HIGH STRENGTH POWDER METALLURGY MATERIALS

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CONTENTS

SUMMARY

The main factors controlling the mechanical properties and dimensional stability of sintered iron base alloys are discussed in detail in the literature review of this thesis. From this, the need to examine in detail the iron-carbon-copper-nickel system is established. In order to clarify the industrial aspects of the project, the review also contains a description of the basic stages in the ferrous powder metallurgy process, and is concluded with a cost analysis containing a detailed breakdown of the costs involved in producing a component by the powder metallurgy route and by machining from bar stock.

The experimental work was planned statistically, using a factorial design. The object was to determine the significance of the effect of alloy composition and as-pressed density on the strength and dimensional stability of sintered iron-carbon-copper-nickel alloys, prepared from pre-mixed elemental powders. The magnitude of the significant effects has also been determined using multiple regression analysis.

The importance of as-pressed density in determining mechanical properties, is confirmed. A number of main effects and interactions between variables, particularly between alloying elements, appear to have a significant effect on strength and dimensional stability during sintering. The multiple regression equations obtained are complex since they contain a number of interaction terms and the magnitude of the effect of each variable, particularly alloying elements, is highly dependent on the

levels of the other variables. A second regression analysis was made using the results obtained from alloys of "commercial significance". Thus, the highly alloyed materials were excluded on the basis of poor properties and expense.

Mechanisms involved in strengthening and dimensional control are briefly discussed to illustrate that there is some agreement between the effects observed from this investigation and those proposed in published literature.

1 INTRODUCTION

The powder metallurgy process is by no means a new technique and early examples of its use date back to the Egyptians who produced iron implements by reducing iron ore with charcoal and forging the resultant sponge iron to the desired shape. One of the most outstanding examples of the early use of powder metallurgy is the famous Dehli pillar in India. This pillar weighs approximately six and a half tons and was made in 300 BC by joining several blocks of reduced iron ore.

As high grade iron and other metallic ores were discovered in large quantities, extraction, melting and casting techniques were developed. In the early 19th Century, many metallic elements could be extracted in the powder form by chemical means, and there was considerable interest in the consolidation of these powders, especially those which could not be melted. Thus, the real foundation of the powder metallurgy industry was the work of Wollaston[1] in England and Sobolevskie in Russia who, in the early 19th Century, produced platinum ingots from platinum powder. This was followed in the 1920's by the work of Coolidge[2] who produced ductile tungsten wire for incandescent light bulb filaments by sintering tungsten briquettes and repeatedly hot working the metal until it was sufficiently ductile to be drawn at relatively low temperatures.

Following these early uses, the range of applications grew such that prior to World War II powder metallurgy techniques were applied to the production of:

i sintered refractory carbide dies used mainly for the drawing of tungsten wire, which soon developed into the production of cemented tungsten carbide for cutting tools, rock drills, etc.

ii copper-graphite bushes for electric dynamos and motors

iii self-lubricating bronze and iron bearings

iv porous metal filters

v small engineering components

Throughout this period, the emphasis was on the production of sophisticated multiphase systems which were difficult to produce by other techniques rather than the mass production of components from relatively simple metallic systems.

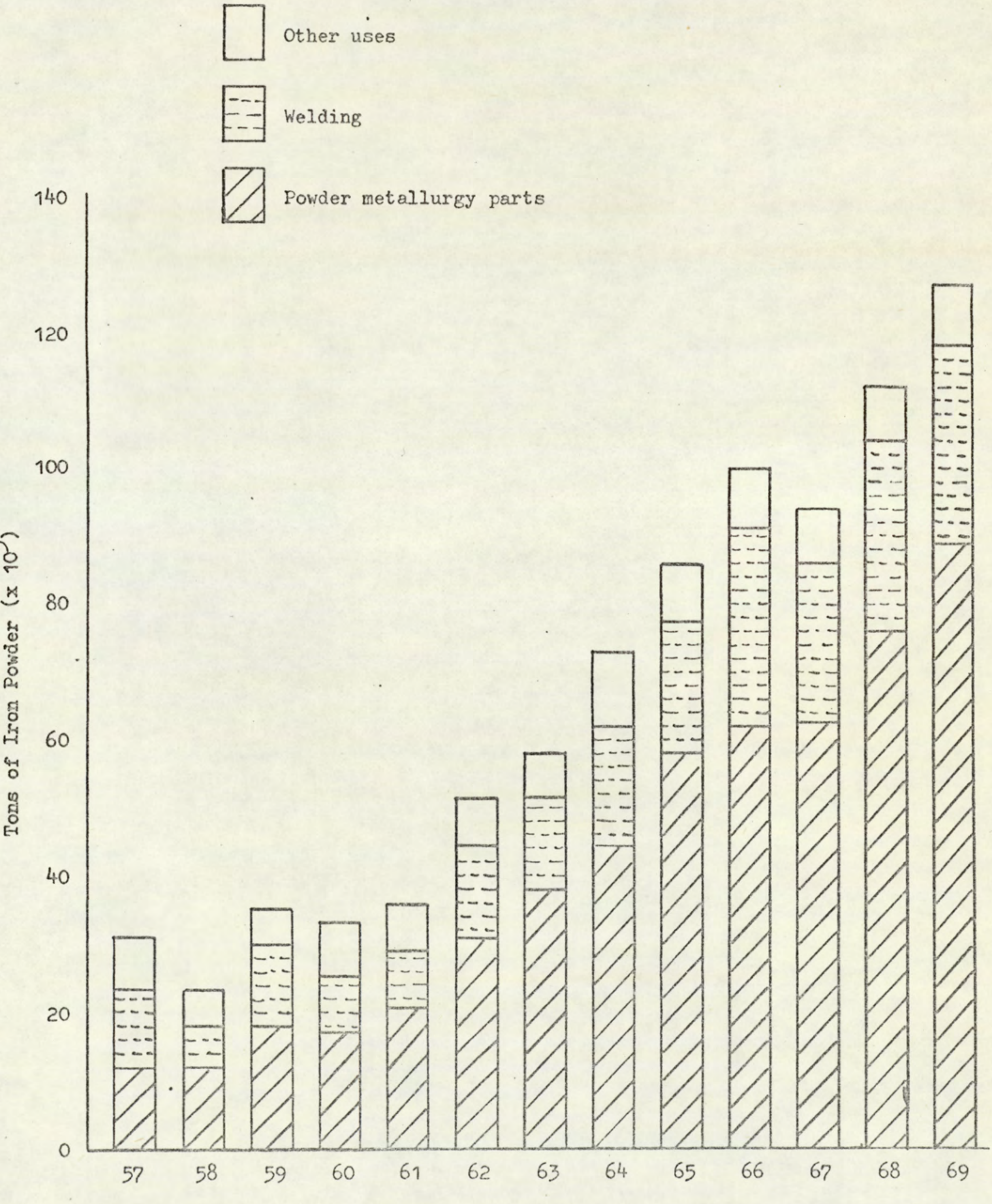
Since the war, there has been a major development in the use of powder metallurgy techniques for the manufacture of engineering components for it was soon realised that certain shapes could be produced more efficiently and cheaply by powder metallurgy techniques than by the conventional casting, forging and machining route. Traditionally, powder metallurgy parts were simply space fillers since there was a lack of confidence in components which contained pores and generally had a much lower strength and ductility than similar wrought or cast components. However, this lack of confidence was overcome by assessing the performance of sintered parts on the basis of service and simulated service tests rather than the interpretation of mechanical property test results.

Increased confidence in the use of sintered components, combined with a greater appreciation of the advantages of the powder metallurgy process, has produced a rapid expansion of the component industry. The most significant growth has been in the field of iron base components and Figure 1 shows the growth of this industry and the annual production of iron powder in the USA since 1950.

No detailed information concerning the volume of the UK iron powder parts industry has been published, and both component manufacturers and the British Metal Sintering Association are extremely reluctant to provide any useful statistics. However, several estimates have been reported and it appears that the UK production of sintered parts is three to eight years behind that of America.[3,4] A detailed quantitative analysis of the UK industry has been presented by Derry and Tron[4] who claim that the UK consumption of iron powder parts is six years behind that of the USA. However, this analysis is based on extremely limited information concerning the UK position in 1965 and comparisons between the two industries are based on per capita consumption and estimated consumption by the motor industry. The main reasons suggested for this estimated lag are a greater appreciation, in the USA, of the economic advantages of the powder metallurgy technique, particularly with regard to labour utilisation, and the availability of larger presses. The component industry accounts for the majority of iron powder consumption and its share of the total US iron powder market has grown from ~59% in 1962 to >70% in 1969, the remainder being used in magnetic and electronic appliances, welding electrodes, etc.

