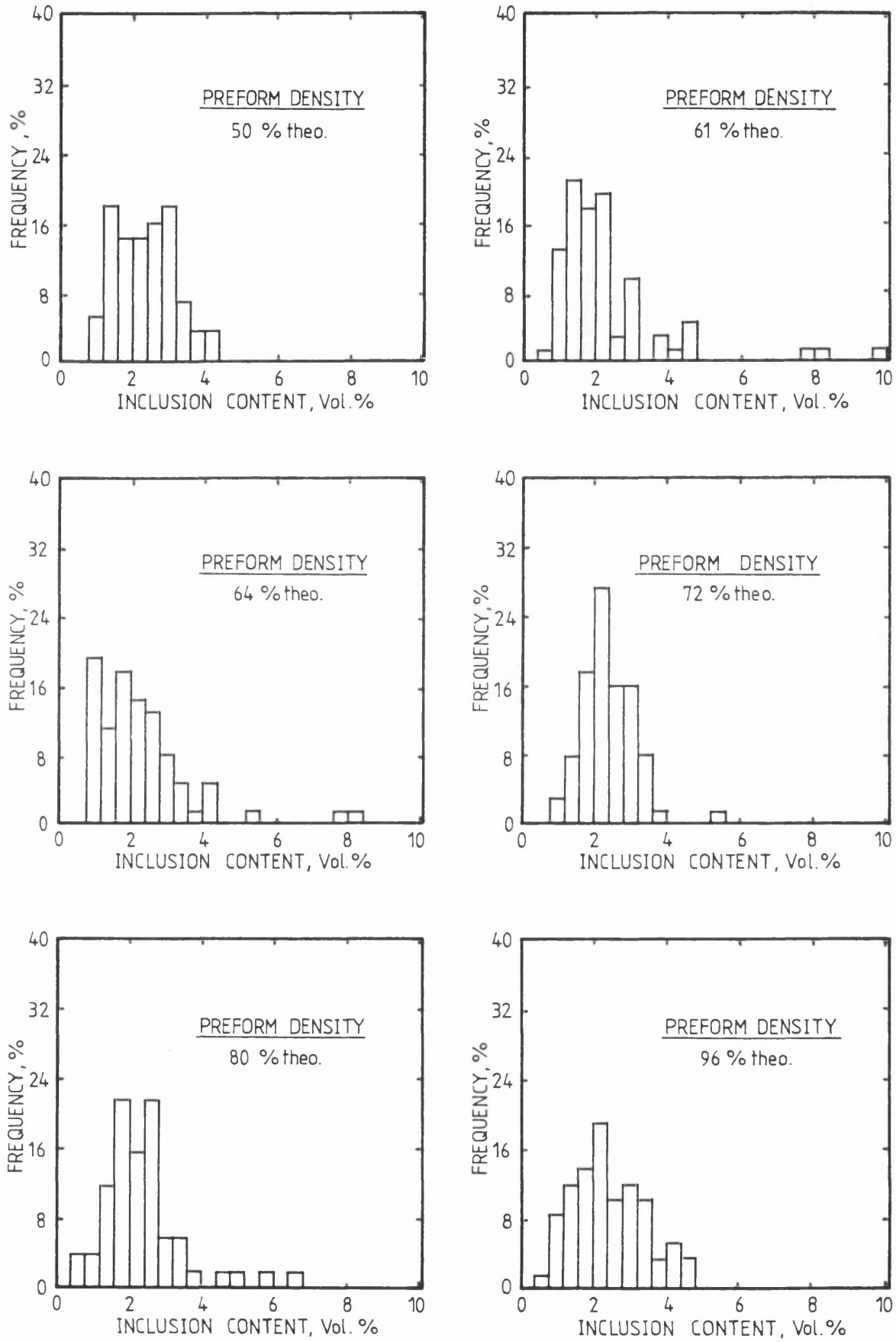


Fig. 10.2. Distribution of volume % of inclusions in the hot forged and annealed NC 100.24 iron powder pellet forgings.

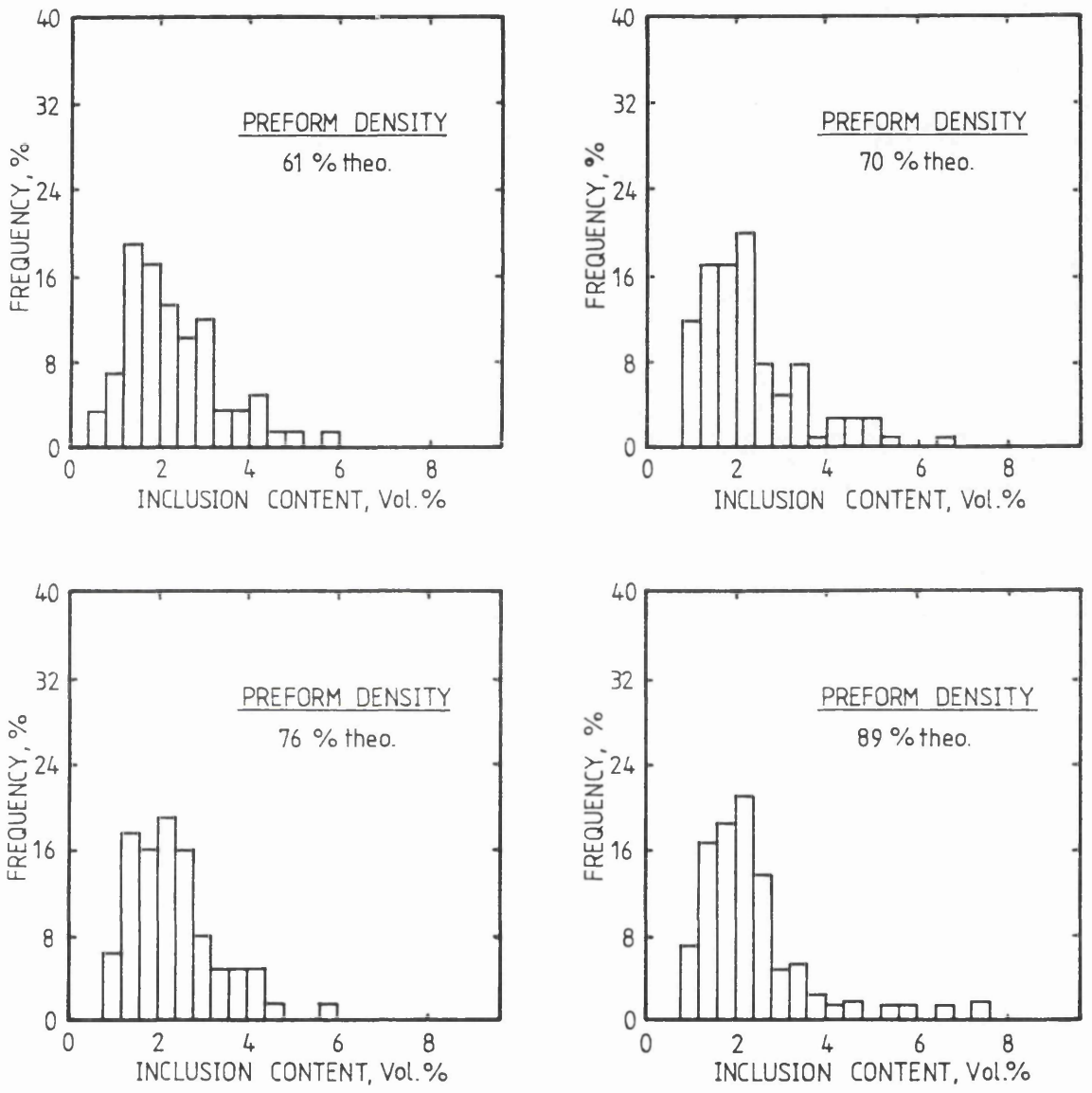


Fig. 10.3. Distribution of volume % of inclusions in the hot forged and annealed NC 100.24 iron powder forgings.

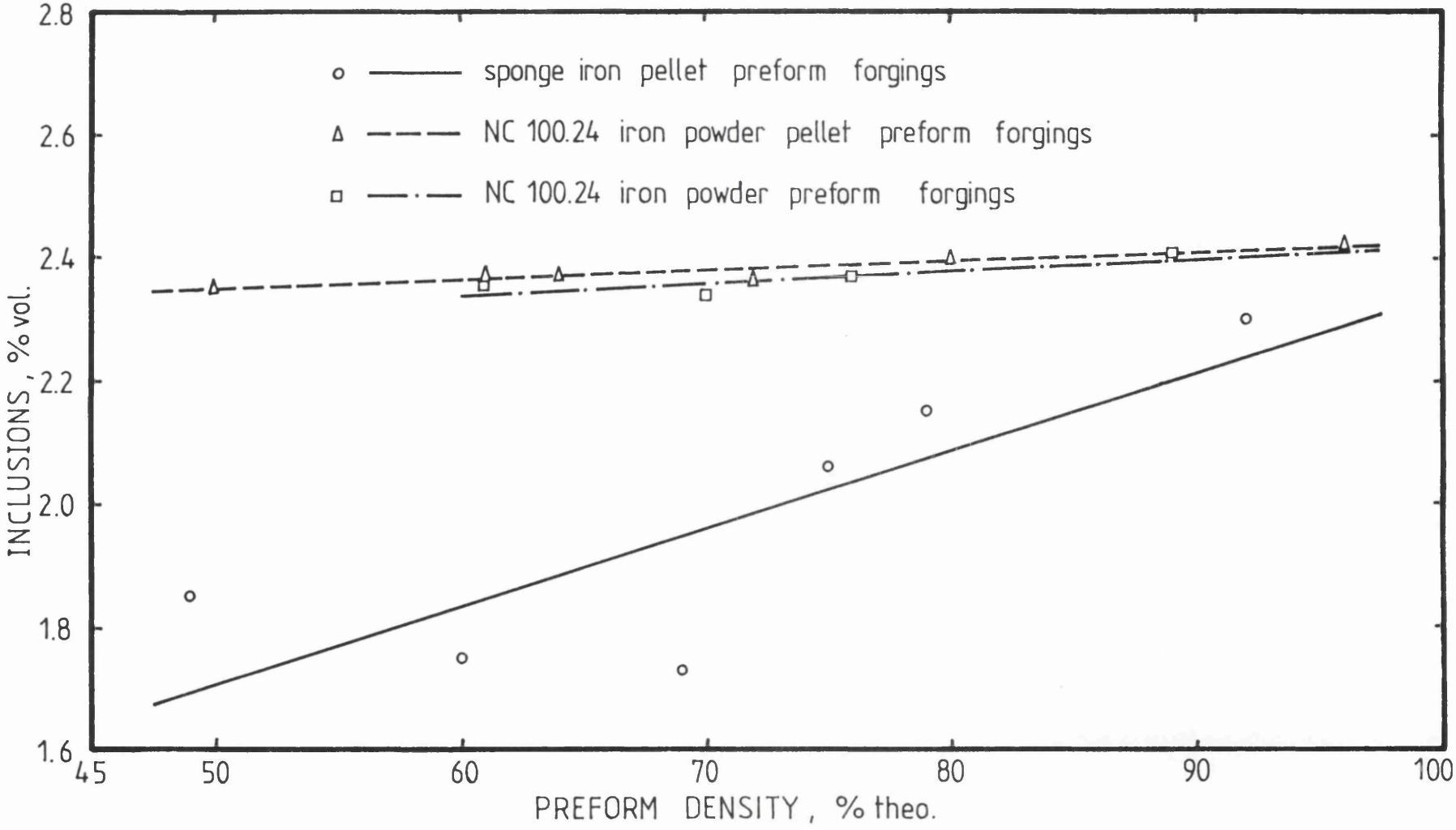


Fig. 10.4. Measured volume percent inclusions in forgings.

Table 10.V (a). Number Of Inclusions Present In The Hot Forged And Annealed Forgings.

No. of fields : 70
Area per field : 0.0288 mm^2
Hot-Forging temperature : 1100°C
Annealing temperature : 700°C
Annealing time : 1 hour

Material	Preform Density (% theo)	No. of inclusions greater than x in length per 0.0288 mm^2 area (A)							
		Length of the inclusions (x), μm							
		0.24	0.48	1.2	2.4	3.6	6.0	9.6	14.4
Sponge Iron Pellets	49	177	151	100	43	20	6	1	0.2
	60	153	135	93	40	19	5	1	0.1
	69	127	111	78	36	18	5	1	0.2
	75	139	123	87	41	20	6	1	0.3
	79	170	152	106	48	23	7	1	0.1
	92	130	119	89	46	24	8	1	0.3
NC 100.24 Iron Powder Pellets	50	82	74	56	31	18	8	3	1
	61	92	80	58	30	17	7	2	1
	64	68	61	46	27	18	8	3	1
	72	94	80	55	27	16	7	3	1
	80	83	75	56	31	18	8	3	1
	96	82	73	53	29	17	8	3	1
NC 100.24 Iron Powder	61	72	64	47	27	16	7	3	1
	70	72	65	49	27	17	7	3	1
	76	73	66	49	28	17	8	3	1
	89	70	63	51	27	18	9	4	1

Table 10.V (b). Number of Inclusions Present In The Hot Forged And Annealed Forgings.

No. of fields : 70
Area per field : 0.0288 mm^2
Hot-forging temperature : 1100°C
Annealing temperature : 700°C
Annealing time : 1 hour

Material	Preform Density (% theo.)	No. of inclusions greater than x in length per 100 mm^2 area, $[A \times \frac{100}{0.0288}] \times 10^5$							
		Length of the inclusions (x), μm.							
		0.24	0.48	1.2	2.4	3.6	6.0	9.6	14.4
Sponge Iron Pellets	49	6.15	5.24	3.47	1.49	0.69	0.21	0.03	0.01
	60	5.31	4.69	3.23	1.39	0.66	0.17	0.03	0.00
	69	4.41	2.84	2.71	1.25	0.63	0.17	0.03	0.01
	75	3.82	3.27	3.02	1.42	0.69	0.21	0.03	0.01
	79	5.90	5.28	3.68	1.67	0.80	0.24	0.03	0.00
	92	4.51	4.13	3.09	1.60	0.83	0.28	0.03	0.01
NC 100.24 Iron Powder Pellets	50	2.85	2.57	1.94	1.08	0.63	0.28	0.10	0.03
	61	3.19	2.78	2.01	1.04	0.59	0.24	0.07	0.03
	64	2.36	2.12	1.60	0.94	0.63	0.28	0.10	0.03
	72	3.26	2.78	1.91	0.94	0.56	0.24	0.07	0.03
	80	2.88	2.60	1.94	1.08	0.63	0.28	0.10	0.03
	96	2.85	2.53	1.84	1.01	0.59	0.28	0.10	0.03
NC 100.24 Iron Powder	61	2.50	2.22	1.63	0.94	0.56	0.24	0.07	0.03
	70	2.50	2.26	1.70	0.94	0.59	0.24	0.07	0.03
	76	2.53	2.29	1.70	0.97	0.59	0.28	0.07	0.03
	89	2.43	2.19	1.77	0.94	0.62	0.31	0.14	0.03

Table 10.VI (a). Size Distribution Of The Non-Metallic Inclusions In The Hot Forged And Annealed Forgings.

No. of fields : 70
Area per field : 0.0288 mm^2
Hot-forging temperature : 1100°C
Annealing temperature : 700°C
Annealing time : 1 hour

Material	Preform Density (% theo.)	No. of inclusions greater than x in length per 0.0288 mm^2 area (A)							
		Length of the inclusions (x), µm							
		0.24	0.48	1.2	2.4	3.6	6.0	9.6	14.4
Sponge Iron Pellets	49	25.50	51.34	57.19	22.55	14.31	4.95	0.91	0.22
	60	17.66	42.34	53.01	21.17	14.11	4.21	0.79	0.09
	69	15.72	33.43	41.72	18.21	13.03	3.66	0.57	0.18
	75	16.15	35.82	46.37	20.61	14.02	4.96	1.18	0.33
	79	17.37	45.71	58.33	24.95	16.49	6.23	1.03	0.12
	92	10.75	29.31	43.37	21.88	16.24	6.61	1.16	0.31
NC 100.24 Iron Powder Pellets	50	7.93	17.91	24.70	13.40	10.65	5.30	1.67	0.82
	61	12.42	22.44	28.21	12.90	9.92	4.45	1.34	0.61
	64	7.00	15.40	19.19	9.37	9.77	3.77	1.58	0.87
	72	13.66	24.97	28.06	11.55	9.28	4.40	1.60	0.89
	80	7.89	18.94	25.13	13.16	9.98	5.61	1.89	0.83
	96	8.84	20.25	24.03	11.69	9.33	5.34	1.52	0.92
NC 100.24 Iron Powder	61	7.95	16.91	20.63	10.73	8.98	3.91	1.73	0.88
	70	7.01	15.93	21.76	10.21	10.04	4.53	1.71	0.80
	76	6.78	17.29	21.51	11.10	9.25	4.84	1.67	0.97
	89	6.58	12.35	24.05	9.43	9.28	4.51	2.89	0.93

Table 10.VI (b). Size Distribution Of The Non-Metallic Inclusions In The Hot Forged And Annealed Forgings.