Figure 1

Consumption of Iron Powder in USA 1957-1969



The largest consumer of powder metallurgy parts is the motor industry which accounted for ~60% of the total US component production in 1965. However, due to the increasing use of sintered components in other industries, eg business machines, domestic appliances, etc (see Table 1), the motor industry's share of the US powder metallurgy parts market decreased to ~45% in 1970.[5] Although the British industry is lagging behind the Americans, the British car manufacturers are well aware of the availability of sintered parts, as shown in the BLMC 1100 which weighs ~16 cwt and contains 11 lb of sintered parts, viz: 14 lb/ton of car, compared with an average American car of almost twice this weight containing ~16 lb of sintered parts, viz: ~10 lb/ton of car.[6]

Table 1

Applications of Powder Metallurgy Parts in the USA[5]

Application	1965	1970
Motor industry	60%	45%
Appliances	16%	18%
Farm and garden, hardware, tools	10%	17%
Business machines	6%	8%
Hobby and sport	5%	6%
Others	3%	6%
Ratio of ferrous to non ferrous	7:3	6:2

Originally, powder metallurgy components were limited to ~½ lb in weight with a maximum cross-sectional area of ~2 sq.in, compared with component weights of 10-50 lb and cross-sectional areas up to 50 sq.in which are achievable today. The major reason for this size limitation was the limited loading capacity of early presses which had a load range of 5-50 tons compared with modern press capacities of thousands of tons. In fact, many early components were compacted on pharmaceutical tableting presses. A further consideration was the inferior compressibility of early powders arising from the presence of impurities, but improved production techniques have resulted in purer, higher quality, higher compressibility powders.

Although advances in alloying techniques and alloy powder production have enabled material costs, for specified mechanical properties, to be reduced, material costs still form a major proportion of the final cost of large sintered parts, thus reducing the effect of the advantages of the process, and hence the powder metallurgy route is usually used to produce large numbers of parts weighing only a few ounces. However, the technique is extremely competitive in the production of large complex parts which are difficult to produce conventionally and it can also offer considerable cost savings in situations where extensive machining operations, used in more conventional techniques, can be reduced or completely eliminated.

The development of larger loading capacity presses and improvements in the purity and compressibility of iron powder have been accompanied by improvements in sintering furnace design and operation, and in the production and control of protective atmospheres. Such developments, combined with improvements in the design and operation and manufacture of tools for the compaction of metal powders, have enabled powder metallurgy techniques to be extended to the large scale production of relatively complex shaped components (Figure 2).

The performance of modern components compares favourably in many applications with those made from wrought material, whereas the properties of early components tended to be inferior. This improvement is a result of:

a improvements in alloying and heat-treatment techniques

b the use of new alloying elements

c improved iron powder purity

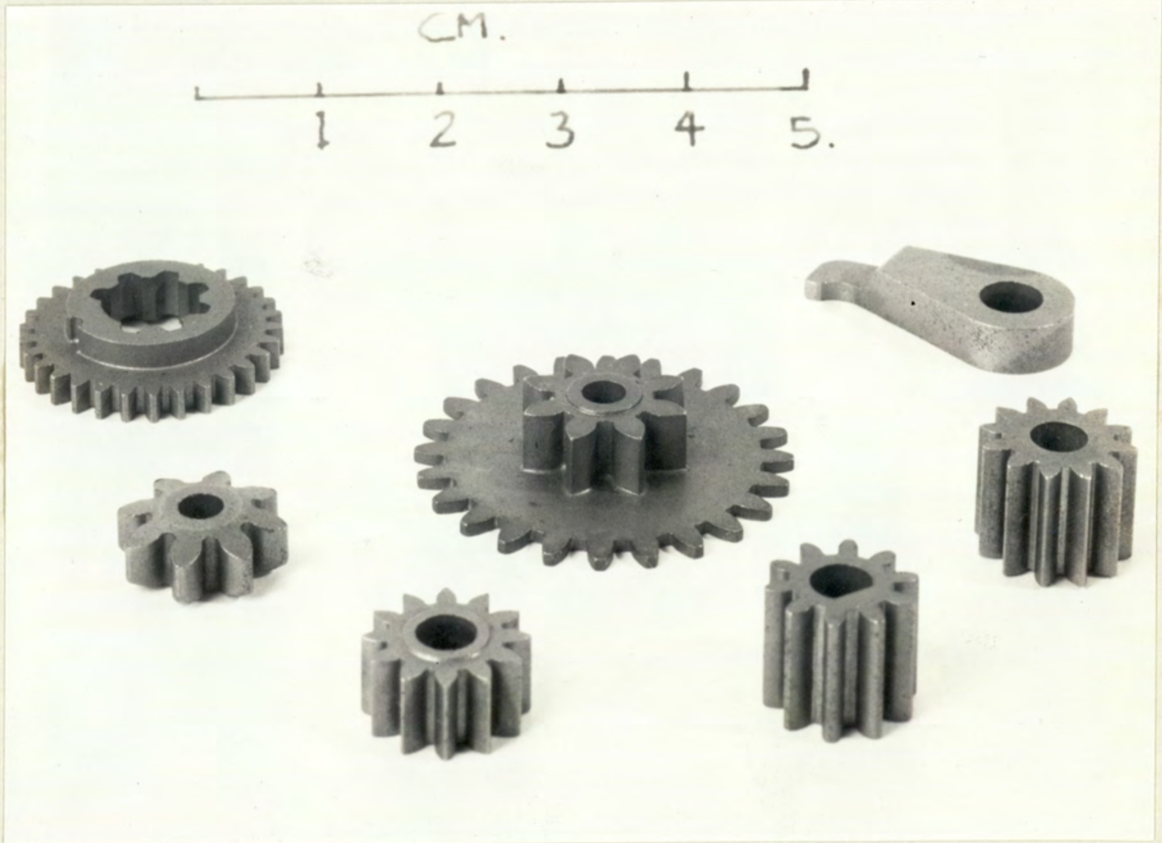
d attainment of higher densities

Today, it is even possible to produce certain materials by specialised powder metallurgy techniques which have superior mechanical properties to those of the wrought material and strengths of the order of 100 tsi (1544 MN/m^2) can be achieved in sintered alloy steels. High levels of ductility and toughness can also now be achieved and hence old ideas of completely brittle and fragile powder metallurgy materials are no longer valid.

These improvements in mechanical properties have also been accompanied by improvements in the control of the dimensions of sintered parts and the dimensional accuracy of modern components is comparable, and in many cases superior, to that of parts produced by other precision forming techniques, eg precision casting,

Figure 2

Typical Powder Metallurgy Parts



machining, cold forging, precision stamping, etc. Tolerances of ±.001 in/in (±.03 mm/mm) can be readily maintained and even closer control is possible in certain specific dimensions. Unlike some other forming techniques, eg cold forging, precision casting, this close control of dimensions can be achieved during the production of complex shapes, some of which cannot be produced by any other technique, without any significant decrease in the production rate.

The possibility of varying the density of powder metallurgy parts provides engineers, designers, etc with an additional and unique material property. Porosity, via density, can be closely controlled and hence parts such as counter balance and governor weights, where controlled weight/density ratio is essential, can be easily produced. The damping characteristics of a material can also be controlled by controlling porosity. The pores dampen mechanical vibration and can in some cases, due to surface bedding-in effects when parts are put into service, improve mechanical fatigue strength and wear resistance. Improved damping characteristics can also lead to a reduction in the noise level of consumer goods, such as typewriters. The presence of porosity also enables parts to be impregnated with a suitable lubricant and hence the application of the powder metallurgy technique in the production of self-lubricating bearing. Medium strength structural components can also be oil impregnated and hence provide an additional self-lubricating property. However, the most obvious effect of porosity is that it reduces the weight of the component, giving advantage to sintered parts in applications where there are weight limitations. Reductions in component weight also increase its specific strength, ie strength/weight ratio, and reduce material costs.

As far as the future of the powder metallurgy industry is concerned, a major increase in the demand for sintered components for use in cars and consumer products is expected within the next few years. The main reasons for this are that engineers are becoming more familiar with the use of sintered components and, as labour costs rise and become a greater consideration in the total cost, the advantages of powder metallurgy techniques become more obvious.