No. of fields : 70
Area per field : 0.0288 mm^2
Hot-forging temperature : 1100°C
Annealing temperature : 700°C
Annealing time : 1 hour

Material	Preform Density (% theo.)	No. of inclusions greater than x in length per 100 mm^2 area, $[A \times \frac{100}{0.0288}] \times 10^4$							
		Length of the inclusions (x), µm.							
		0.24	0.48	1.2	2.4	3.6.	6.0	9.6	14.4
Sponge Iron Pellets	49	8.85	17.83	19.86	7.83	4.97	1.72	0.32	0.08
	60	6.13	14.70	18.41	7.35	4.90	1.46	0.27	0.03
	69	5.46	11.61	14.47	6.32	4.52	1.27	0.20	0.06
	75	5.61	12.44	16.10	7.16	4.87	1.72	0.41	0.12
	79	6.03	15.87	20.25	8.66	5.73	2.13	0.36	0.04
	92	3.73	10.18	15.06	7.60	5.64	2.30	0.40	0.11
NC 100.24 Iron Powder Pellets	50	2.75	6.22	8.58	4.65	3.70	1.84	0.58	0.28
	61	4.31	7.79	9.80	4.48	3.44	1.55	0.47	0.21
	64	2.43	5.35	6.63	3.25	3.39	1.66	0.55	0.30
	72	4.74	8.67	9.74	4.01	3.22	1.53	0.56	0.31
	80	2.74	6.58	8.73	4.57	3.47	1.95	0.66	0.29
	96	3.07	7.03	8.34	4.06	3.24	1.85	0.53	0.32
NC 100.24 Iron Powder	61	2.76	5.87	7.16	3.73	3.12	1.36	0.60	0.31
	70	2.43	5.53	7.56	3.55	3.49	1.57	0.59	0.28
	76	2.35	6.00	7.47	3.85	3.21	1.68	0.58	0.34
	89	2.28	4.29	8.35	3.27	3.22	1.56	1.00	0.32

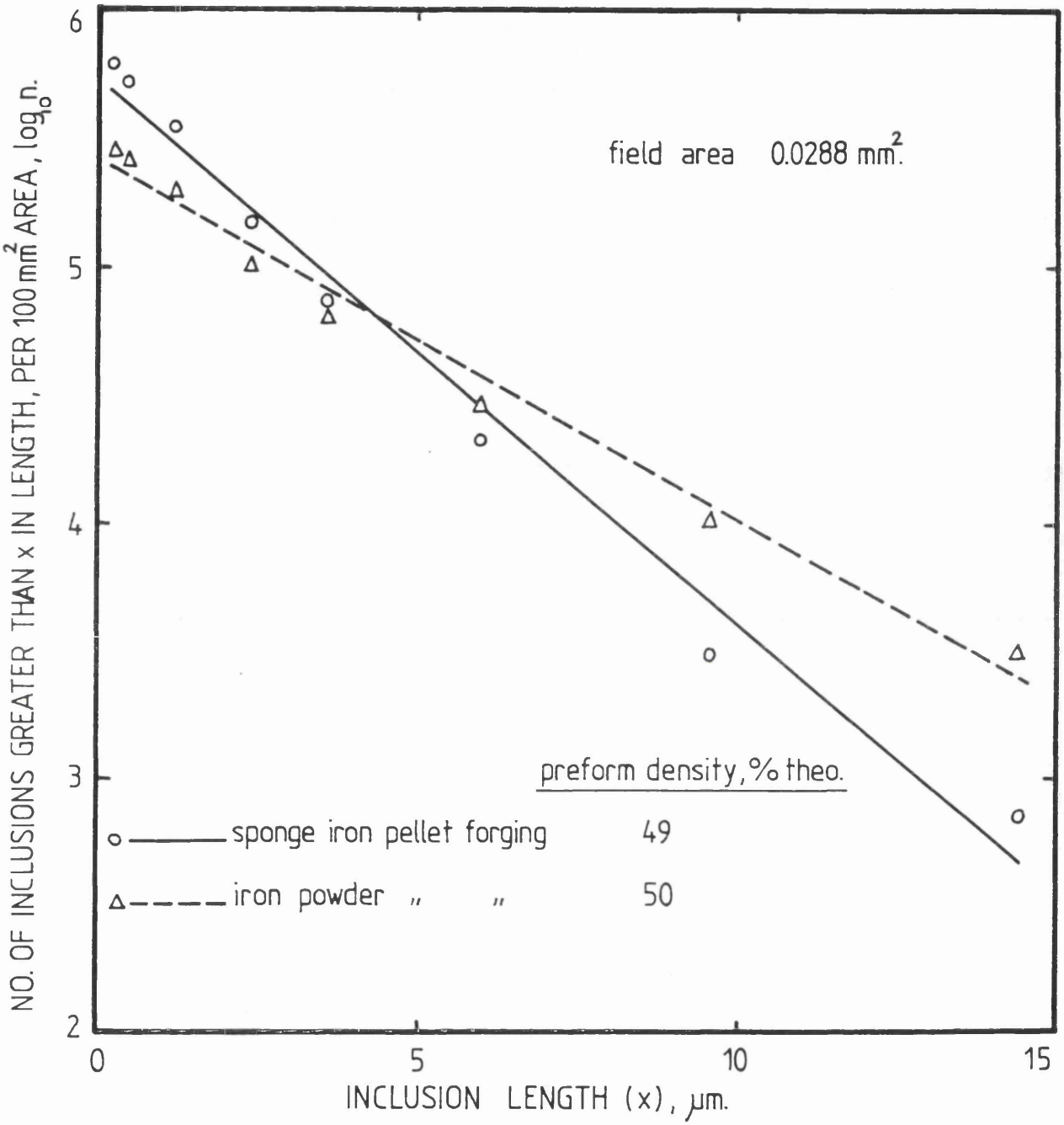


Fig. 10.5. Cumulative frequency/size distribution of non-metallic inclusions in the forgings.

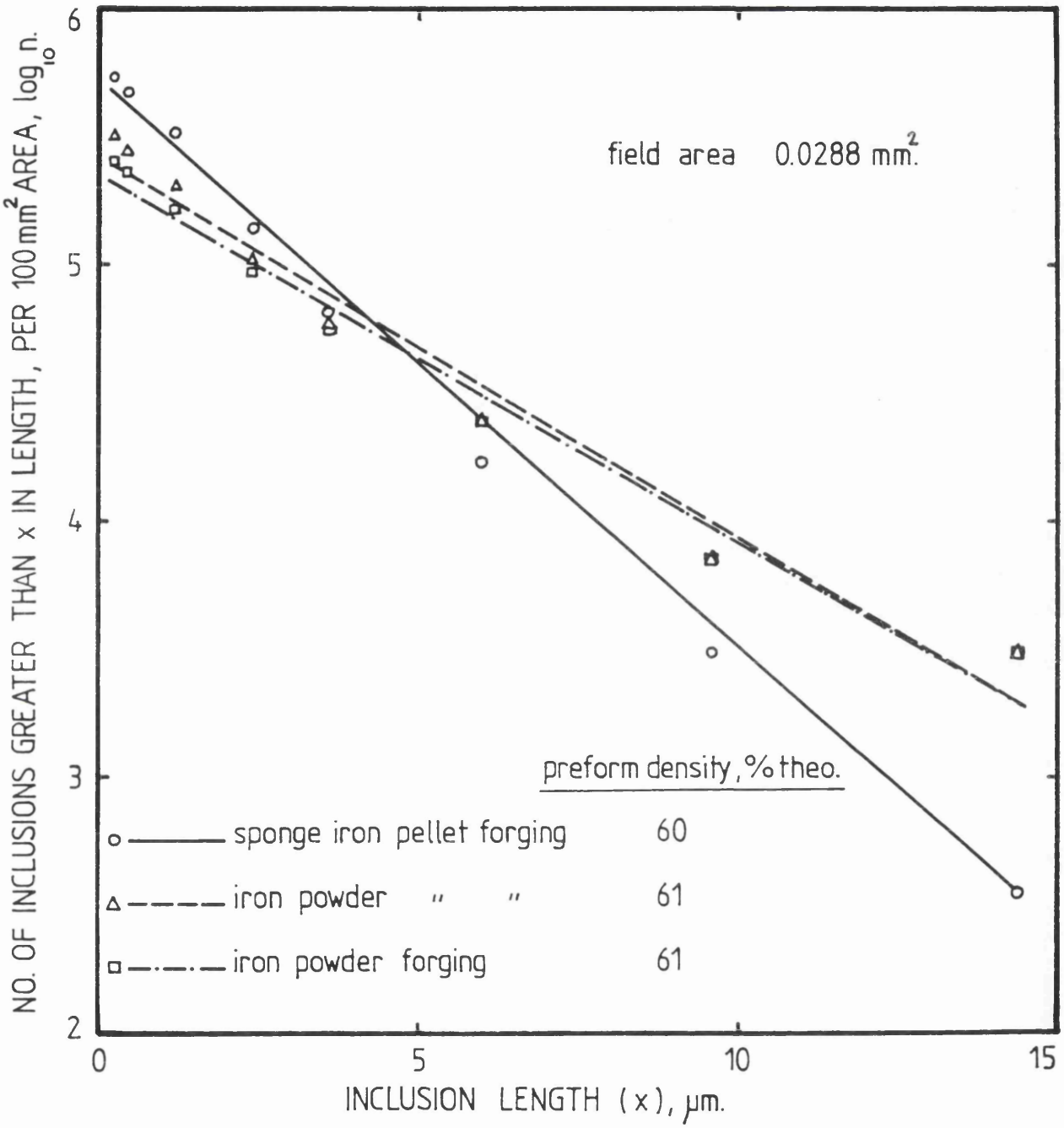


Fig. 10.6. Cumulative frequency/size distribution of non-metallic inclusions in the forgings.

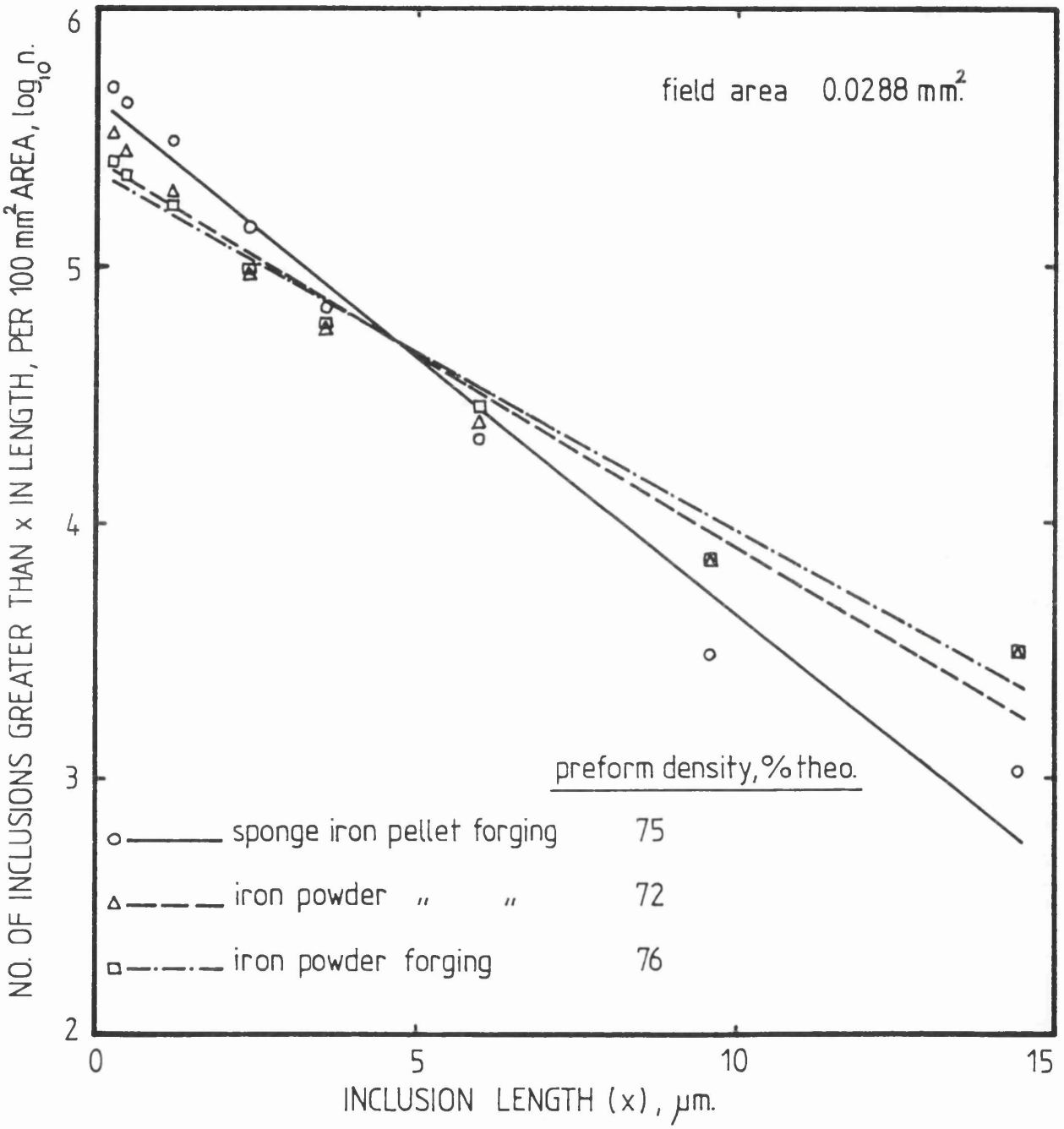


Fig. 10.7. Cumulative frequency/size distribution of non-metallic inclusions in the forgings.

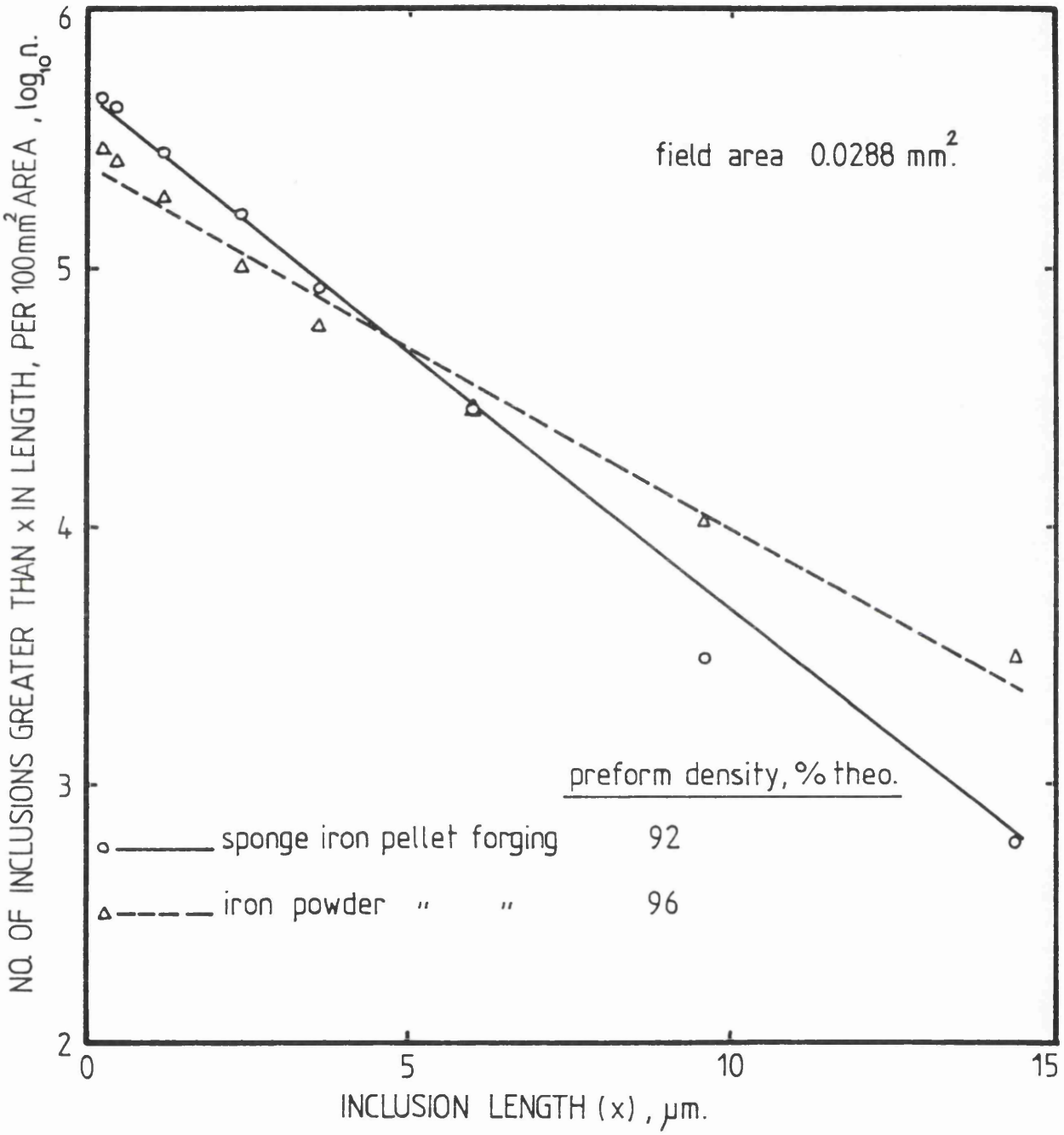


Fig. 10.8. Cumulative frequency/size distribution of non-metallic inclusions in the forgings.