At the present time, the main areas of expansion appear to be in the manufacture of components which are relatively large, geometrically complex and of high strength. The strength of sintered parts can be increased by increasing density, alloying and heat treatment. In the past, high densities have been achieved by infiltration or repressing after sintering but since both of these techniques are expensive, several alternative techniques have recently been investigated. Of these, the process of "sinter forging", which can produce forged components weighing 10-15 lb and of almost 100% density, appears to have the greatest potential. It has been suggested that this technique may eventually replace conventional drop forging but at least one industrial source, viz: GKN, estimates that at the most only ~15% of the drop forging industry will be replaced. At the present time, the technique is only at the development stage and a number of economic and technical factors have yet to be overcome before the technique is in extensive use, viz:

a The only suitable powder currently available is A. O. Smith 4600 which is expensive and hence the use of alternative powders is being investigated. The majority of cost analyses that have been carried out are based on a predicted reduction in powder price derived from an assumed increase in demand. However, there are as yet no firm predictions of when this reduction will be forthcoming.

b Available powder metallurgy equipment, viz: compacting presses and tools, sintering furnaces, etc, are in relative short supply and it is doubtful, particularly in the UK, that present facilities could cope with a sudden large increase in demand.

c Die wear remains the largest unknown feature and is considered to be one of the major factors restricting the expansion of the process.

d Large amounts of capital are at present invested in conventional forging equipment and manufacturers are unlikely to be enthusiastic about investing large amounts of money in a process that could make their present equipment obsolete.

The use of powder metallurgy techniques in the fabrication of exotic materials, eg refractory metals, which are difficult to fabricate conventionally, should expand and as metal powders are the end point of some non-ferrous extraction techniques, considerable interest is being shown in the direct fabrication of such powders by rolling and extrusion with the intermediate melting process being omitted. A powder rolling technique is already in use for manufacturing nickel strip directly from nickel powder extracted from sulphide ore concentrates. There is also considerable development work being carried out to evaluate the use of metal powders for the production of mild steel and stainless steel strip.

The introduction of sinter forging and powder rolling are expected to produce a major increase in the demand for iron powder and this, in turn, may lead to a reduction in its price, which has remained relatively stable for the last fifteen years. However, at present, iron powder production is approximately three times greater than demand and this serious over capacity must be taken up before any price reductions are possible. The possible effect of a reduced iron powder cost has been quantitatively assessed by Zapf[7] who claims that a reduction to 3.1p/lb (7.5d/lb) would enable 20,000 tons of screw machined parts to be replaced by sintered parts. He also claims that a further reduction to 2.9p/lb (6.9d/lb) would enable malleable irons to be replaced, and a third reduction to 2.4p/lb (5.75d/lb) would enable 60,000 tons of grey iron castings for the motor industry to be replaced. When the price of iron powder is reduced, the production of alloy steel structural parts by powder metallurgy will become even more attractive.

The development of steels for powder metallurgy components has generally been limited to manufacturers simply aiming to meet and maintain customer specifications. Much of this work has tended to be "ad hoc" and very few comprehensive investigations have been carried out. The assessment of the large number of variables within the powder metallurgy process and the determination of the significance and magnitude of the effect of these variables on mechanical strength and dimensional stability would involve a large amount of classical experimentation. It is even questionable whether all the information required concerning all combinations of variables could be obtained. However, it has been shown that a statistical approach to this type of problem can provide a reliable assessment of all factors using the minimum amount of practical work. The main part of this thesis describes the use of a statistically designed experiment, viz: factorial design, to determine the significance of the effect of alloy composition and as-pressed density on the strength and dimensional stability of sintered iron-carbon-copper-nickel alloys prepared from pre-mixed elemental powders. The magnitude of the significant effects is also assessed using multiple regression analysis.

2 THE FERROUS POWDER METALLURGY PROCESS

The conventional form of the ferrous powder metallurgy process, together with some of the more common secondary operations, is outlined in a flow diagram (Figure 3). The four basic stages are:

1 Powder production
2 Powder blending
3 Powder compaction
4 Sintering

2.1 Powder Production

2.1.1 Iron Powder Production

Several techniques are used for the production of iron powder and these are described in detail in various publications.[8,9,10,11] Some of the properties, costs, applications, etc, of various commercial iron powders are compared in Table 2.

2.1.2 Alloy Powders

Although the majority of iron base alloy components are produced from pre-mixed elemental powders, several other alloying techniques are available and Table 3 compares their use with that of pre-mixed powders.

2.1.3 Future Developments in Powder Production

Iron powders are economically produced by a number of different routes. A high level of control is achievable