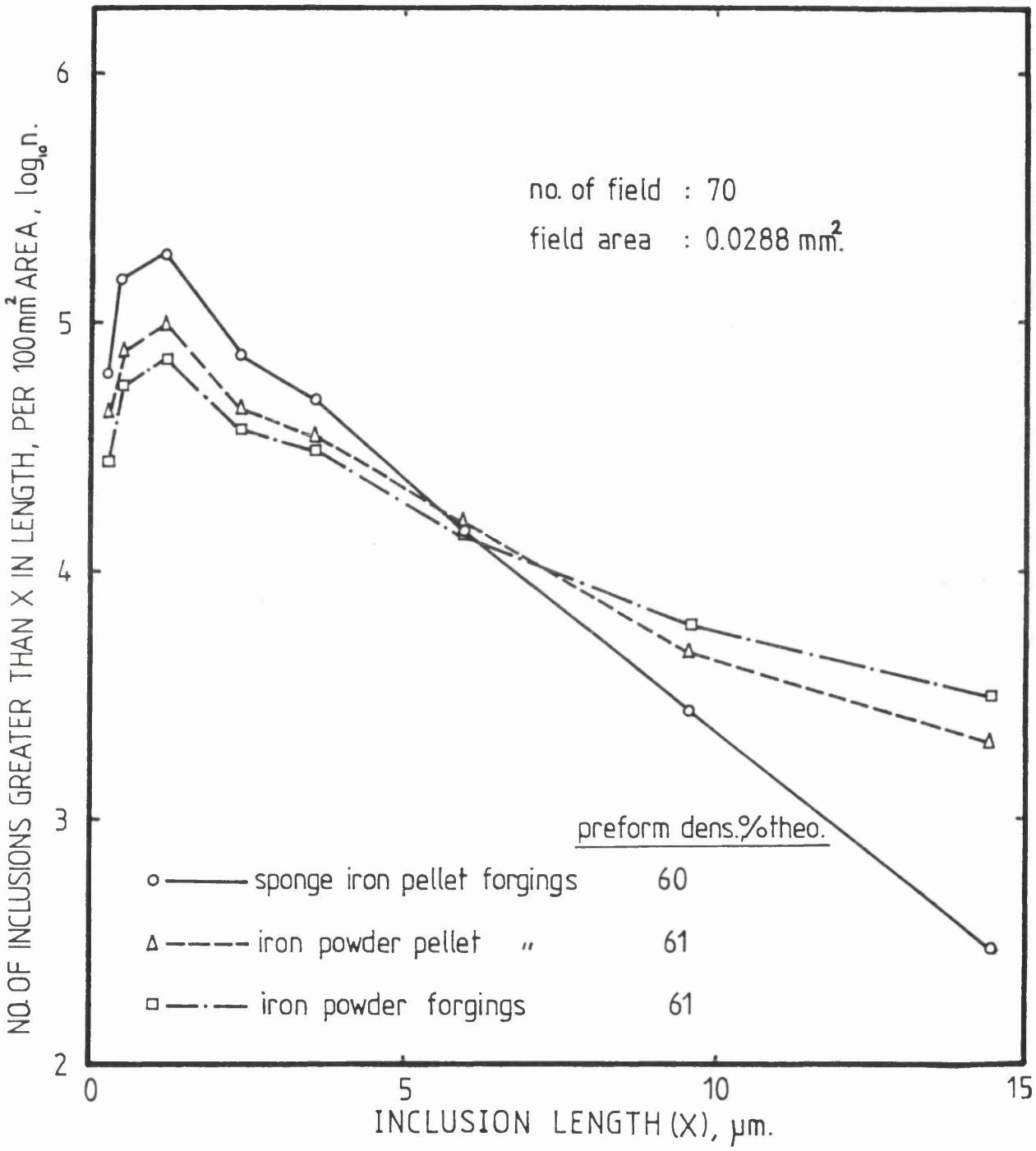


Fig. 10.9. Inclusions size distribution of forgings.

As mentioned in Section 10.1.1., most of the inclusions present in the sponge iron forgings originated from the unreducible oxides present in the magnetite superconcentrate. It is therefore possible to calculate the theoretical volume percentage of non-metallic inclusions which will remain in the finished forgings from the chemical composition of magnetite superconcentrate, as shown in Tabe 9.XII. The theoretical volume percentage of non-metallic inclusions which will remain in the forgings was calculated to be 2.26%. All the experimental values for sponge iron pellet forgings, Table 10.II, with the exception of the 92% density preform forging, were lower than the theoretical figure. This data supports the hypothesis that some inclusions exist in sub-micron sizes and Fig. 10.4. indicates that the amount of such particles decreased with increasing preform density. As for the NC 100.24 iron powder pellet forgings and NC 100.24 iron powder forgings, small changes in the volume percentage of inclusions were found with changing preform density, see Fig. 10.4.

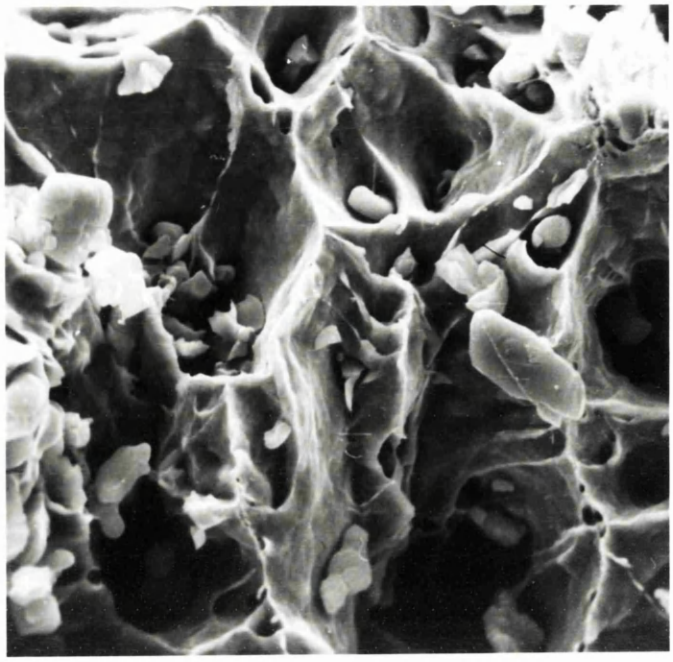
The formation of sub-micron size particles is increased with increasing amounts of deformation during hot forging, as mentioned in Section 9.4. Sliding contact promotes bonding across pore surfaces and can break-up oxide films, with constituent parts of the sponge iron pellet preforms being subjected to major plastic deformation, resulting in the non-metallic inclusions already present in the material and the inclusions formed during the process being broken into fine particles. The size and quantity of fine particles produced is dependent on the amount of deformation given during hot forging, see Fig. 10.4. Although the variation in each value

is large because of the technique used (see Section 7.6.4), there is a clear increasing trend in the average volume percent of inclusions and the preform density. For higher preform densities the volume percent of inclusions approaches to the minimum theoretical value of 2.26%. It is also expected that some additional oxide inclusions would be formed during the process, hence increasing the theoretical value. It is possible that the lower observed volume percent of inclusions in lower preform density forging (e.g. 1.85 ± 0.87% at 49% preform density) can be accounted for by the presence of inclusions which were too small to be observed by the QTM technique. Additional evidence for the presence of such inclusions was obtained from microscopic examination as shown in Fig. 10.10.

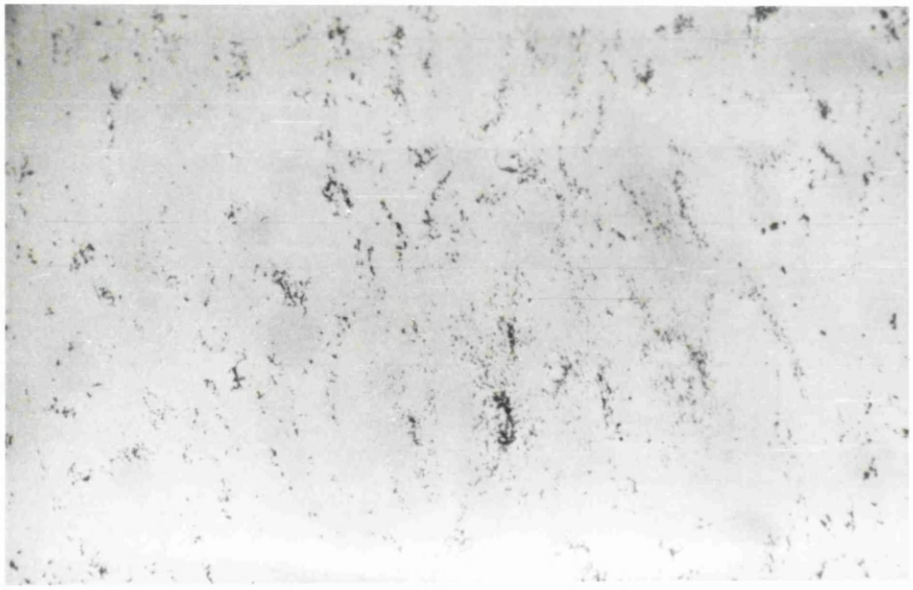
Figs. 10.11 - 10.14 show typical inclusions found in forgings produced in this investigation. Sponge iron pellet forgings are illustrated in Figs. 10.11 and 10.12 with 10.13 showing a typical cross section of an iron powder pellet forging and 10.14 an iron powder forging. There is a significant increase in non-metallic particle size in both the iron powder pellet and iron powder forgings compared with the sponge iron pellet forgings, regardless of preform density. Increased preform density gave an increase in particle size regardless of source. Fig. 10.11 illustrates also that for the lowest preform density the inclusions are small and evenly distributed throughout the forgings.

For comparison, typical micrographs of ASC 100.29 atomised iron powder pellet and ASC 100.29 atomised iron powder forgings made from high density preforms are also given in Figs. 10.15 and 10.16.

Fig. 10.10. Fine inclusions in sponge iron pellet forgings from 49% theo. density preform.
(a) X3,000
(b) X900



a



b

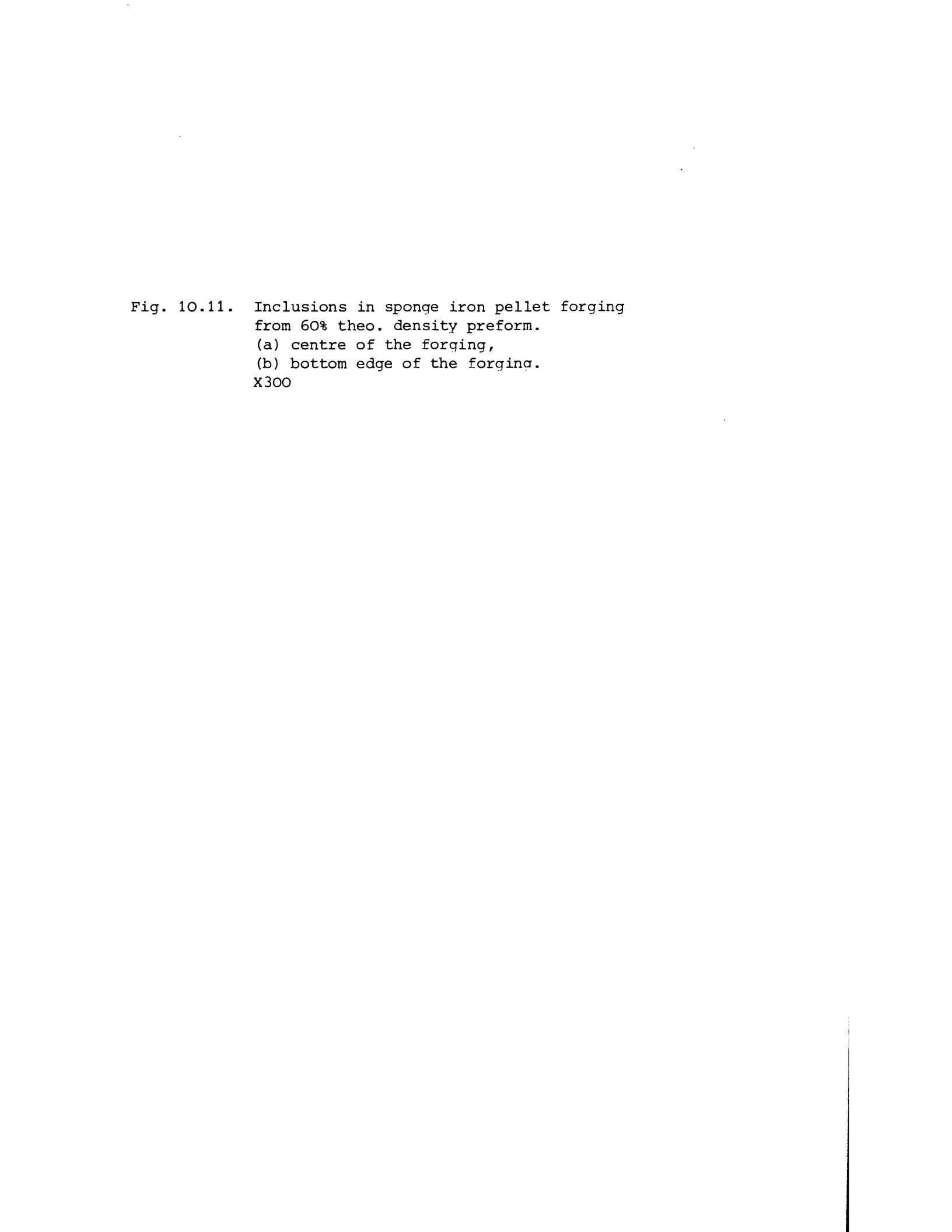
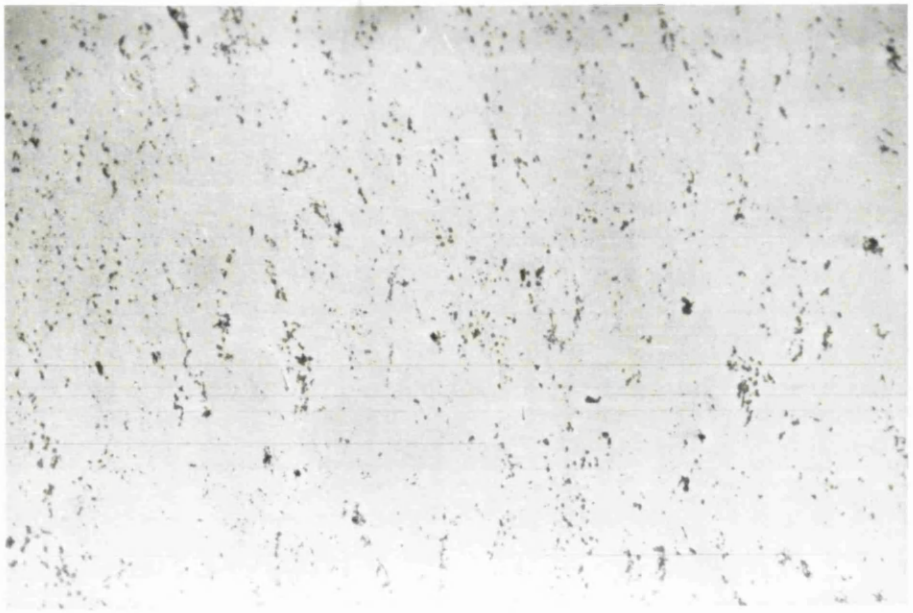
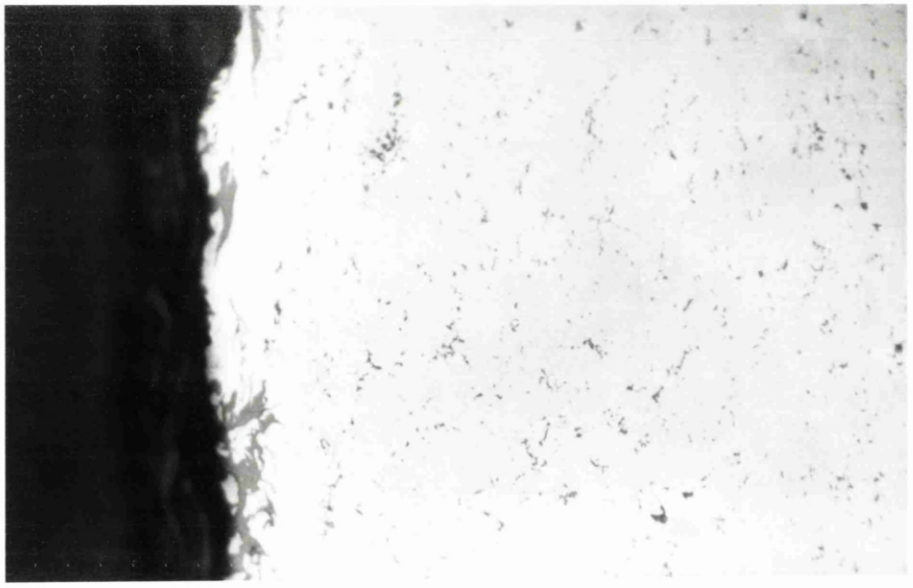


Fig. 10.11. Inclusions in sponge iron pellet forging from 60% theo. density preform.
(a) centre of the forging,
(b) bottom edge of the forging.
X300

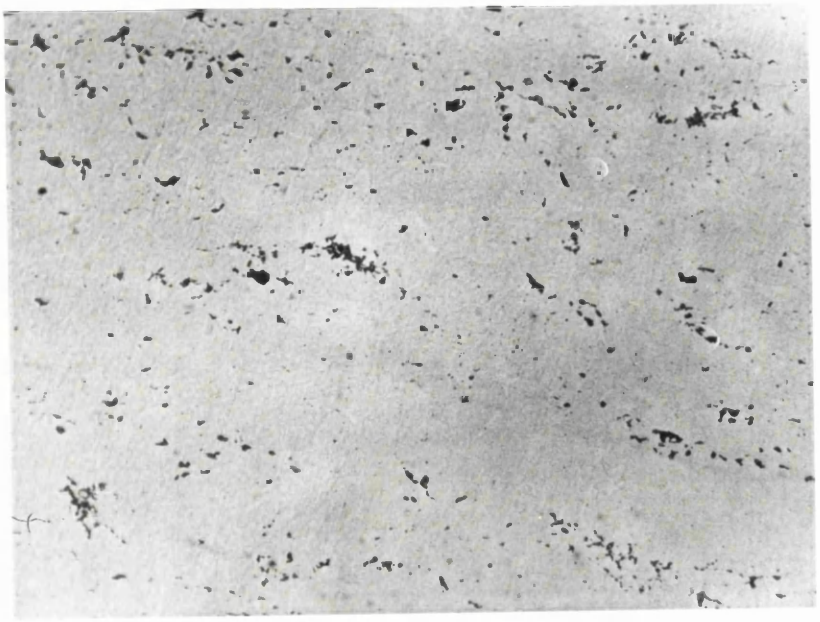


a



b

Fig. 10.12. Inclusions in sponge iron pellet forgings produced from preforms of
(a) 69%,
(b) 80% theo. density.
X300



a

b

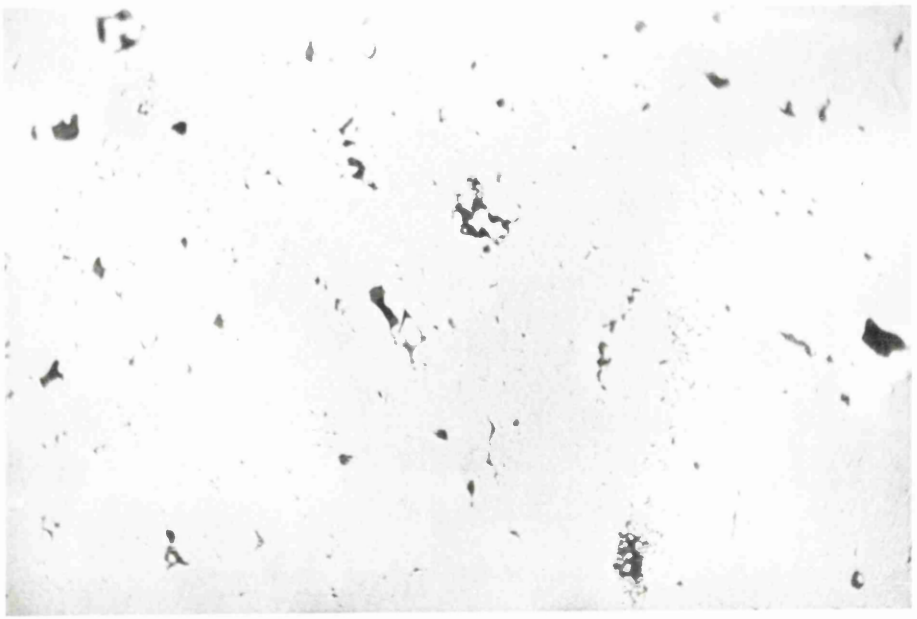
Fig. 10.13. Inclusions in NC 100.24 iron powder pellet forgings produced from preforms of
(a) 50%,
(b) 64%,
(c) 96% theo. density.
X300

a

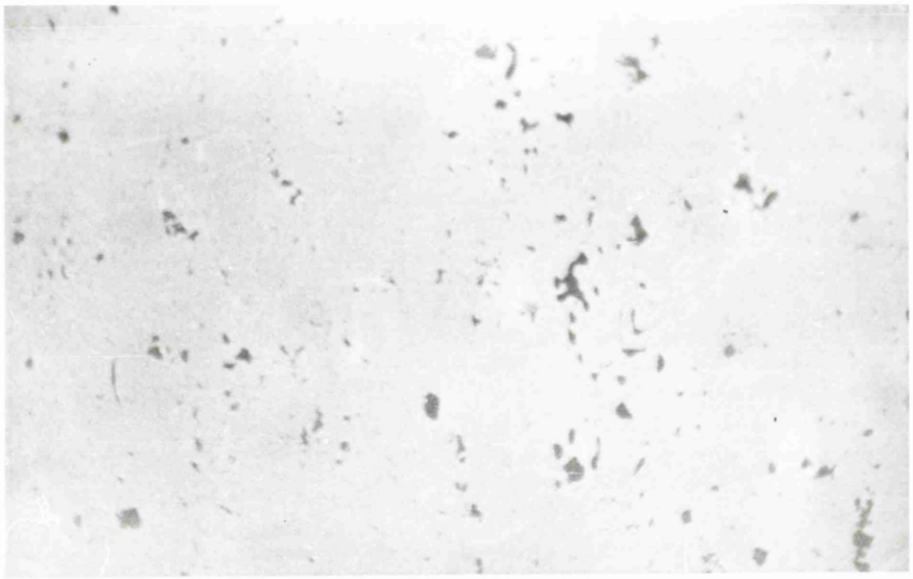
b

c

Fig. 10.14. Inclusions in NC 100.24 iron powder forgings produced from preforms of
(a) 70%,
(b) 89% theo. density.
X300



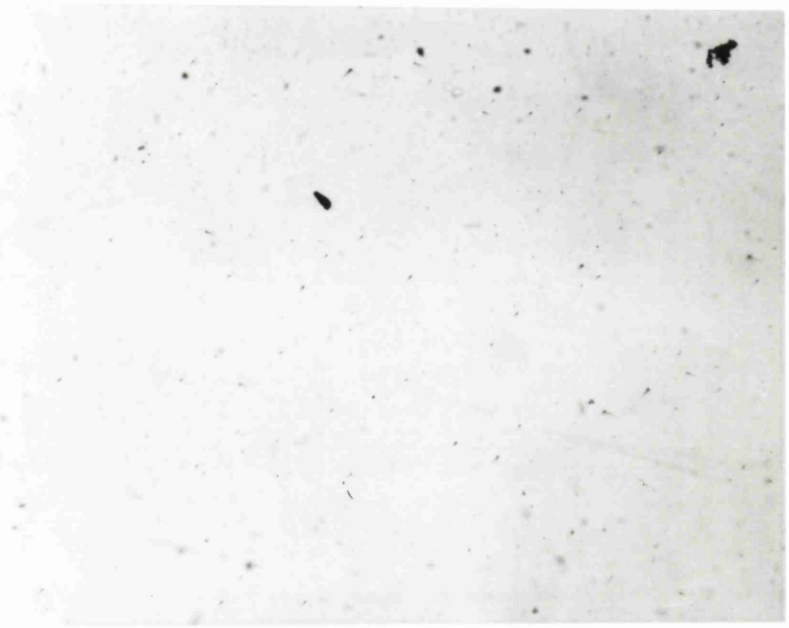
a

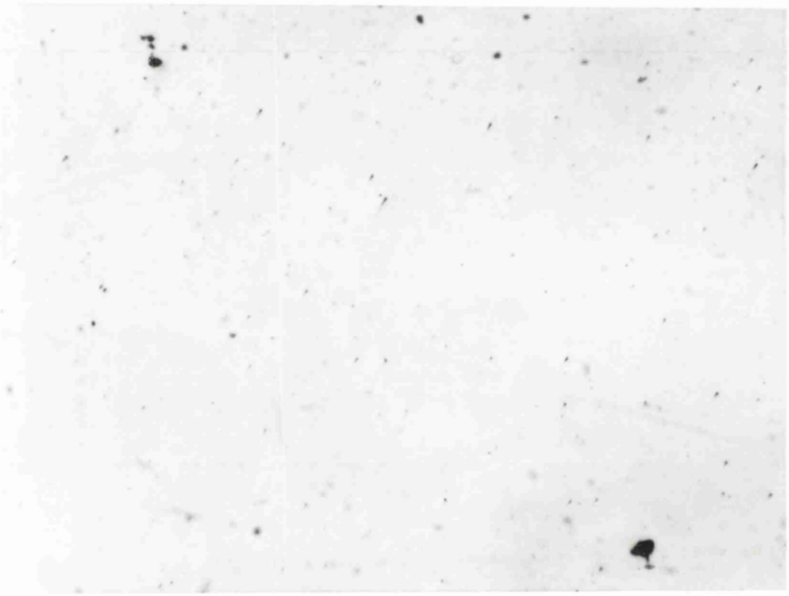


b

Fig. 10.15. Inclusions in ASC 100.29 atomised iron powder pellet forging produced from 86% theo. density preform.
X110

Fig. 10.16. Inclusions in ASC 100.29 atomised iron powder forging produced from 88% theo. density preform.
X110





10.2. GRAIN SIZE EVALUATION

The mean linear intercept method (sometimes referred to as the Heyn intercept) was used for grain size determination. The results obtained for the sponge iron pellet forgings, NC 100.24 iron powder pellet forgings and NC 100.24 iron powder forgings in the as-forged and annealed conditions are given in Table 10.VII and are schematically shown in Figs. 10.17 and 10.18 respectively. Each result was an average of the average values obtained from 65 equally sectioned regions on the cross-section of the forgings, as explained in Section 7.6.5. Photomicrographs in the etched condition of a selection of forgings are to be seen in Figs. 10.19, 10.20 and 10.21 showing the effect of preform density on grain size for each group.

The finest grain size was obtained in the 49% preform density sponge iron pellet forgings after annealing at 700^{o}C for 1 hour (see Fig. 10.19 (a)) giving an average grain size of 6.3 ± 0.89μm. The grain size of sponge iron pellet forgings was smaller than iron powder pellet forgings and iron powder forgings for a given preform density. The grain size of all forgings was increased with increased preform density (see Figs. 10.17 and 10.18). Annealing treatment refined the grain size of all forgings except the highest preform density forgings in each group. Generally, deviation from average grain size increased with increasing preform density. There was no indication of elongated grains in the forging direction and usually they were equiaxed.

The probable reasons for the fine grain size are firstly the large amount of deformation applied to the forgings, secondly, the resulting break-up and uniform distribution of the non-metallic

Table 10.VII. Grain Size of Forgings.

Material	Preform Density (% theo.)	Grain Size , (μm)	
		As-Forged	Annealed
Sponge Iron Pellets	49	8.62 ± 0.99	6.30 ± 0.89
	69	17.63 ± 1.76	11.38 ± 0.98
	92	38.66 ± 5.83	47.51 ± 4.09
NC 100.24 Iron Powder Pellets	50	12.74 ± 1.28	10.72 ± 1.05
	72	19.14 ± 3.10	19.07 ± 0.97
	96	70.11 ± 17.05	70.00 ±28.58
NC 100.24 Iron Powder	61	16.09 ± 2.21	16.66 ± 3.14
	70	32.15 ± 8.07	21.59 ± 4.10
	85	62.55 ±20.00	62.50 ±20.10

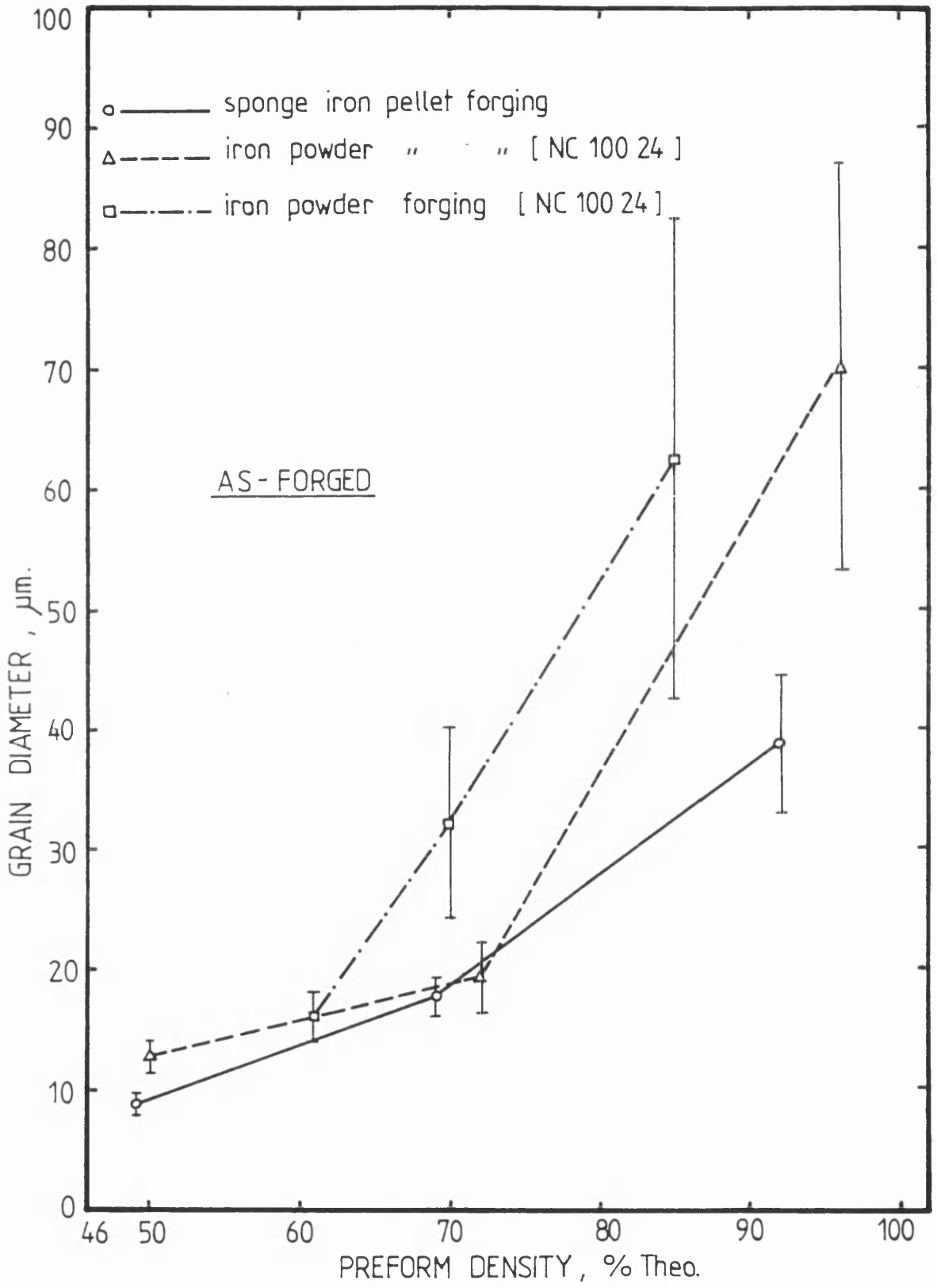


Fig. 10.17. Grain size of forgings.