FIG3 THE POWDER METALLURGY PROCESS

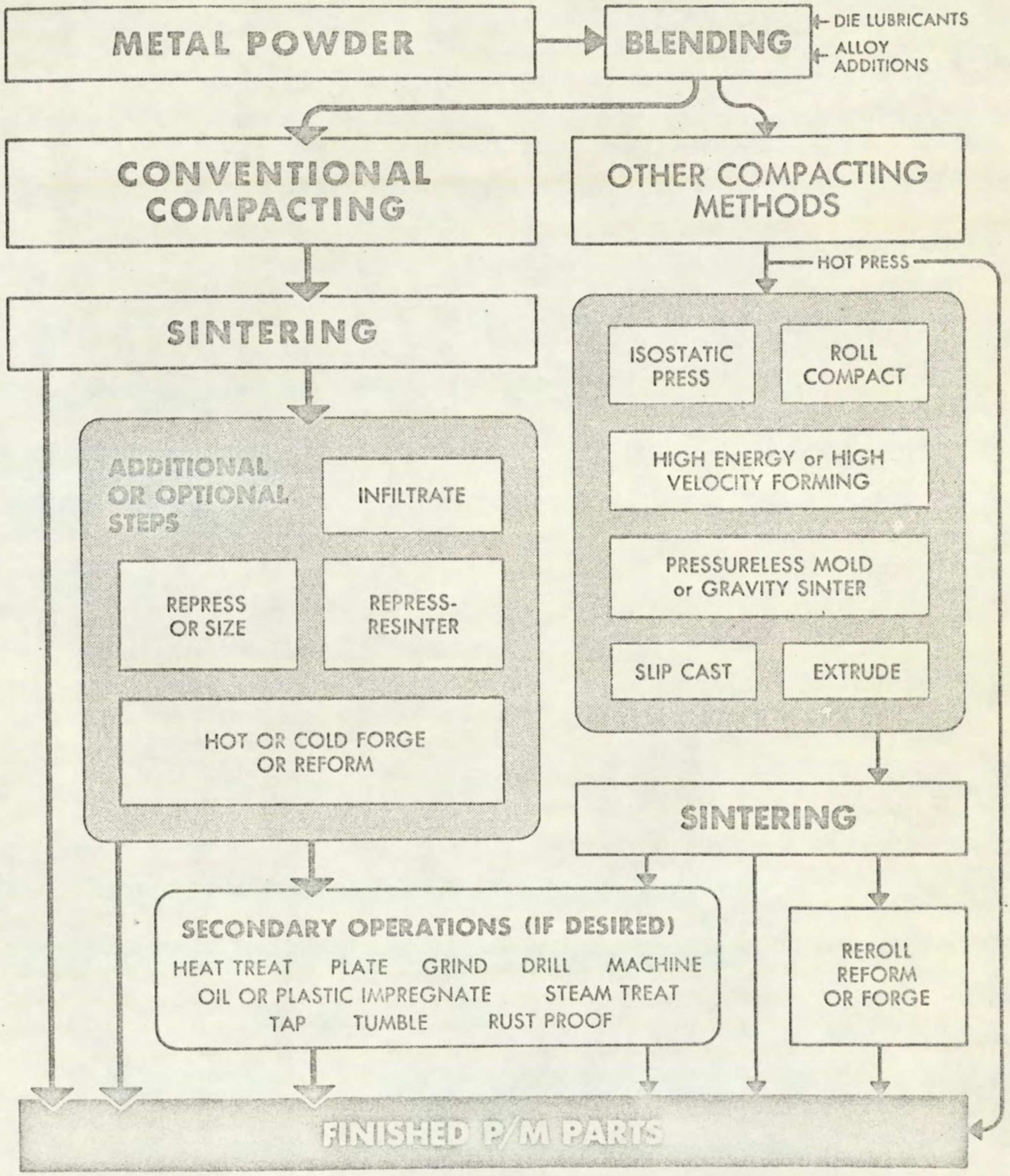


Table 2

Iron Powders

Powder	Particle Characteristics	Size Range	typical Cost P/lb New Pence	Major Manufacturers	Powder Properties	Applications
Oxide Reduced	Spongy, irregular	100 mesh	3.8	Hoganas - carbon reduction	Good green strength, good compressibility	Widely used in UK and USA for components Cheaper variety used for welding.
		300 mesh	6.3	Pyron - hydrogen reduction	Poor compressibility, shows no expansion when used to produce Fe/Cu alloys	Not widely used in UK - requires cheap source of hydrogen. Mainly used as additive to aid control of dimensions during sintering.
Atomised	Irregular spheres. Range of sizes, thus screening is essential and yield is below 100%	100 mesh 300 mesh	3.8 41.6	Hoganas Quebec Metal Powders Mannesmann Pulvermatall A. O. Smith Corporation	Generally purer than oxide reduced	Shares world market with oxide reduced powders for component production. Suitable for powder rolling.
Electrolytic	Dendritic	100 mesh 300 mesh	9.5 14.2	George Cohen	Pure. Can be pressed to high densities with magnetic properties almost equivalent to Armco iron	Production of high density powder metallurgy components. Magnetic components.
Carbonyl	Uniform spheres	Approximately 5 mesh narrow range	37.5	International Nickel	Pure. Poor flow properties due to fine particle size. Highly reactive, hence requires lower sintering temperatures.	As an addition to other powders to improve strength and dimensional stability. Production of magnetic dust cores, special alloys and controlled thermal expansion alloys. Mainly used in research work.

Table 3

Iron Base Alloy Powders

Alloy Powder	Powder Production and Alloying Technique	Advantages	Disadvantages	Applications
1 Premixed elemental powders	Elemental powders, ie iron and alloying elements, are mixed together, in the desired proportions, prior to compaction and sintering. Alloying occurs by diffusion during sintering.	a Good compressibility b Liquid phase sintering when using Cu c Cheaper than 2 and 4 d Flexible - changes in composition can be effected without powder supply or storage problems	a Alloying elements limited to C, Cu and Ni. Elements such as Mn, Cr have stable oxides which can't be reduced during conventional sintering b Inhomogeneous structures, especially when using Ni	Production of powder metallurgy components
2 Pre-alloyed powders	Powders produced by atomisation of liquid metal containing required alloying elements except carbon. Powder is pressed and sintered in the normal manner.	a Produced homogeneous structures b Can use elements such as Cr, Mn	a Poor compressibility due to solid solution hardening during atomisation b Expensive c Limited alloy composition range d Increased die wear	Hard facing alloys. Limited use in component production which could expand due to introduction of "sinter forging" process. Future use in powder rolling to produce stainless steel strip and other alloy steel strip.
3 Partially pre-alloyed powders	Mixture of iron powder and alloying elements or compound containing alloying elements (except carbon) are heated in a reducing atmosphere. Diffusion occurs to a limited and controlled extent. Resultant powder is then pressed and sintered and further alloying occurs during sintering.	a Good compressibility due to limited alloying b Competitive price c Can use Mn and Cr d More homogeneous structures than 1	a Limited alloy composition range	Technique patented by Hoganas.[25] Potential large scale used in component industry since good mechanical properties and good dimensional stability can be achieved. Possible use in sinter forging.
4 Master alloy	Non ferrous alloying elements, except C, are produced as a pre-alloyed powder. Process then proceeds as for 1.	a Improved compressibility compared with 2. b Can use Mn and Cr c Competitive price with 1	a Limited alloy composition range	Used for component production by some UK manufacturers. Possible future use in sinter forging.

in most of the techniques, thus ensuring consistent powder properties. However, as the industry expands, there is an increasing demand for powders with compressibilities and purities equivalent to electrolytic powder but at a lower cost. Once such powders are available, the production of larger components should become economically viable and tool wear should be considerably reduced.

In the field of medium strength alloy components, the use of pre-mixed elemental powders should continue to expand, since apart from producing specified compressibilities, manufacturers are tooled up for these powders and are familiar with their use. One of the major problems encountered when using pre-mixed powders is the control of dimensions during sintering and very careful balancing of alloying elements is required to minimise dimensional change and optimise control.

Hoganas AB, Sweden, have developed partially pre-alloyed powders which have not only been designed to produce little or no dimensional change during sintering, but also overcome the problem of poor compressibility encountered when using homogeneous pre-alloyed powders.

The use of homogeneous pre-alloyed powders is at present extremely limited in the component industry due to their poor compressibility and the fact that adequate strength can be produced using elemental powders. However, where specialised properties such as corrosion resistance, wear resistance, etc, are required, homogeneity is essential and hence the necessity for pre-alloyed powders. The major future outlet for pre-alloyed powders appears to be in the sinter forging process, but it has been found that very pure powders low in oxygen and nitrogen are essential in order to attain the required properties, and hence new atomisation techniques are being developed to meet these requirements.

2.1.4 Powder Properties

Several comprehensive reviews of metal powder properties have been published[8,9,10,11] and a brief discussion of some of the more important properties is given below:

a Apparent Density

The apparent density of a metal powder refers to the weight per unit volume of loose powder. It is usually expressed in gram/cc and is determined by allowing powder to flow into a container of known volume and weighing the powder required to fill the container. The American Metal Powders Industries Federation, MPIF, have published a series of standard tests and their particular standard regarding apparent density (MP1 4-45)[8] involves the use of apparatus shown in Figure 4, together with a standard density cup with a capacity of 25 cc.

Figure 4

Apparatus used for Determining the Apparent Density and Flow Rate of Metal Powders

For commercially available powders, apparent density is generally in the range 20-50% of the theoretical density of the solid material. Its value is controlled by other powder properties, such as:

a the density of the powder particles

b particle size, shape, size distribution and surface area

c the manner in which the particles are packed in the container

Apparent density is an important powder property since it ultimately determines the size of compacting tools and the magnitude of the press motions required to form powder compacts. It is also an important consideration in the choice and design of equipment used to transport the loose metal powder.

For the production of high strength structural components, it is generally advantageous to use powders with a high apparent density, eg electrolytic (Table 4) that produce high green densities. This concept can be expressed in terms of the 'compression ratio', viz final density/apparent density, and in practice a low compression ratio is desirable since it:

a reduces the size of the die cavity and tooling

b reduces press motions

c permits faster filling of the die and hence higher production rates

Table 4

Typical Properties of Various Iron Powders

Nature		Iron Powder		
		Reduced	Atomised	Electrolytic
Sieve analysis % mesh	-70	-	-	-
	-70 + 100	1	11	13
	-100 + 140	20	25	28
	-140 + 200	26	19	10
	-200 + 230	10	15	10
	-230 + 325	15	16	24
	-325	28	14	15
Apparent density (gm/cc)		2.3	2.5	3.2
Flow rate (sec/50 gm)		33	25	25
Compressibility /gm/cc at 25 tsi (386 MN/m^2)7		6.2	6.2	6.7

b Flowability (Flow Rate)

The flowability or flow rate of a metal powder is usually quoted as the time taken, in seconds, for a given mass of powder to flow through a standard orifice. The MPIF standard relating to flow rate is MP 13-45[8] and the equipment used is identical to that used for determining apparent density. A standard weight of 50 gm of dry powder, weighed to the nearest .1 gm, is recommended.

The significant factor controlling flowability is particle size, the smaller the particles the slower the flow rate. Metal powder must have adequate flowability to ensure uniform filling of intricate die configurations and to maintain a uniform density distribution through the component.

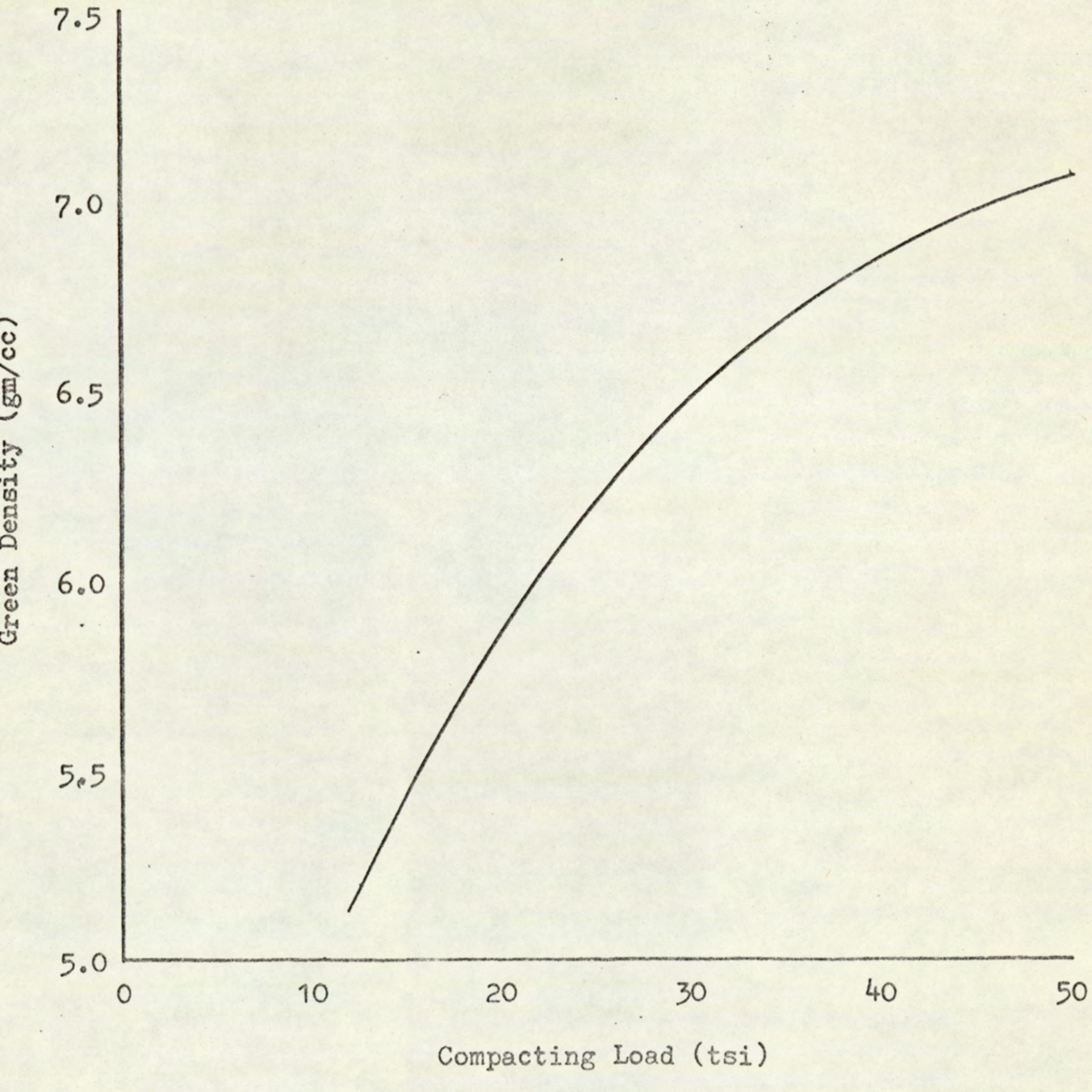
d Compressibility

The compressibility of a metal powder is defined as the amount by which powder will densify on the application of pressure. One technique of measuring compressibility is to determine the green density produced at a given compacting load (BS 3029) but a more useful method is to produce a green density-compacting load curve (Figure 5). A number of factors control compressibility, eg particle size, shape, size distribution, chemical composition, plasticity, presence of surface films, and in practice a high compressibility is desirable since:

Figure 5

Green Density versus Compacting Load

Hoganas NC 100 24 Iron Powder[8]



a lower compacting loads are required to achieve a given green density, thus reducing power consumption and in some cases even reducing the size of compacting press required

b die life can be improved due to the use of lower compacting and stripping loads

c larger components can be produced on a given press

d <u>Green Strength</u>

Green strength, which is the strength of the pressed component, is usually assessed by a transverse rupture test (MP 1 13-51 T)[8] and is directly related to the as-pressed density of the component. A high green strength is desirable to aid ejection of complex components containing thin sections and to minimise damage of components during handling prior to sintering.

e <u>Dimensional Change During Sintering</u>

This can be assessed by comparing the dimensions of a standard test piece before and after sintering (MP 1 13-51 T)[8]. However, this will only give a general indication of dimensional change since the shape and size of the part, as well as the composition, density and sintering conditions used, have a major effect on dimensional change. Hence, a better indication of dimensional change during sintering can be established by using the actual component instead of a standard test piece.

Dimensional changes during sintering can be allowed for in the design of the compacting tools but close control of the size of the sintered component is only possible if such changes are small, viz ±1%. In order to minimise dimensional change during sintering close control of all sintering variables, viz sintering time, temperature and atmosphere composition is essential, together with close control of alloy composition and a minimal density variation within the as pressed components.

f Consistency

Although consistency is not a specific powder property when compared with those discussed above, it is of paramount importance to the manufacturers of powder metallurgy parts. Variations in the consistency of powder properties can lead to variations in mechanical and physical properties and dimensional accuracy, thus resulting in extensive rejection of the finished components. Detailed investigations have been carried out in America, Germany and Britain to establish standard metal powder testing procedures. The American and German standards are compared in the Hoganas Handbook[8] and the relevant British Standards are BS 3209 and 2590.

From the above discussion, it can be seen that the powder properties reviewed are themselves controlled to a large extent by the basic characteristics of the powder, ie particle shzpe, size, size sistribution, etc. These in turn are dependent on the powder production process used (Table 4) and any subsequent mechanical treatment, eg grinding, crushing, etc, applied to the powder. Hence, good quality control during powder manufacture is essential and detailed powder property specifications are required, together with stringent testing procedures.

2.2 Powder Blending

The main object of the mixing or blending process is to:

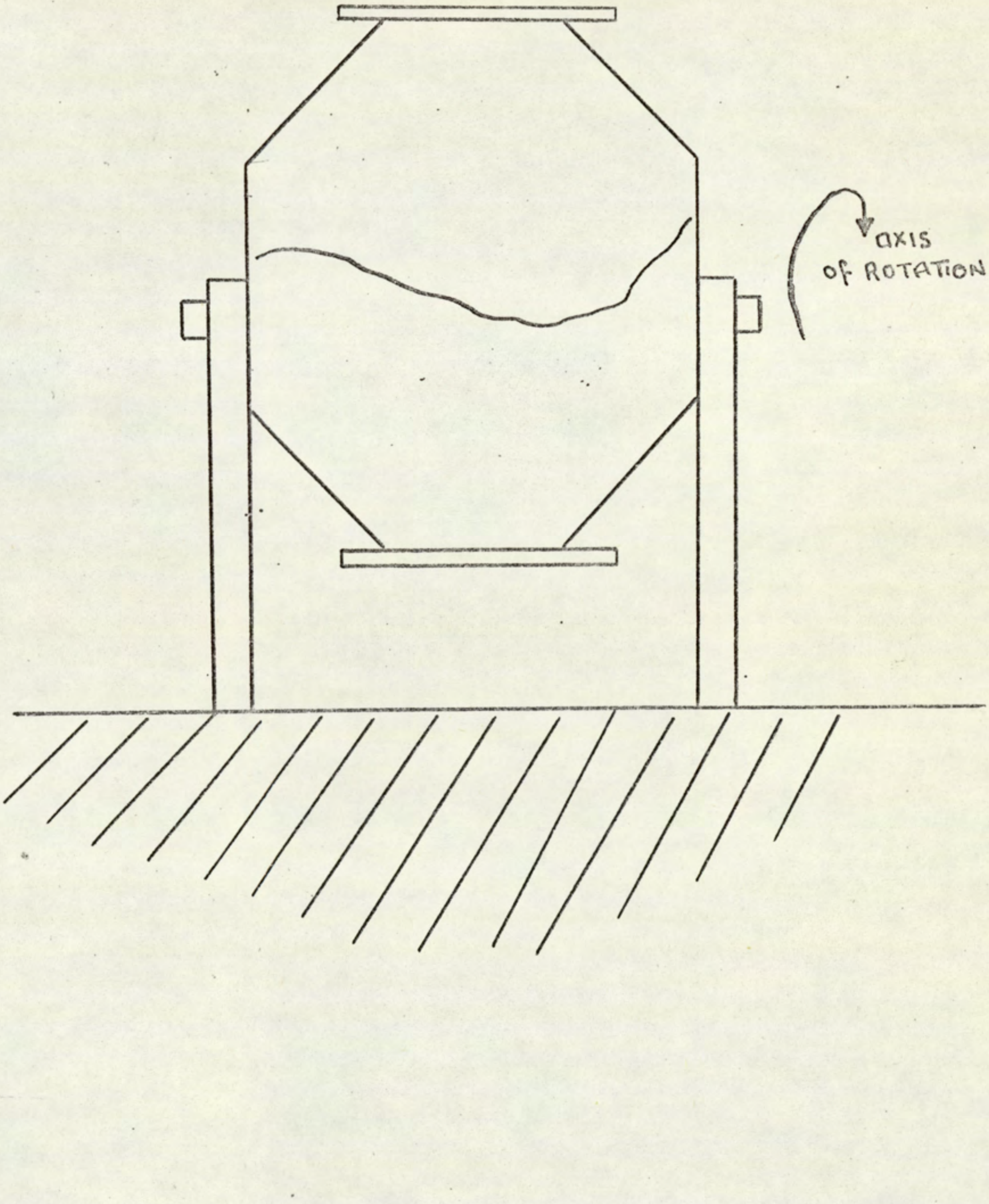
a re-mix powders which may have segregated after transportation

b produce a homogeneous mixture of metal powder and a suitable lubricant

c produce a homogeneous mixture of elemental powders used in the production of alloy components