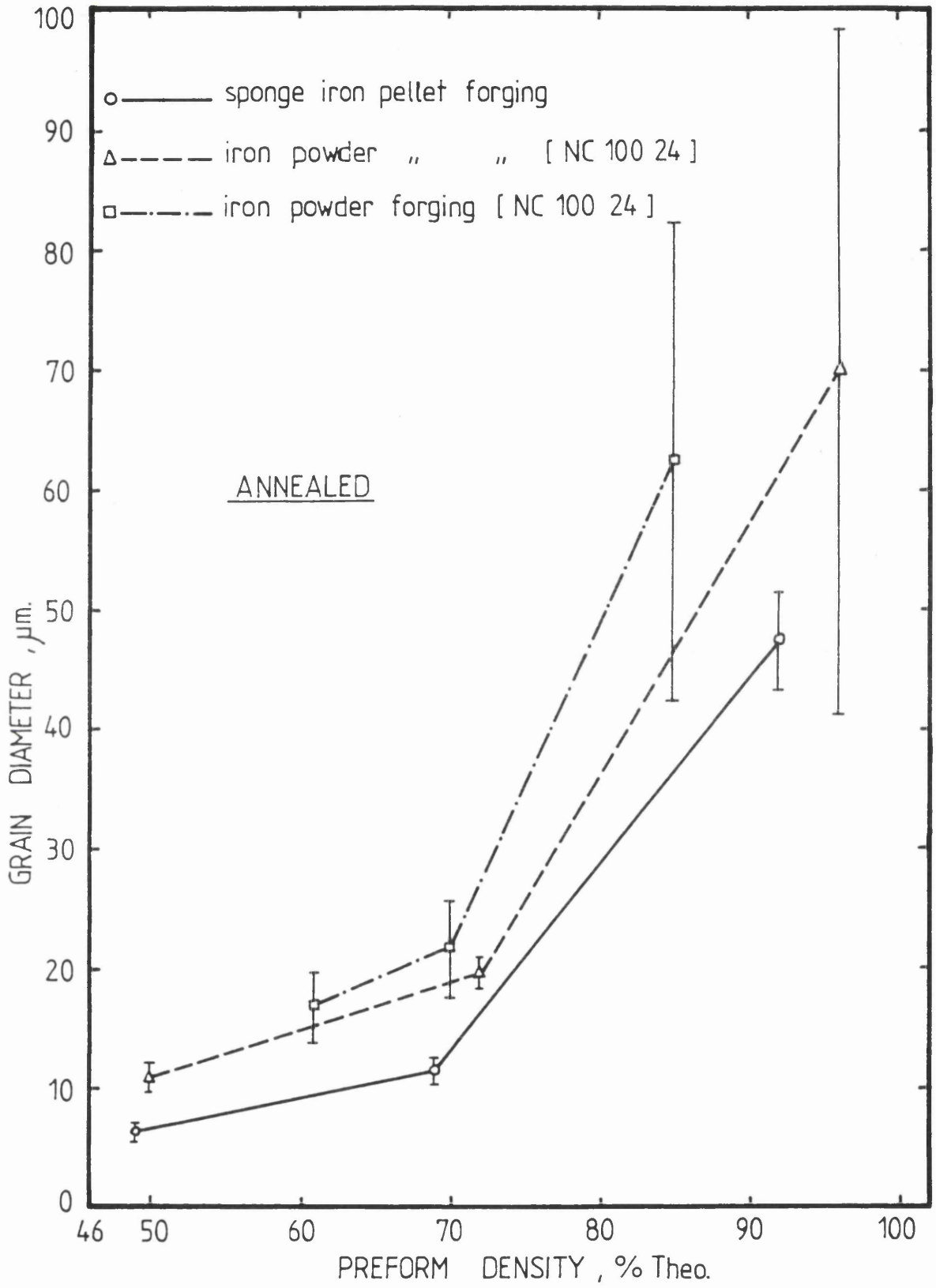
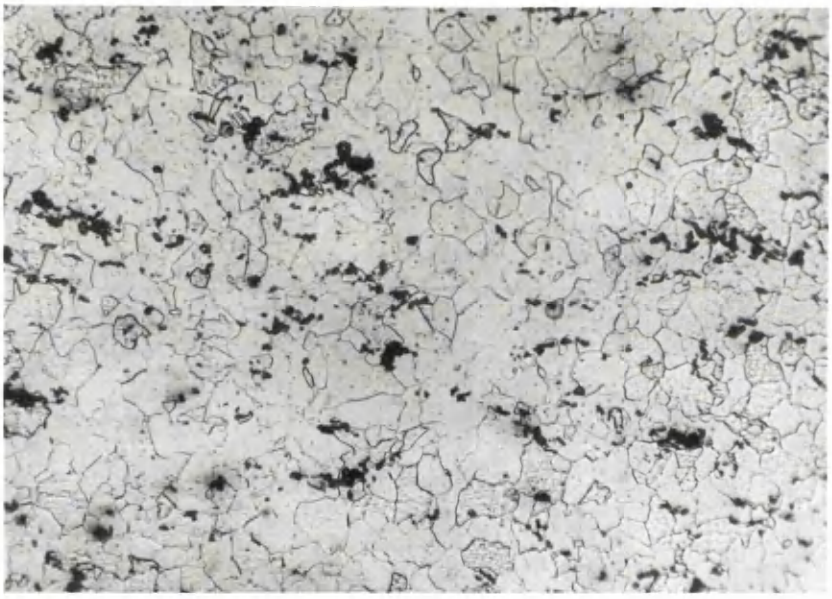


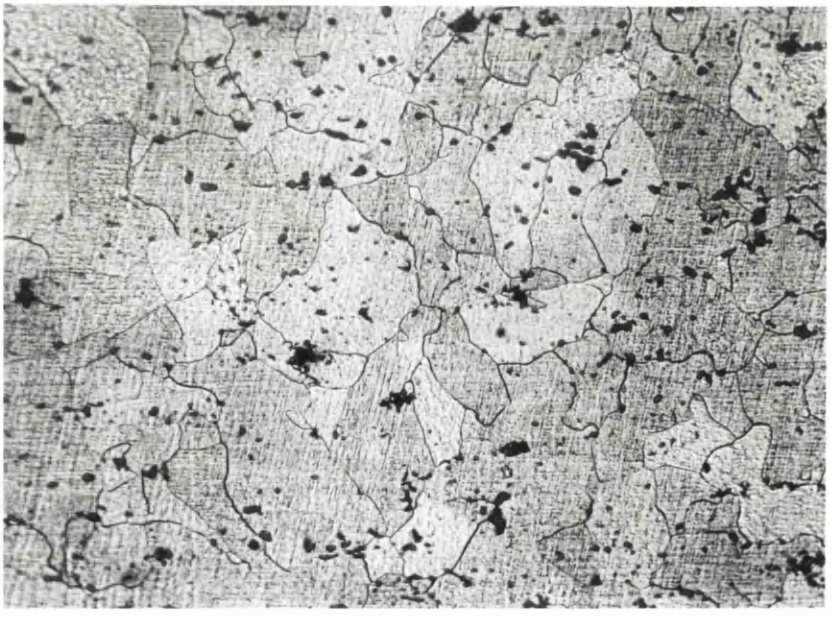
Fig. 10.18. Grain size of forgings.

Fig. 10.19. Microstructures of sponge iron pellet forgings produced from preforms of
(a) 49%,
(b) 69%,
(c) 92% theo. density.
X310. Etchant : 2% Nital.

a

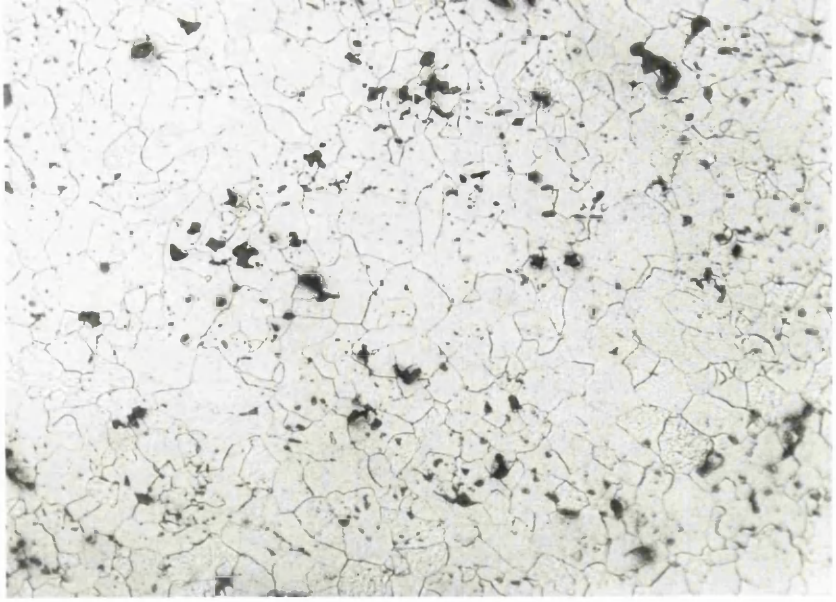


b

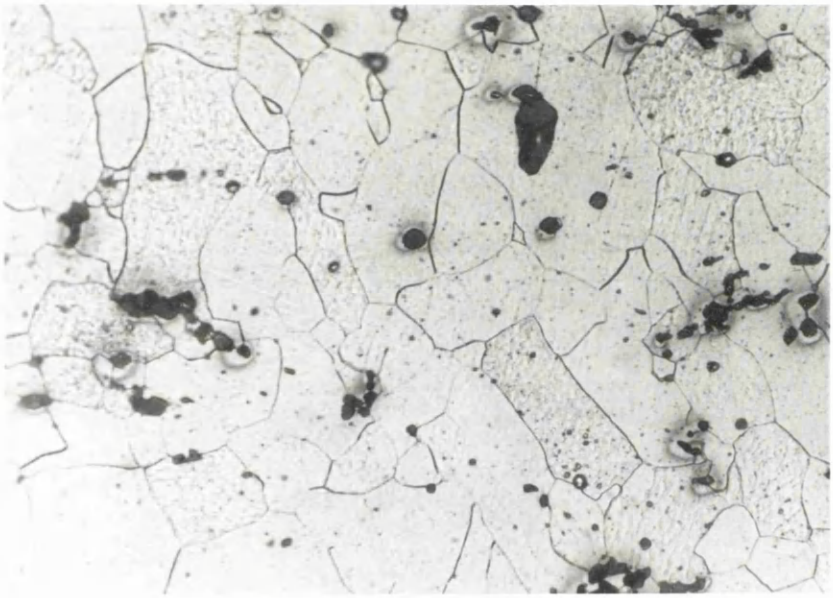


c

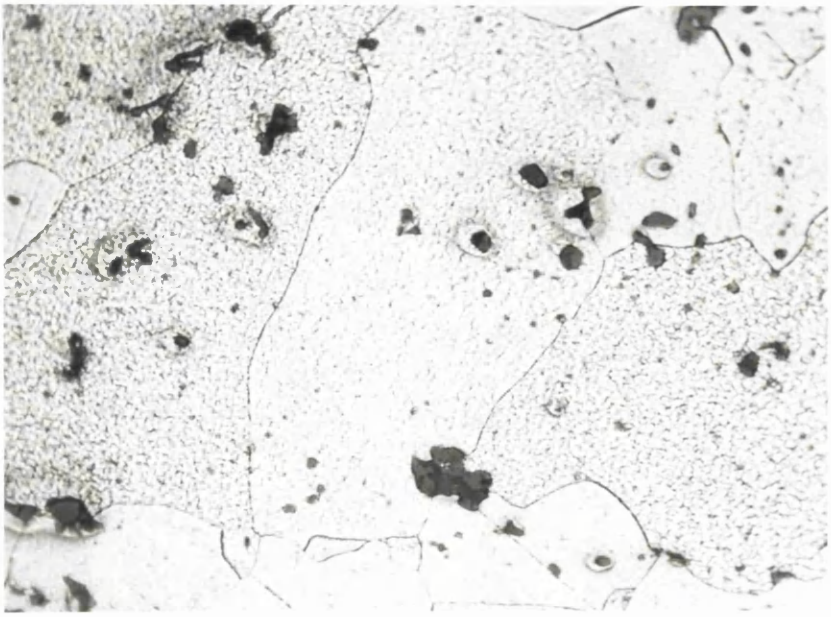
Fig. 10.20. Microstructures of NC 100.24 iron powder pellet forgings produced from preforms of
(a) 50%,
(b) 64%,
(c) 96% theo. density.
X310. Etchant : 2% Nital.



a

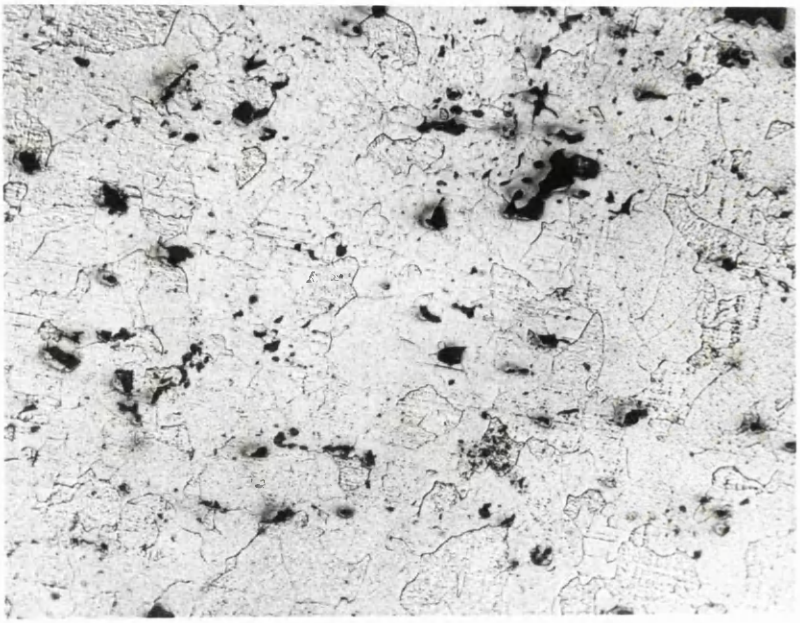


b

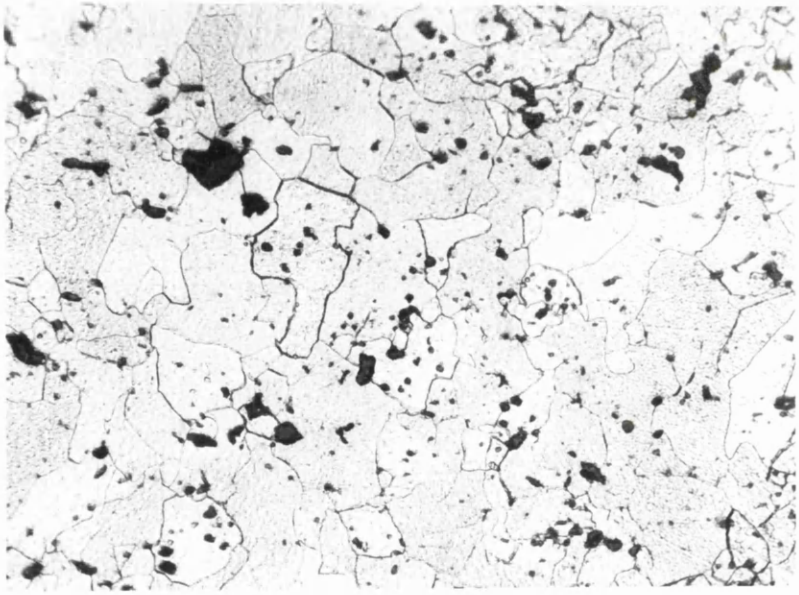


c

Fig. 10.21. Microstructures of NC 100.24 iron powder forgings produced from preforms of
(a) 61%,
(b) 70% theo. density.
X310. Etchant : 2% Nital.



a



b

inclusions, and thirdly, the increased number of nucleation sites as a consequence of the fine dispersion of non-metallics which become apparent after annealing. These aspects will be dealt with in the following discussion.

At any temperature of recrystallization, increased previous strain leads to reduced grain size. As can be seen in Table 9.XlV strain is increased with decreasing preform density. In addition to this, large die movements are involved with lower preform density forgings which causes a longer contact time with the cold die during hot deformation. This results in higher die chilling of the work piece. It is therefore possible that the lower preform density forgings are worked at relatively lower temperatures

Recrystallization in hot-worked silicon-iron was studied by English and Backofen[119] using a 3 - 0.25% Si-Fe compressed to true axial strains of 0.1 to 0.7 at mean strain rate of 100 sec^{-1} and temperatures between 710°C and 911°C. In their findings, the time required for recrystallization was decreased with increasing strain and/or temperature. Nucleation of new grains occurred predominantly at pre-existing grain edges, with some additional nucleation taking place at grain boundries and inclusions. With nucleation confined to boundary sites, the recrystallised grain site was decreased as the original grain size was reduced. It was also suggested[119] that substantial grain refinement by hot working was possible, particularly if intergranular nucleation was introduced. The presence of finely dispersed inclusions could give a high density of nucleation centres within the grains. These findings are similar to those in the present work in that larger strains (lower density preforms) and even distribution of fine inclusions produced finer grain structure.

The recrystallization behaviour of a metal/alloy is dependent upon its structural properties as mentioned earlier. The major structural differences between the forgings made in this investigation was the size and the size distribution of the non-metallic inclusions present in the forgings. The effect of a dispersed second-phase on the recrystallization of a metal or alloy has been studied in great detail for a variety of alloy compositions.[119-129] The dispersed second-phase particles in a matrix were found to originate in two different ways. They may precipitate during the course of annealing after working, alternatively, they may already be present in the matrix before working. Workers in this field have shown that the dispersed second-phase particles in a matrix-irrespective of origin- can either accelerate[119,124-129] or retard[121,122,123] recrystallization, depending upon their size and inter-particle spacing, when compared with a particle-free matrix. A review of relevant literature was made of this subject.

Gladman et al.[128] in their work on plain carbon and grain refined steels found that the coarse second-phase particles (pearlite and spheroidal cementite in their case), of size 0.5 - 20μm, accelerated recrystallization, nucleation occurring at the surface of the second-phase particles. The increased nucleation rate caused more rapid recrystallization and a finer recrystallized ferrite grain size. On the other hand, very fine second-phase particles (alloy carbides and nitrides) of the size $\sim$ 50Å retarded recrystallization when present in an amount of $\sim$ 0.05 - 0.1 vol.%, and the small volume fraction of particles, $\sim$ 200Å in diameter, had little or no effect on the recrystallization rate. In the above investigation, the particles were introduced into the matrix by

different heat treatment methods and by adjusting the chemical composition of the steels. Baird and Arrowsmith[125] studied the effect of the size of second-phase particles on the recrystallization behaviour of some high purity iron of variable oxygen content. The high oxygen content (0.08 - 0.1%) iron had a uniform distribution of 2 - 10μm iron oxide globules. The low oxygen (0.01%) iron was relatively clean, and various heat treatments prior to cold working, as well as annealing after cold working, produced fine inclusions of the size 200 - 2000 Å in the matrix. Recrystallization was favoured in the high oxygen content iron containing numerous oxide globules. No attempt was made in the above investigation to determine the effect of inter-particle spacing of the second-phase particles on the recrystallization behaviour.