The most widely used mixers in the powder metallurgy industry are double cone mixers, Figure 6. The British Metal Sintering Association[12] (BMSA) has published a critical review of mixing equipment and recent developments in the mixing process and equipment have been reviewed by Scarlett.[13]

FIG 6 DIAGRAM OF A DOUBLE CONE BLENDER



The determination of optimum mixing time requires careful investigation since simply extending it in the hope that this will increase homogeneity can result in:

i work hardening of the particles, thus decreasing compressibility

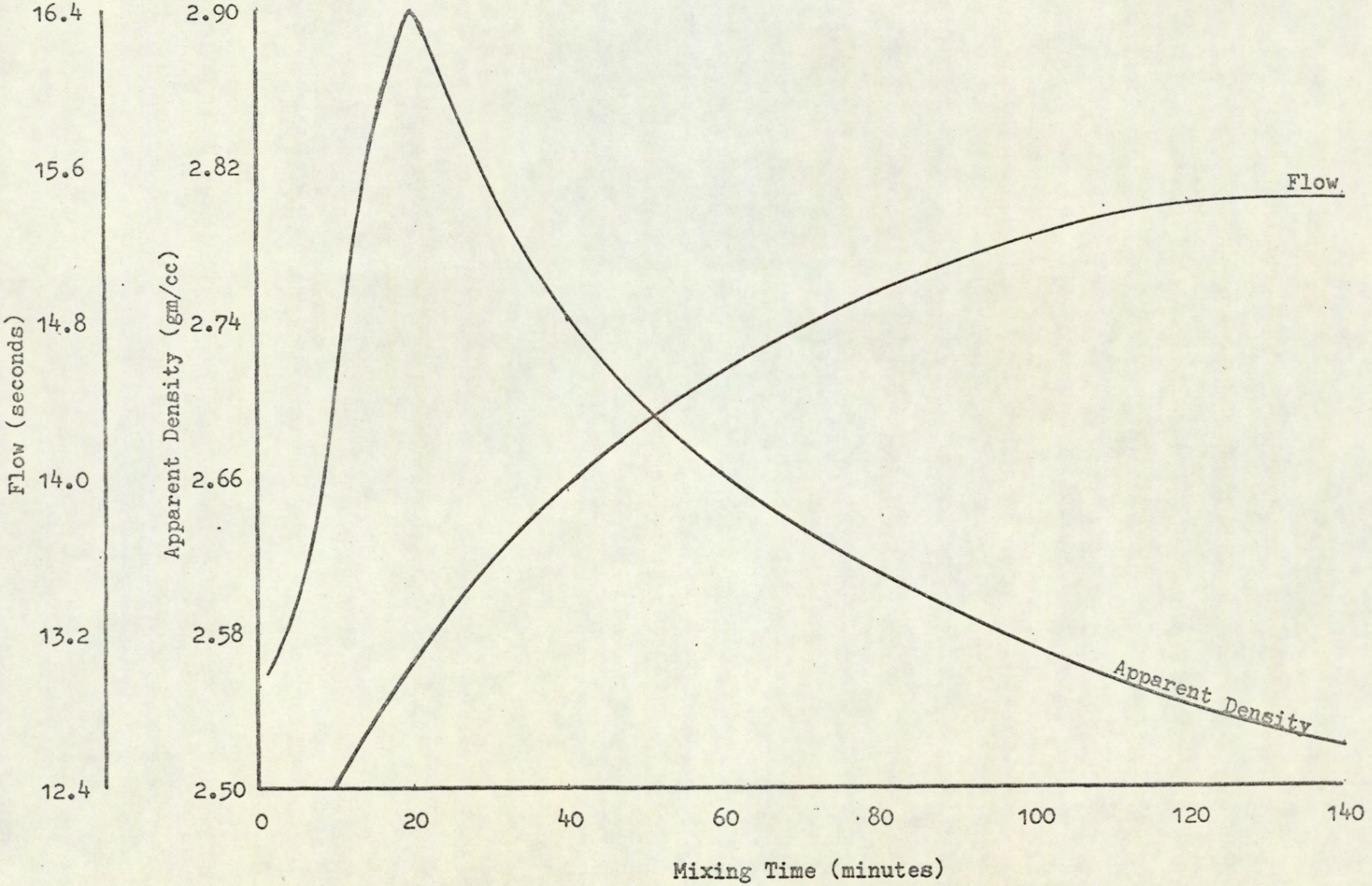
ii alteration of particle shape, size and size distribution resulting in changes in bulk properties such as flow rate and apparent density, Figure 7

iii admixed lubricants becoming soft and gummy, resulting in agglomeration of powder particles

At the present time, the only satisfactory technique for determining when a suitable mix has been obtained is to measure properties such as flow rate and apparent density after mixing and to assess the performance of the powder mix during pressing and sintering.

Mixing is clearly a very important stage, especially in the production of alloy components from elemental powders where homogeneity is a significant consideration. However, little is known about the fundamentals of the process and its importance is often overlooked until problems arise at a later stage in production.

Effect of Mixing Time on the Apparent Density and Flow of an Iron 3% Copper Mixture[15]



2.3 Powder Compaction

A variety of techniques can be used to compact metal powders and these are outlined in Figure 3. However, many of these processes have a very limited application in the component industry and the majority of powder metallurgy parts are produced by cold die compaction. Die compaction readily lends itself to automation and mass production and is often referred to as "the conventional technique".

2.3.1 Die-compaction techniques

The process of die compaction is discontinuous and allows pressure to be applied in a vertical direction through punches to the powder mass which is contained in a rigid die. To overcome the magnitude of density variations in thicker parts, pressure is applied to opposite faces of the powder bed. This is usually achieved using a fixed die and applying pressure from both ends, ie via top and bottom punches, or by using a die supported on springs so that when pressure is applied to the top punch the die also moves downwards, ie "floating die compaction". Compacting pressures used in industry vary between approximately 20 and 50 tsi (309-722 MN/m^2), depending upon the density and strength level required. A variety of component shapes can be produced using various compaction techniques and these are described in detail in several publications.[8,9,14] A brief summary of a selection of die compacting techniques and their applications is given in Table 5.

Table 5

Die Compaction Techniques

Compaction Technique	Applications
Single action - pressure applied from one end only	Thin single level components
Double action - pressure applied at both ends	Thick single level components
Double action floating die - pressure applied at one end and die table moves down simulating double action	Thick single level components
Multiple motion double action - pressure applied from both ends - system incorporates two or more moveable lower and upper punches	A variety of multi-level components

In Continental Europe, the majority of the components are produced using simple presses and a floating die system with various punch, die and core-rod movements built into a complex tool set. The parts are ejected by continuation of the downward motion used to provide the compacting load until the part is fully withdrawn from the die. This withdrawal system is not widely used in the USA, the majority of American components being produced on complex double action presses where parts are ejected by independent bottom punch motions.

A number of advantages are claimed[8] for the withdrawal type of tooling and these are summarised below:

a structural parts having three or more different levels in the pressing direction can be compacted to a high density

b superior dimensional accuracy in the pressing direction can be obtained using simple mechanical or hydraulic presses than can be obtained with similar double acting tools or more complex presses

c more rigid and shorter punches can be used

No detailed information concerning the relative application of these tooling systems in the UK component industry is available but it appears that the withdrawal system is generally favoured for producing large multi-level parts requiring the use of large loading capacity presses.

2.3.2 Densification, bonding, friction and lubrication

The processes involved in densification and bonding during die compaction are described in detail in several publications.[8,9,10,15] The various stages of densification during die compaction can be summarised under the following general headings:

a Densification during filling

b Densification during bulk powder movement

c Densification due to deformation and fracture

Stages a and b occur during die filling and the initial application of the load. Interparticle friction is overcome under light loads and the particles re-arrange themselves to reduce void space until particle locking occurs. During stage c sufficient pressure is developed at interparticle contacts to break through very thin surface oxide films and hence produce cold welds. These welds, together with some mechanical interlocking, account for the strength of the as pressed component, ie its green strength.

Friction forces between the particles and, in particular, between the mass of the powder and the die wall, inhibit movement of particles and hence the transmission of pressure through the powder mass. This, in turn, gives rise to density variations within the compact, Figure 8, which, if large, can produce startling shape changes during sintering, Figure 9. Such density variations can be reduced by using a lubricant, and two lubrication techniques are available:

i Admixed lubrication which involves premixing the iron powder with the desired lubricant prior to compaction

ii Die-wall lubrication which involves the introduction of a lubricant on to the die wall before the powder is poured in

Several investigators[15] have shown that die-wall lubrication is superior in minimising the compacting pressure required to produce a specified green density. However, at the present time, the technique cannot be applied to modern high output presses and hence admixed lubrication is widely employed. The most commonly used lubricants are paraffin wax, stearic acid and various metallic stearates and they are usually in the proportion of ~1% by weight.

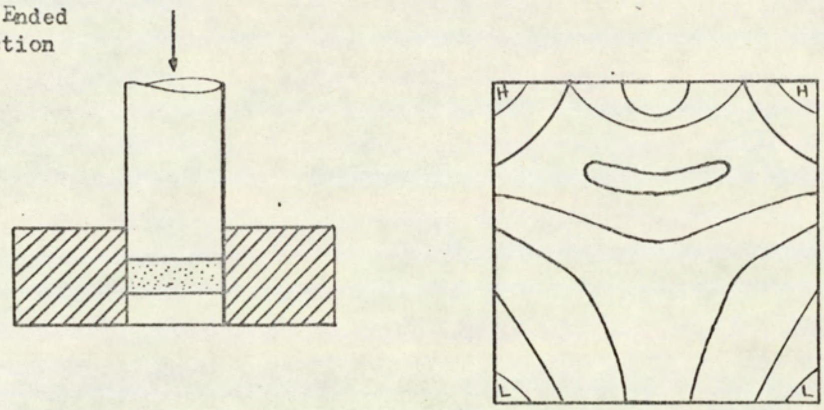
2.3.3 Elastic recovery

After the release of the compacting load, elastic stresses developed in the compacting direction are, to some extent, relieved but those normal to the pressing direction are not since the component is constrained in the die. During the ejection cycle, a stage is reached where part of the component is free from the

Figure 8

Density Distributions Obtained by Single Ended and Double Ended Die Compaction[10]

a Single Ended Compaction



H - High density

L - Low density

b Double Ended Compaction

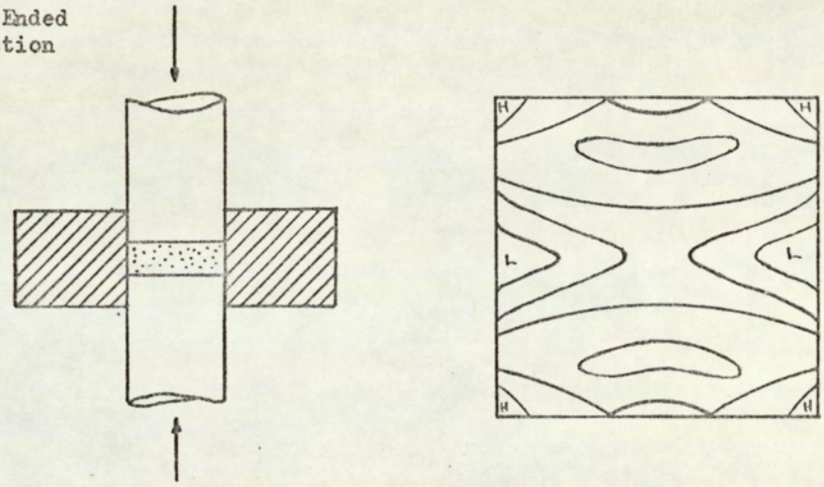
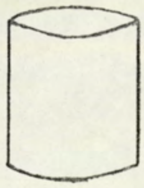


Figure 9

Changes in Shape on Sintering of a Cylinder with Density Variations in the Original Green Compact[9]



Original Green Cylinder

Cylinder After Single Ended Compaction and Sintering

Cylinder After Double Ended Compaction and Sintering die and part is still constrained. The free portion expands slightly, due to the release of elastic stress, and if the strength of the powder is insufficient to withstand this stress release, horizontal laminations appear.

This type of defect can be reduced by a slight radius on the upper rim of the die and/or tapering the die. However, such precautions are useless unless the component has a sufficiently high green strength.

2.4 Sintering

2.4.1 Objects of sintering

a Consolidation of bonds and densification

The main object of sintering is to consolidate the interparticle bonds produced during pressing and thus improve the mechanical properties to a sufficient level to meet the service requirements of the part. This is achieved by heating the components for a specified time at a temperature below the melting point of at least one major constituent. In the case of iron and iron base components, sintering can be carried out at temperatures up to 1300°C, but in practice, adequate strength can usually be produced by sintering at 1120-1150°C for about 30-45 minutes. The reason for using this lower sintering temperature and a short sintering time are basically economic and this temperature (1120-1150°C) is the limit of most continuous mesh belt furnaces. Higher temperatures necessitate the use of more complex and more expensive furnaces, viz walking beam (Section 2.4.5).

b Formation of alloys

When a mixture of elemental powders is used, alloying also occurs during sintering and in certain cases, eg iron-copper, a liquid phase is produced, thus accelerating alloying and densification. However, using commercial sintering conditions, the extent of the interdiffusion is very limited except for elements with high diffusion rates such as carbon in iron. When producing iron base alloys containing varying amounts of carbon, a protective atmosphere whose composition, viz carbon potential, can be adjusted to maintain specified levels of carbon in the components, is essential.

2.4.2 The sintering process

A number of distinct stages which occur during the sintering process have been identified and these are described in detail in several publications.[9,10,11,16,17] A brief summary of some of the more important stages is given below:

a Removal of lubricants (dewaxing)

It has already been pointed out that admixed lubrication is widely used in the compaction of metal powders and before sintering can occur, this lubricant must be removed. Dewaxing is achieved by slowly heating the components to approximately 400-500°C, thus volatising the lubricant. If the components are heated too rapidly during dewaxing, sudden volatilisation occurs, resulting in severe blistering of the components. Dewaxing can be carried out either in a batch furnace prior to sintering or in a lower temperature zone of the sintering furnace, referred to as the dewaxing zone. The latter is generally employed in large continuous furnaces but the presence of lubricant vapour presents some major problems since it may:

i attack exposed heating elements

ii react with complex protective atmospheres, eg endogas, thus presenting problems in controlling atmosphere composition

iii attack welded joints in muffle furnaces

In order to minimise such effects, entry ends of furnaces are usually raised to produce a natural draught from exit to entry, thus sweeping out most of the lubricant vapour.

b Initial increase in particle bonding

As the temperature increases, bonding occurs at areas of intimate contact on powder particles and the small cold welded areas produced during compaction, slowly grow. For these processes to occur, surface oxide films must be removed, and hence the use of a reducing atmosphere in the manufacture of iron base components. Local diffusion processes are induced at these small welded contacts since they are areas of high energy resulting from cold working during compaction.