In some investigations[126,130] it was shown that inter-particle spacing was more important than particle size in determining the recrystallization behaviour of a two-phase alloy. However, when comparing the results from various workers care must be taken because several formulae were used to describe and evaluate the inter-particle spacing in different investigations. Doherty and Martin[130] studied the recrystallization behaviour of cold rolled Al-Cu alloy having dispersed second-phase particles of size >0.5μm, and inter-particle spacing in the range 1-4μm, and found that recrystallization in an alloy having second-phase particles with an inter-particle spacing of 4μm was about 100 times faster than that of a solid solution alloy having the same composition as the dispersion matrix (i.e. no second-phase particles). On the other hand, recrystallization was 10 times

slower in a two-phase alloy with an inter-particle spacing of 1.5μm than the latter and 1000 times slower if the inter-particle spacing decreased to 1.2μm. They did not investigate the effect of inter-particle spacing greater than 4μm. Mould and Cotterill[12] investigated the effect of inter-particle spacing of Al_3Fe particles in the range 4 - 15μm on the recrystallization behaviour of two-phased Al-Fe alloys. The inter-particle spacing was adjusted by varying the iron content from 0.008% to 1.08%. They found that recrystallization was accelerated as the iron content was increased, i.e. as the inter-particle spacing was decreased. This effect was associated with a marked increase in nucleation rate, with no significant change of growth rate, as the Al_3Fe particle spacing decreased from 15 to 4μm. These effects were shown to be independent of the size of Al_3Fe particles within the range 0.6 - 2.2μm. Mould and Cotterill[126] combined their own data with those of Doherty and Martin[130] and postulated a model for these effects, the essential features of which are that recrystallization is accelerated as the inter-particle spacing is decreased towards a value of approximately twice the deformation cell size, and then it is retarded as the inter-particle spacing is further decreased with an intensification of the rate of retardation at inter-particle spacings which are less than the deformation cell size.

From the above experimental observations, it is clear that, irrespective of metal/alloy compositions, the recrystallization in a two-phase metal/alloy is accelerated relative to that of the matrix phase alone by the presence of large particles (>0.5μm size) with a wide inter-particle spacing (greater than approximately twice the deformation cell size). On the other hand, the presence

of small particles (~ 50 Å) with close inter-particle spacing (approximately less than the deformation cell size) retards the recrystallization process. In the case of polycrystalline iron, the cell size increases with increasing deformation up to about 10% strain, and then maintains a constant diameter of about 1μm.[131] Therefore the presence of large particles of >0.5μm size with inter-particle spacing of $\sim 2\mu$m in an iron matrix would accelerate the onset of recrystallization in comparison with a relatively particle-free iron matrix, while the presence of small particles of 50 Å size with inter-particle spacing of $\sim 1\mu$m would retard recrystallization. The above reported experimental observations regarding the effects of second-phase particles on the recrystallization behaviour could be explained in the following manner. Working causes increased dislocation densities around hard second-phase particles. Under such conditions, the large dispersed hard particles (or more accurately, the interface between particles and matrix) act as sites for the formation of recrystallization nuclei. Small particles, on the other hand, lead to increased homogeneity of the dislocation distribution, and therefore impede nucleation. It is clear from the above that the introduction of large diameter particles in a matrix will increase the nucleation rate. Further increase in particle density, and therefore decrease in the inter-particle spacing, will then lead to a continued and proportionate increase in the nucleation rate. This trend will continue until the inter-particle spacing is sufficiently wide so that the nuclei formed simultaneously at the particle/matrix interface do not interfere with each other before reaching a viable size. This is the reason why the large particles with wide inter-particle

spacing (greater than twice the cell size) increase the recrystallization rate as compared with that of a particle-free matrix. Close inter-particle spacing impedes the re-arrangement of dislocations or dislocation configuration to grain boundaries capable of migration.[129] Therefore, small particles with close inter-particle spacing (of the order of the cell size) retard recrystallization because of the greater homogeneity of the dislocation re-arrangement and grain boundary migration.

A similar phenomenon occurred in the work reported in this thesis. Sponge iron pellet forgings had an inclusion content of around 2% vol. most of which were in the size range 0.24 - 3.6μm, and were distributed uniformly throughout the matrix. Details of these were presented in Section 10.1. The exact calculation of average particle size, and hence the inter-particle spacing, in the present case is difficult because of the wide range and skewed size distribution of those inclusions. Also the volume fraction of inclusions in the sponge iron pellet forgings was 2.26%, but experimental observations indicated that generally the values were lower than that of theoretical value. Nevertheless, an appropriate calculation of the average inclusion size was made from the number of particles in each size range, and the value thus obtained was 1.4μm for a 49% preform density sponge iron pellet forging. This density was taken because it was considered that this forging contained the smallest average particle size. The inter-particle spacing was calculated from the following formula, assuming that the volume fraction of the inclusions was 2.26, and also assuming that the inclusions were spherical.[132]

$$\Delta_3 = 0.554\ r\left(\frac{4\pi}{3\ fv}\right)^{\frac{1}{3}}$$

where

Δ_3 = particle centre-to-centre nearest neighbour distance in the volume,

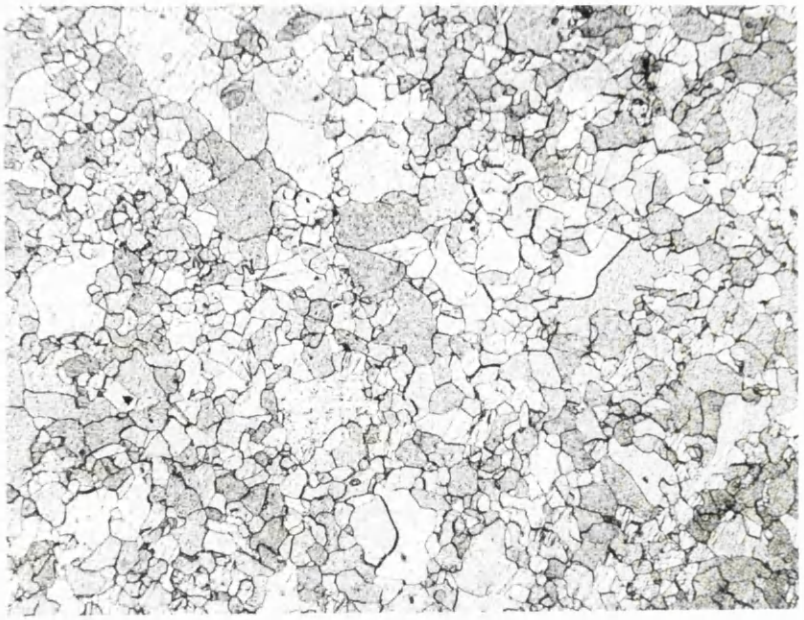
r = average radius of spherical inclusions,

fv = volume fraction of inclusions.

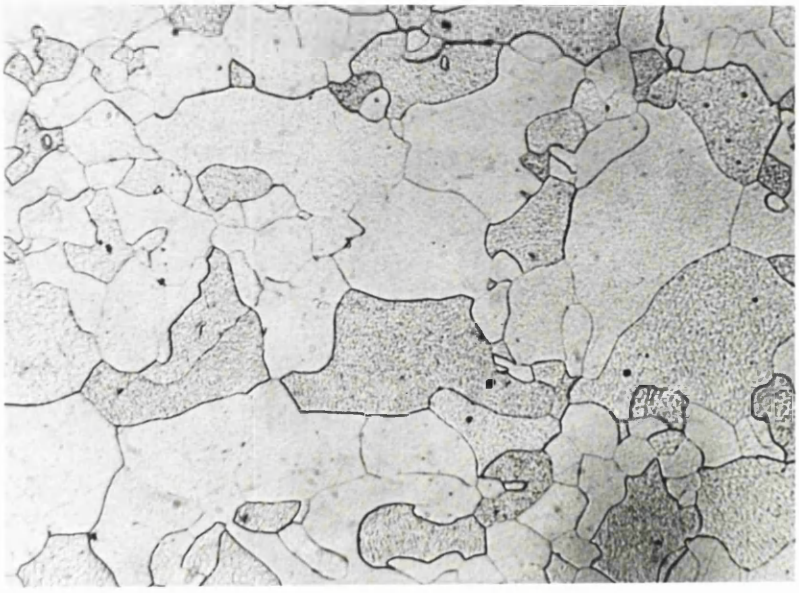
The inter-particle spacing thus obtained was 4.4 μm. Although the above calculated inclusion parameter is only approximate, it does not indicate that most of the inclusions present in the forging were large and widely spaced from the point of view of recrystallization. Therefore, the size and inter-particle spacing of these inclusions were of the right order for the interface between inclusions and matrix to act as sites for the formation of nuclei during annealing. This resulted in a finer grain size, especially for the low preform density pellet forgings.

Some refining of grain size occurred in the forgings produced from the ASC 100.29 atomised iron powder pellet and ASC 100.29 atomised iron powder. Although the amount of refinement was less than that found with sponge iron powder pellet forgings (atomised iron powder contains small amount of inclusions), grain refinement was greatest for the lower density preforms, as previously found. Typical grain structures of these forgings are shown in Figs. 10.22 and 10.23. The average grain sizes for atomised iron powder pellet forgings were about 34μm for 68% density preform forging and about 94μm for 86% density preform forging. For the atomised iron powder

Fig. 10.22. Microstructures of ASC 100.29 atomised iron powder pellet forgings produced from preforms of
(a) 68%,
(b) 86% theo. density.
X110. Etchant : 2% Nital.



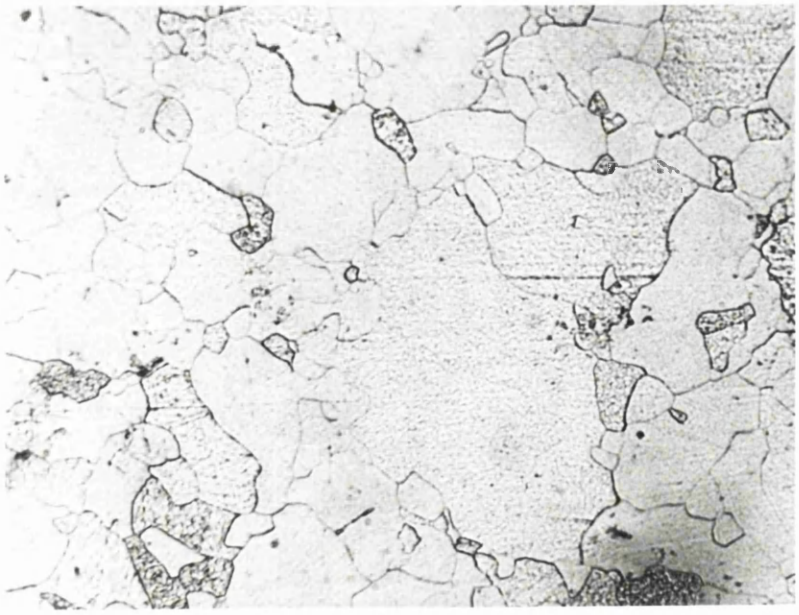
a



b

Fig. 10.23. Microstructures of ASC 100.29 atomised iron powder forgings produced from preforms of
(a) 67%,
(b) 89% theo. density.
X110. Etchant : 2% Nital.

a



b

forgings, the average grain sizes were 37μm for 67% preform density forging and 100μm for 88% density preform forging. These results conform to the findings discussed in the literature review, and support the reasons advanced to explain the phenomenon.

10.3. THE EFFECT OF STRUCTURAL PROPERTIES ON THE MECHANICAL PROPERTIES OF SPONGE IRON PELLET FORGINGS

The mechanical properties results of forgings made in this investigation were already given in Chapter 9. Generally, the mechanical properties of the forgings were dependent on the preform density and annealing treatment. The U.T.S. of the forgings increased as the preform density decreased in both the as-forged and annealed conditions. Annealing also resulted in an increase in U.T.S. values. Ductility increased as the preform density increased with annealing, this was only a minor effect. Impact strengths increased with increasing preform density in all conditions, while annealing treatment resulted in lower impact values for the pellet forgings.

The mechanical properties of materials are generally related to their structural properties. Of the materials investigated the basic structural differences are the inclusions content, size and size distribution. The sponge iron pellet forgings have a large amount of small non-metallic inclusions, especially in the case of the low density preform forgings. Although NC 100.24 iron powder forgings had a similar chemical composition as the sponge iron pellet forgings, non-metallic inclusions in the sponge iron pellet forgings were smaller in size and mechanical properties were better or similar to NC 100.24 iron powder forgings (See Table 10.VIII).

Table 10.VIII Comparison of Typical Mechanical Properties of Forgings.

Material	Preform Density (% theo.)	U.T.S. ($MN.m^{-2}$)	Elong-ation (%)
Sponge Iron Pellets	49	655	12
	75	470	19
	92	348	26
NC 100.24 Iron Powder Pellets	50	442	20
	72	553	13
	96	313	31
NC 100.24 Iron Powder	70	427	15
	89	303	24
ASC 100.29 Iron Powder Pellets	50	453	17
	68	325	35
	86	299	37
ASC 100.29 Iron Powder	67	341	35
	88	312	39
0.46% Steel Powder Forging*	-	638	20

* From reference (139)

It has been shown by others that steels with high non-metallic insoluble inclusions have inferior mechanical properties to those of steels relatively free from these impurities. From the above the mechanical properties of sponge iron pellet forgings would be expected to be inferior to those of the atomised iron powder forgings which contained relatively small amounts of inclusions. The presence of non-metallic inclusions had an effect on the U.T.S. of the sponge iron pellet forgings, in fact U.T.S. values were increased, but ductility suffered (see Table 10.VIII).

When the sponge iron pellet forgings are compared to the low carbon steel powder forgings, the difference between the mechanical properties is small. The sponge iron pellet forging is virtually a pure iron composite in which non-metallic insoluble inclusions are uniformly distributed, while low carbon steel powder forgings contain strengthening elements such as C, Mn, Si, etc. Table 10.8. shows that the presence of non-metallic inclusions has no detrimental effect on the U.T.S. of low preform density sponge iron pellet forgings, and the detrimental effect on the ductility is very small.

Eudier[133] reported that a dispersion of 25 Å particles in a P/M iron matrix results in a strength of 2000 MNm^{-2}; 100 Å (0.01μm) particles result in a strength of 1200 MNm^{-2}, while 4000 Å (0.4μm) particles cause no increase in the strength of pure iron. The presence of particles greater than 4000 Å (0.4μm) size results in a reduction of the strength of iron. The Q.T.M. equipment used in this investigation was unable to detect inclusions less than 0.24μm in size, therefore from Table 10.II

it can be seen that the detected average volume fraction of non-metallics in the sponge iron pellet forgings was less than the calculated min. theoretical value of 2.26 vol %. The amount of undetected fraction of non-metallics increased with decreasing preform density. It would appear that three types of inclusions were present in the sponge iron pellet forgings, particularly the lower density preform forgings:

(i) Very fine inclusions which would impart dispersion strengthening to the iron matrix

(ii) Medium size inclusions which would not affect the mechanical properties of the pure iron matrix

(iii) Relatively large inclusions which would be harmful to the mechanical properties.

On comparing the mechanical properties shown in Table 10.VIII, it seems that the relative proportion of the inclusions of different sizes and the amount uncounted in the sponge iron pellet forgings were such that their nett effect on the strength of the matrix were to increase it, while on the elongation the effects were to reduce it.

Backstiegel and Blände[65] also investigated the influence of slag inclusions on the mechanical properties of powder forged ASC 100.29 atomised iron powder in which iron powder was deliberately contaminated with varying amounts (up to 1.3 vol.%) of coarse (150 - 180μm) and fine (>60μm) slag inclusions. They found that increasing amounts of slag inclusions slightly increased the tensile strength and reduced the elongation for the forgings containing coarse and fine added inclusions. Increasing amounts of slag inclusions produced a sharp drop in

impact strength and higher percentages of coarse inclusions were found less harmful than fine ones. In the present case, the sponge iron pellet preforms behaved in a similar way. With increasing preform density non-metallic inclusion size increased and the impact strength increased (Figs. 9.24 and 9.25). It should be noted that the impact values generally were low compared to relatively clean full density ASC 100.29 iron powder forgings ($\sim$130J which is extracted from ref.(65)) because sponge iron pellet forgings contained a large amount of non-metallic inclusions.

Grain size measurements indicated that the grain size of forgings increased with increasing preform density in both the as-forged and annealed conditions; the annealed condition giving the finer grain size (Section 10.2). Sponge iron pellet forgings produced smaller grains than NC 100.24 iron powder forgings whereas NC 100.24 iron powder pellet forgings had intermediate grain sizes. When the U.T.S. values of these three materials are compared with grain size the sponge iron pellet forgings have higher tensile strengths than both the NC 100.24 iron powder pellet forgings and iron powder forgings even though they have similar inclusion contents. This is corroborative evidence that the sponge iron pellet forgings of the same grain size as iron powder forgings contain a higher amount of fine non-metallic inclusions which result in higher tensile strength.

As a result, the mechanical properties of sponge iron pellet forgings compared very closely with those of iron powder forgings (similar inclusion - containing - matrix). The U.T.S. of the sponge iron pellet forgings was greater than that of the iron

powder forgings, while the percent elongation was lower. The inclusions imparted a slight dispersion strengthening and substantial grain refinement strengthening to the sponge iron pellet forgings, noticeable at lower preform density forgings.

10.4. BEHAVIOUR OF THE SPONGE IRON PELLET FORGINGS IN MECHANICAL TESTING

Fig. 10.24 shows some typical force-extension curves of sponge iron pellet forgings, NC 100.24 iron powder pellet forgings and NC 100.24 iron powder forgings, all hot forged from two extreme preform densities at 1100°C and annealed at 700°C for 1 hour in H_2. The interesting feature of the curves for all materials is the absence of an obvious yield point for the high preform density forgings. But for the lower density preform forgings the yield point is apparent.

Fig. 10.25 shows that significant necking did not occur before fracture. Fractured impact test specimens are also shown in Fig. 10.26. However, necking increased with increasing preform density and the reduction in area after fracture was rather low. This is attributed to the high oxygen content of the forgings. Tensile test results[134] on stainless steel with an oxygen content as high as 0.5 wt.% have demonstrated that the material had a strength and elongation comparable with those of relatively oxygen-free material, but the reduction in area dropped very rapidly as the oxygen content increased. This resulted in a steel that did not neck down at all during tensile testing.

Figs. 10.27 - 10.30 show some typical scanning electron micrographs of the fractured surfaces of the forgings produced

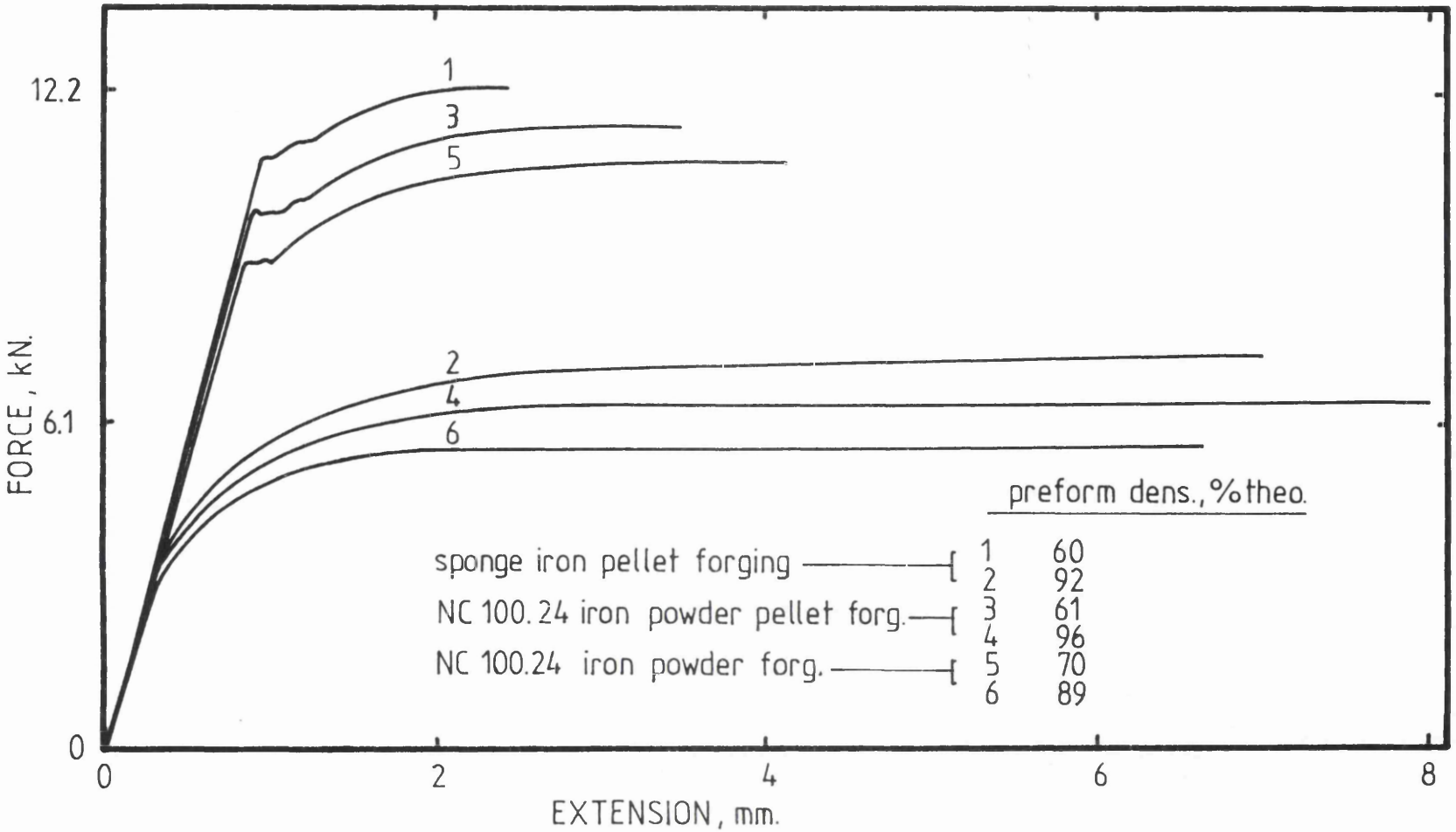


Fig. 10.24. Typical Force/Extension Curves of Forgings.

Fig. 10.25. Tensile test specimens of forgings after fracture.

SPONGE IRON PELLET FORGINGS	preform density, % theo.	NC 100.24 IRON POWDER PELLET FORGINGS	preform density, % theo.	NC 100.24 IRON POWDER FORGINGS	preform density, % theo.
	49		50		
	69		72		70
	92		96		89

Fig. 10.26. Izod impact test specimens of forgings after fracture.

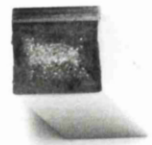
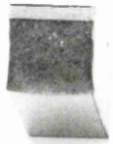
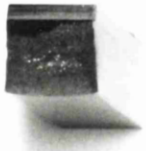
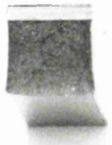
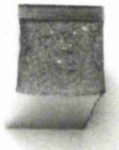
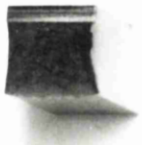
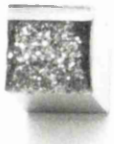
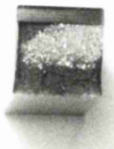
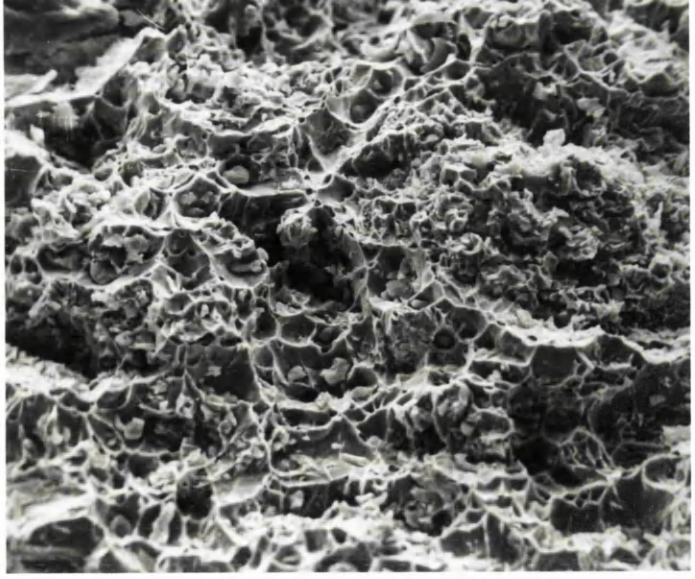
SPONGE IRON PELLET FORGINGS	preform density, % theo.	NC 100.24 IRON POWDER PELLET FORGINGS	preform density, % theo.	NC 100.24 IRON POWDER FORGINGS	preform density, % theo.
	49		50		
	69		72		70
	92		96		89

Fig. 10.27. Fracture surfaces of sponge iron pellet forgings produced from preforms of
(a) 49%,
(b) 75%,
(c) 92% theo. density
X450



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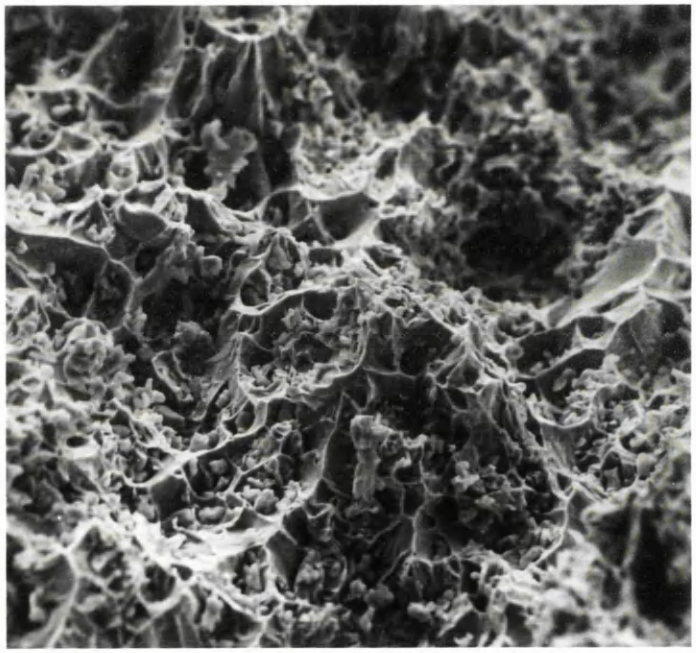

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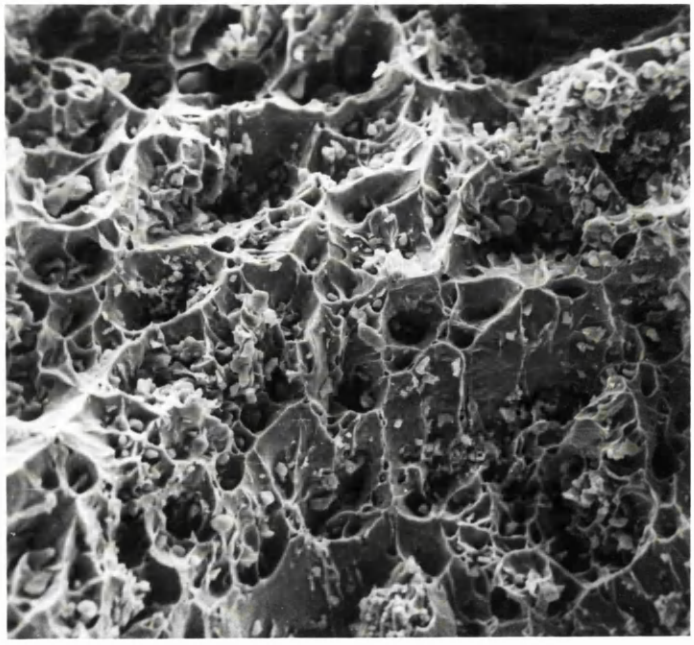
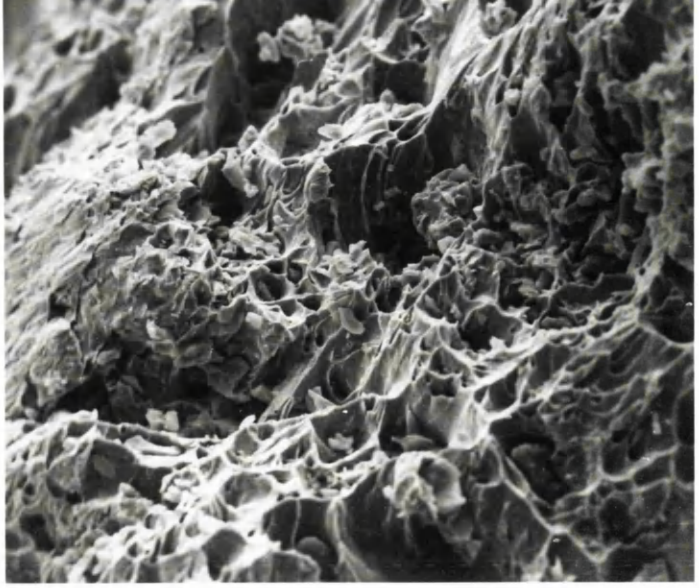


c

Fig. 10.28. Fracture surfaces of NC 100.24 iron powder pellet forgings produced from preforms of
(a) 50%,
(b) 72%,
(c) 96% theo. density.
X450



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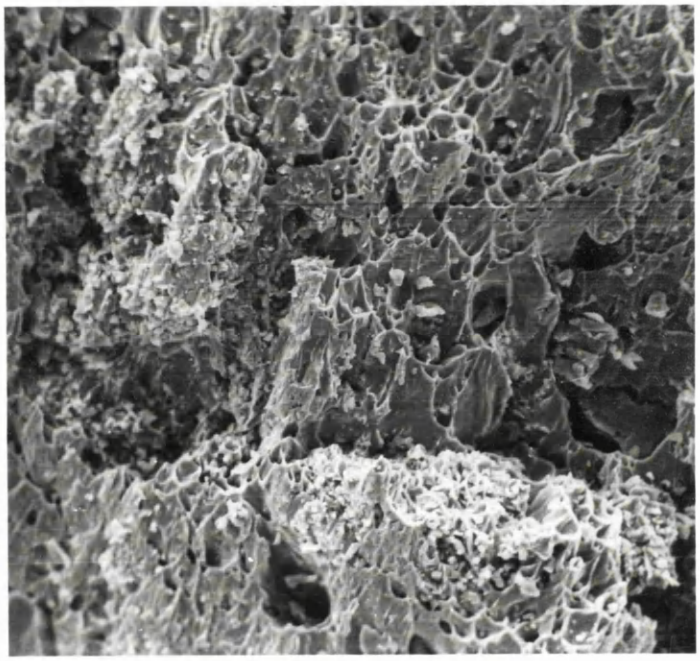
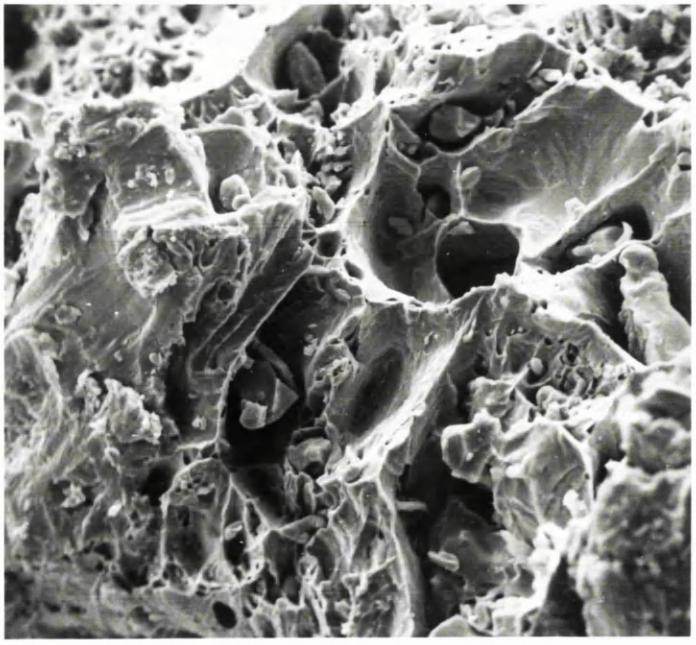
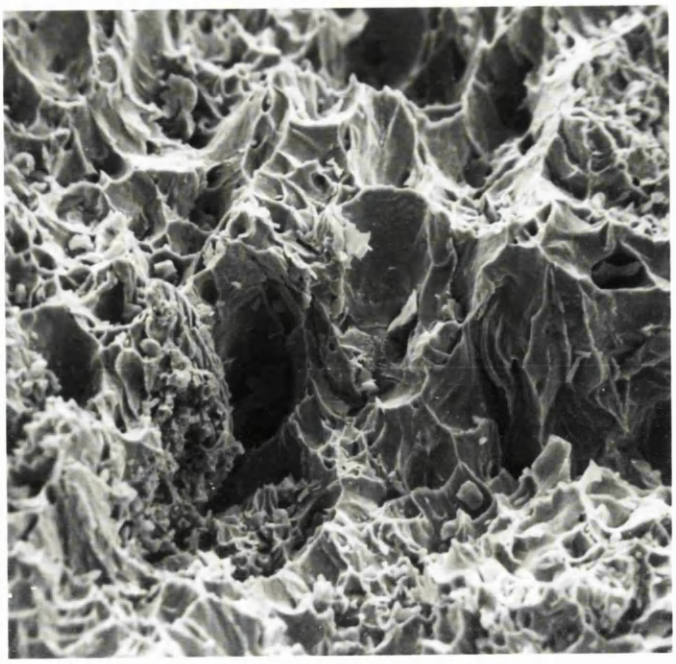


b



c

Fig. 10.29. Fracture surfaces of NC 100.24 iron powder forgings produced from preforms of
(a) 70%
(b) 76% theo. density
X450



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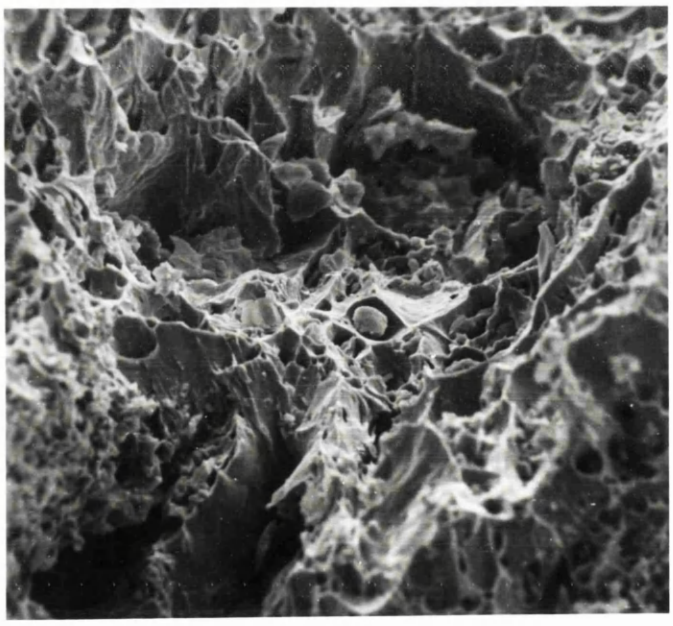
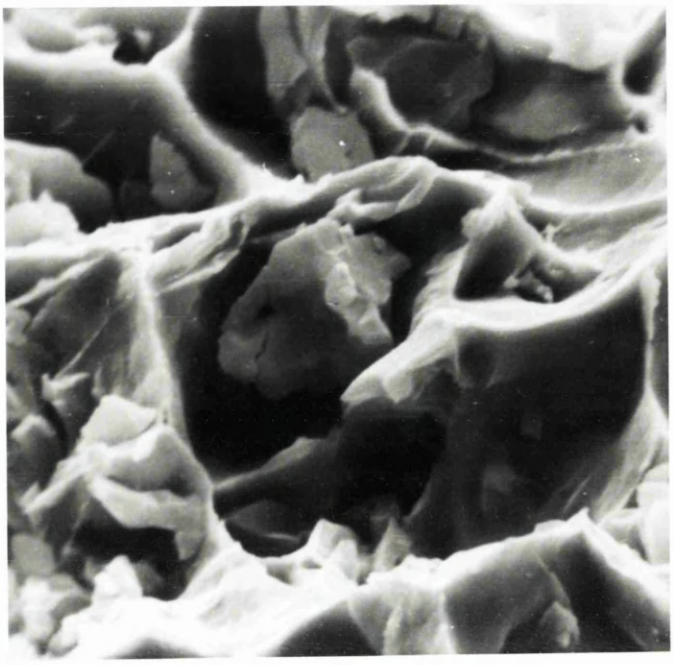
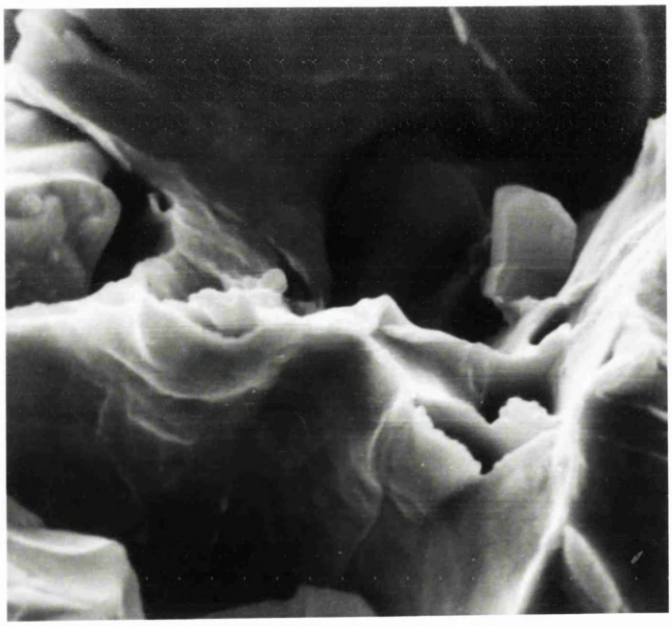


b

Fig. 10.30. Fracture surfaces of
(a) sponge iron pellet forgings produced from 69% theo. density preform,
(b) NC 100.24 iron powder pellet forging produced from 80% theo. density preform.
X4,500



a



b

in this investigation, illustrating the presence of characteristic ductile fracture dimples. Many of the dimples contained inclusions in the centre and there were not many inclusions which were not fractured during tensile testing. Examination of the surface of the tensile specimens after fracture revealed many voids formed around the inclusions near the fracture edge. The cracks were formed at the interface of the matrix and large inclusions, in preference to that of matrix and small inclusions.

In the case of ductile metals containing hard second phase particles, e.g. dispersion strengthened materials, it has been reported[135] that a ductile fracture in these materials took place in three stages:

(i) formation of cracks at the matrix/second phase particle interface,

(ii) growth of the cracks,

(iii) coalescence of the cracks leading to failure.

From the observations made, it appears that a similar mechanism applies to the ductille fracture of sponge iron pellet forgings. However, it might be possible that the cracks were already present at the matrix/inclusion interface of the forgings or that they were already present in the non-metallic inclusions then grew during tensile testing, and finally coalesced, leading to fracture.

The matrix/inclusion bond in a composite material, such as sponge iron pellet forgings, is greatly dependent on the manufacturing technology. For example, it has been reported[137] that when liquid metal technology, i.e. mixing the particles with a liquid metal and then solidifying it, is used to make the

composite material, the matrix/inclusion bond depends upon the relative thermal contraction of the matrix and inclusion. Iron, containing SiO_2 inclusions, prepared by the above method, did not show any voids surrounding the inclusions, suggesting that the bond was good, while the iron containing FeO inclusions showed cracks at the inclusion/matrix interface because of the difference in thermal contraction.[137]

The method of preparation of forgings consisted essentially of hot forging the sponge iron pellet preform at 1100°C followed by annealing. The material was not liquid at any stage of the processing used, and therefore relative thermal contraction of the inclusions and iron has less significance in this context. Moreover, the hot forging pressure, coupled with the ductile nature of sponge iron at the hot forging temperature, was sufficient to form a good bond between the inclusions and iron.

CHAPTER 11

TECHNICO-ECONOMIC CONSIDERATIONS

The properties of sponge iron pellet forgings with different process variables, as discussed in the last three chapters, indicated that hot forged and annealed forgings with U.T.S. from 300 MNm^{-2} to as high as 655 MNm^{-2}, coupled with elongations of 29% to 10% dependent on preform density, can be obtained from a 99.3% purity magnetite superconcentrate by the proposed powder technology route. The finished forging is essentially a fine grain pure iron with a dispersion of fine non-metallic inclusions. The strength of the sponge iron pellet forgings is similar to NC 100.24 iron powder pellet forgings and NC 100.24 iron powder forgings, although the structure is different. Sponge iron pellet forgings contained a uniform dispersion of non-metallic inclusions, in greater amounts than the iron powder forgings; the median size was smaller, depending upon the preform density, and most of the inclusions were present in the size range sub-micron to 6 μm. The small inclusions enhance the rate of nucleation during annealing, restrict the grain growth, and hence help in the development of a fine grain size in the finished forgings.

The proposed new route has all the advantages of the traditional P/M forging routes starting from iron/steel powder. In addition, because the sponge iron pellets are inherently highly porous, it is, therefore, possible to make even 50% theoretical density preforms with enough strength to carry out the subsequent process step. Moreover,

the starting material is a cheap iron oxide superconcentrate powder, instead of expensive iron/steel powder.

Sponge iron pellet forgings in the present investigation were made by the proposed route on a batch basis, involving separate operations. The process at the various important stages is shown in Fig. 11.1 illustrating its simplicity and compactness.

No attempt was made to obtain forgings on a large scale operation in which the different unit steps could be integrated, and therefore it is not possible to comment on this aspect. Nevertheless, an attempt could be made to examine the technico-economic considerations involved in the process if adopted for large scale production.

Magnetite superconcentrate was used as raw material in this case. But there is no reason why cheap haematite superconcentrate cannot be used. The purity of the iron oxide concentrate is the main concern because all the impurities ultimately appear in the final product. To maintain a suitable product composition it is therefore necessary to remove as much of the undesirable impurities as possible from the superconcentrate at the very beginning of the process, especially when impact strength is considered. In the case of magnetite, wet magnetic separation in various stages, followed by froth flotaion if necessary, is the most widely used method for superconcentrate production. Lawver and Hays[138] proposed three ways of concentrating haematite ores:

(i) an all froth flotation process.

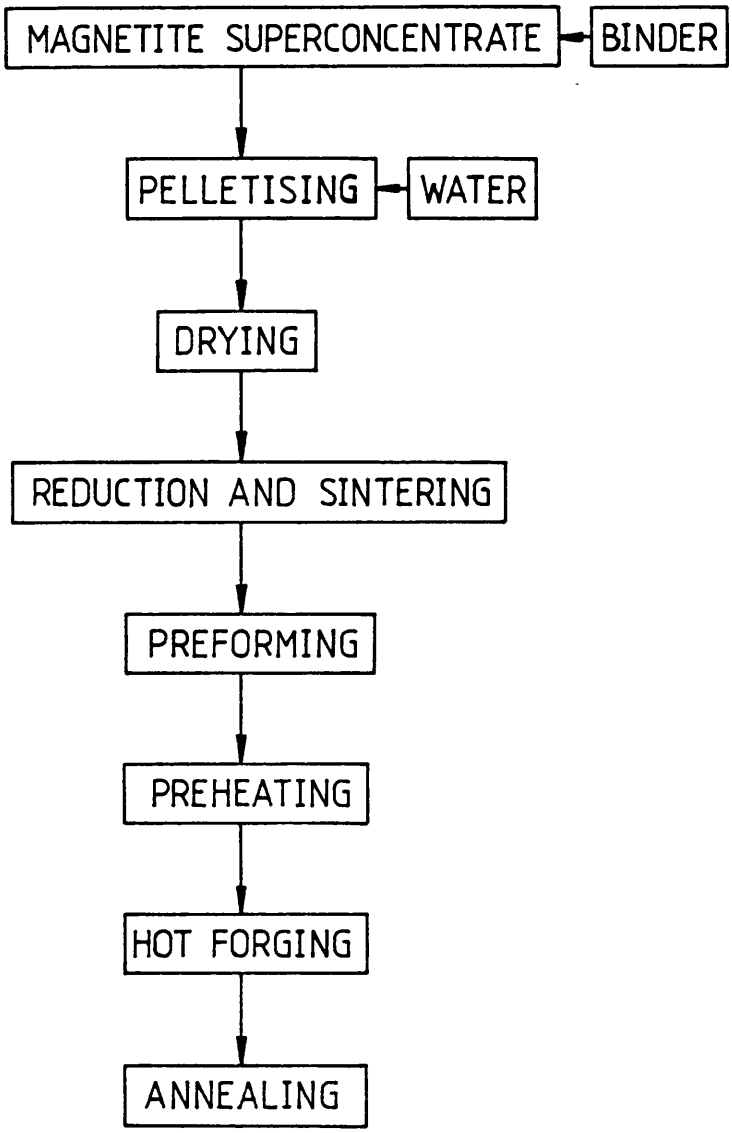


Fig. 11.1. Important stages of the process.

(ii) a magnetic roasting process, followed by flotation

(iii) a process consisting of high intensity wet magnetic separation, followed by flotation, if necessary.

The high intensity wet magnetic separation-flotation method is considered to be the most economical way of concentrating semitaconite and haematite ores. Notable superconcentrate sources are Brazil, Peru and Sweden. Superconcentrates are already made on a small scale in Sweden as a source of iron powder and production can easily be increased in response to market demand.[140]

The binder material (Celacol in this work) would involve appreciable cost, but the use of a resin-type binder might be cheaper. Ahier and Singer[80] investigated the use of bitumen as a binder for making high-purity sponge pellets. Bitumen is cheap and widely available either in its naturally occurring forms or as residues from oil refining. It also has the advantage of conferring high strength on the pellets after low temperature baking (200°C). This may be contrasted with the high temperature firing (1300°C) required for bentonite bonded pellets.

Pelletising of finely ground concentrates is a high volume, low-cost operation already carried out on a large scale worldwide, and no particular problems are envisaged in the pelletising of superconcentrates once the organic binder has been added as a first step.[80]

Another raw material required in quantity by the process is gas for the reduction and sintering operation. The reducing gas/gases can be obtained in large volumes and

at low cost by the steam reforming of naphtha or natural gas to produce a hydrogen-carbon monoxide mixture, and the technology is well established.[82] The reducing gas could also be obtained from low grade coal.

The technical requirement and cost of a reduction and sintering furnace could present a problem. But, recent work in the University College of Swansea[81] has developed a rotary kiln furnace capable of reducing up to 10 kg of green concentrate pellets using a gaseous reductant. The sticking of pellets either to themselves or to the kiln walls was eliminated.

The subsequent preforming, preheating and hot forging operations would be carried out in the same way as in powder preform forging.

One of the objectives of the proposed new technique as an industrial process would be to produce lower cost steel forgings and perhaps challenge some of the casting processes. Even though sponge iron pellet forgings were obtained using laboratory equipment and operating on a small batch basis, it seems likely that the structural and mechanical properties obtained would also be produced from an industrial unit working on a tonnage basis.

CHAPTER 12

CONCLUSIONS

(1) Steel forgings can be prepared from magnetite superconcentrate by the sponge iron pellet forging route. The following processing sequence is necessary to produce properties at least equal to forgings made from equivalent materials on a laboratory basis involving separate operations: Mixing of magnetite superconcentrate powder with Celacol binder and water $\longrightarrow$ Pelletising $\longrightarrow$ Drying $\longrightarrow$ Combined reduction and sintering at 1100°C for 2 hours in an H_2 atmosphere $\longrightarrow$ Cooling in an inert atmosphere for 15 minutes $\longrightarrow$ Preforming to predetermined density in a closed die $\longrightarrow$ Heating to 1200°C for 20 minutes in H_2 $\longrightarrow$ Hot forging in a closed die at 1100°C with minimum of 925 MNm^{-2} $\longrightarrow$ Cooling in air $\longrightarrow$ Annealing at 700°C for 1 hour.

(2) The reduction of static magnetite superconcentrate pellets at 1100°C was found to be acceptable. Sticking of the pellets either to each other or to the pellet container did not present any major problem.

(3) Sponge iron pellets exhibited two basic forms of porosity, intra-particle and inter-particle.

(4) Sponge iron pellets during preforming receive a large amount of deformation even when 50% theoretical density is produced; these low density preforms are sufficiently strong to be machined.

(5) Three types of porosity are present in preforms; these are inter-pellet, inter-particle and intra-particle. This results in the inter-pellet porosity being small even for the 50% density preform.

(6) Because of (4) and (5), oxygen penetration into the preform is limited during transfer of the hot-preform to the hot forging die.

(7) Among the process variables studied, preform density and annealing treatment have significant effects on the mechanical properties of the sponge iron pellet forgings. Pellet size and type of deformation have lesser effects.

(8) The U.T.S. of the sponge iron pellet forgings increases with decreasing preform density while elongation and impact strength increase with increasing preform density. This is probably because of the die chilling during hot deformation and extensive deformation applied to the lower density preforms during hot forging which result in smaller grain size.

(9) U.T.S. values increased with annealing treatment compared with as-forged values because the inclusions impart a substantial grain-refinement strengthening to the iron matrix during annealing. Annealing treatment has a small effect on the elongation and impact strength.

(10) Mechanical properties of the sponge iron pellet preforms are similar or better than those of NC 100.24 iron powder pellet forgings and NC 100.24 iron powder forgings.

(11) The structure of the sponge iron pellet forgings is essentially a fine grain pure iron loaded with ~2.0 volume % of non-metallic insoluble fine inclusions, distributed uniformly throughout the matrix. The size of the inclusions decreased with decreasing preform density. The inclusions were generally too large to impart dispersion strengthening. However, they accelerate the nucleation of freshly recrystallized grains during annealing and also restrict the grain growth, thereby resulting in a fine grain size in the finished product. The grain size reduces with decreasing preform density and deviation from mean grain size is small.

(12) The mechanical properties of the sponge iron pellet forgings prepared from a 99.3% pure (and, it seems, even from a 99.0% pure) magnetite superconcentrate, are quite adequate for adopting this material for many uses where high ductility and impact strength are not essential.

(13) It would appear that the process would lend itself very well to the manufacture of larger rather than smaller components. This follows because the pellets are significantly larger than powder particles and could give inadequate die filling for small forgings.

SUGGESTIONS FOR FURTHER STUDY

(1) Although a reasonably good indication of the properties of the sponge iron pellet forgings can be obtained from the results of the present investigation, it will be necessary to investigate other properties such as fatigue, friction and wear, joining, corrosion, etc., in order to have a full knowledge of their properties.

(2) Further investigation should be made to optimise the processing conditions to obtain optimum properties at various levels of deformations.

(3) Further investigation should be carried out to establish more clearly the effects of such parameters as forging temperature, annealing time and temperature on the mechanical properties of forgings.

(4) The problems associated with large deformations and die movements should be investigated.

(5) The effect of chilling caused by large die movements should be investigated.

(6) The means of carbon introduction into the forgings and the properties of these forgings should be investigated. One possible way, which does not require any extra processing step in the adopted route, might be to add carbon to the starting magnetite superconcentrate, such as carbon or graphite powder, or a suitable organic binder (bitumen) which would provide carbon.

(7) It seems likely that the properties of the sponge iron pellet forgings prepared from a lower grade iron oxide concentrate would be adequate for many applications, and therefore the properties of such low cost steel forgings should be investigated.

(8) Haemetite concentrates should also be investigated.

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