This stage usually occurs in commercial sintering furnaces as the components are being heated to a specific sintering temperature and since relatively small amounts of material are involved no changes in dimensions or significant increases in strength occur. However, this initial production of necks results in a rapid increase in electrical conductivity, Figure 10, and represents an important change in the material since this bonding imparts a high degree of coherency to the material.

c <u>Pore rounding and rapid neck growth</u>

As the sintering temperature is further increased, the rate of neck growth increases rapidly, thus increasing mechanical strength, Figure 10. The pores gradually change to a regular shape and after prolonged sintering become spherical. There is a general reduction in pore size and to some extent a decrease in their number. However, the majority of the porosity remains interconnected, thus providing escape routes for entrapped gases and gases produced by metal/protective atmosphere reaction. As the

Figure 10

Effect of Sintering Temperature on the Properties of A Typical Sintered Body

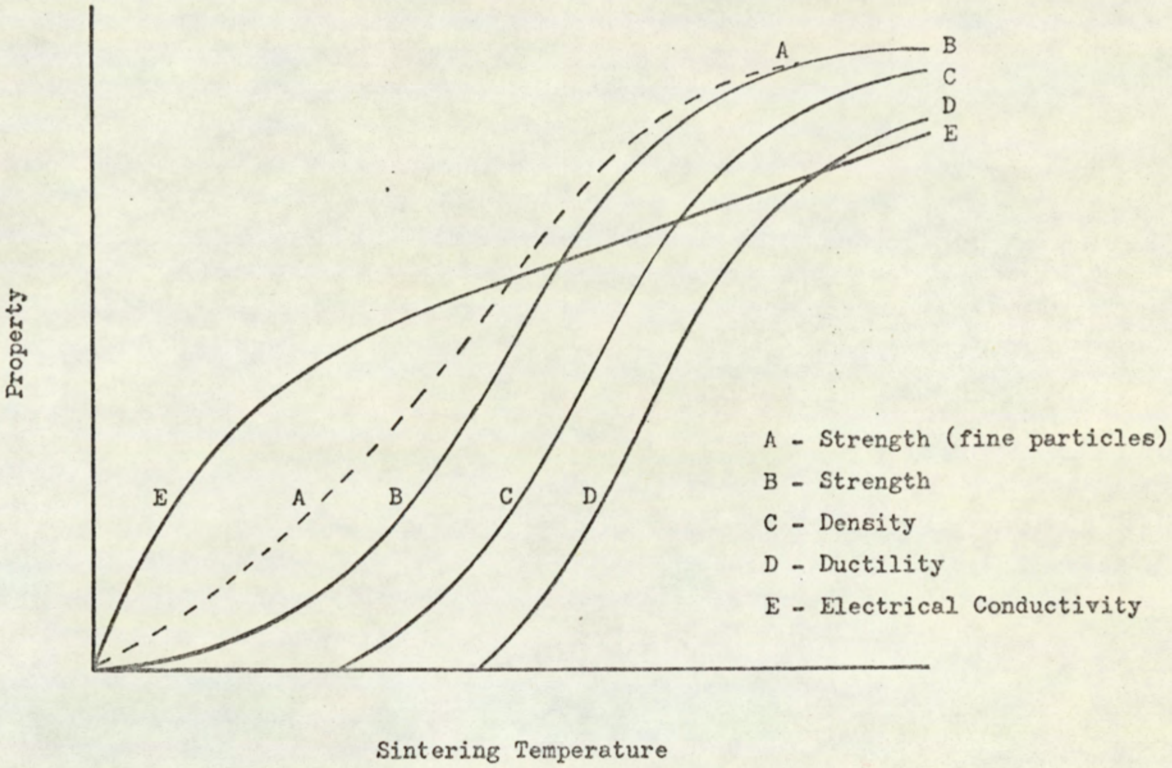
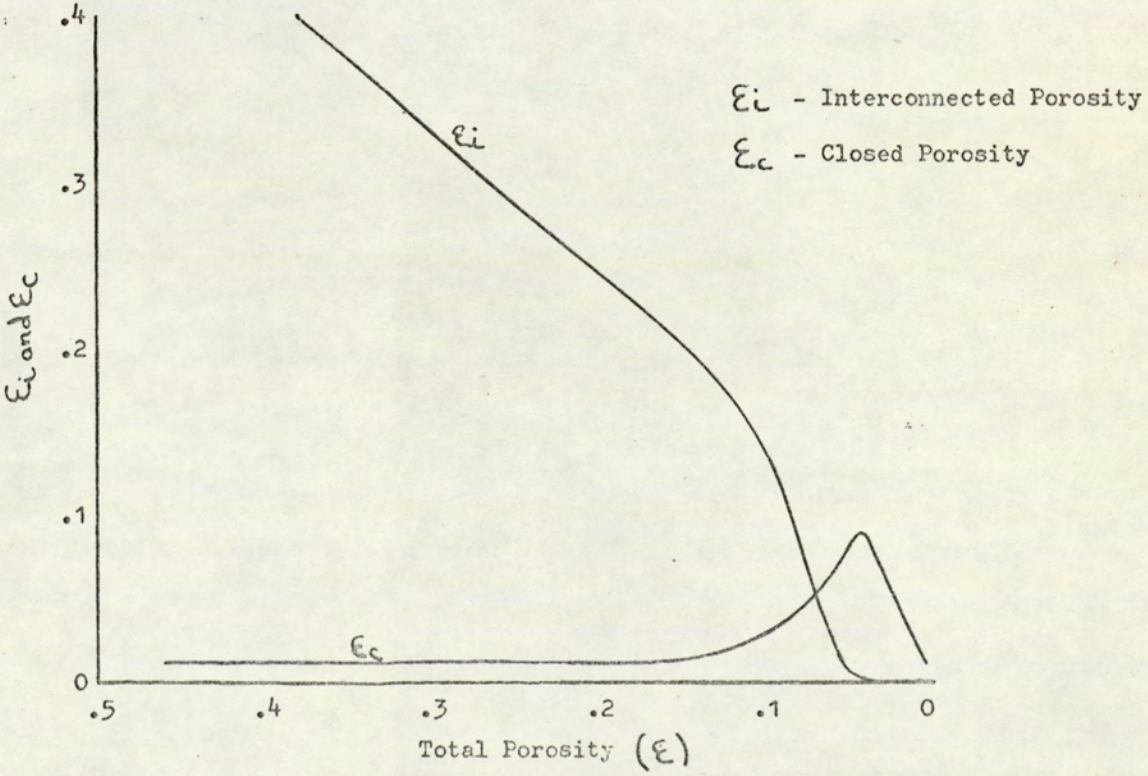


Figure 11

Variation of Interconnected and Closed Porosity With Total Porosity for Copper Compacts[17]



density approaches 90% theoretical the relative proportion of closed pores increases rapidly, leading to the final stage of sintering, Figure 11.

Although a reduction in dimensions, ie shrinkage, is usually associated with a reduction in pore size, large density changes are not generally produced since weight loss due to the reduction of oxides also occurs.

As far as practical sintering is concerned, this is the most important stage, since alloying of premixed alloying elements occurs, together with rapid increases in mechanical strength. In practice, the majority of commercial sintering operations are terminated at the end of this stage.

From this brief discussion, it can be seen that sintering involves a gradual change in the shape and the size of pores. The driving force for this change is the excess internal energy of the system above equilibrium requirements for a specified temperature. Energies contributing to this driving force include: excess grain boundary energy, excess lattice energy and the surface energy of the pores. Small irregular shaped particles have a large excess energy, ie driving force, resulting in a rapid increase in strength with increasing sintering temperature, Figure 10. However, extremely fine powders have a limited application in the production of components since they are expensive to produce and have poor flowability.

The rate of increase in mechanical properties gradually falls off with increasing temperature, ie follows an exponential function, and this is a result of a decrease in internal energy and hence driving force as the sintering process proceeds. The elimination of all porosity is highly desirable but is very rarely attempted since extremely high temperatures and long sintering times are required.

2.4.3 Material transport mechanisms and quantitative analyses

The process of sintering involves a gradual change in the shape and size of pores and a general reduction in their size and number. This requires the transport of material and several mechanisms have been proposed:

a Diffusion mechanisms:

    a Surface diffusion
    b Volume (bulk or lattice) diffusion
    c Grain boundary diffusion

b Plastic flow mechanisms which result from the presence of surface tension forces and the increased plasticity of metals at elevated temperatures

c Condensation and evaporation. Although this has been suggested as a material transport mechanism, it is unlikely to be of significant importance in the production of ferrous base materials where all the elements have relatively low vapour pressures at normal sintering temperatures.

Many attempts have been made to predict and demonstrate the influence of the above mechanisms on the various stages of sintering and many differing views are held. Several quantitative theories have been produced and an extremely comprehensive review of these has been published by Thummler and Thommas.[17] The majority of this theoretical work has been carried out using model idealised systems, ie spheres, wires, etc, and a large number of practical aspects, such as particle size and shape variation, deformation during compaction, shape ~~and~~ size and orientation of pores, surface contamination, etc, have been ignored. As a result of this, such theories, whilst providing a deeper insight into the process of sintering, do not have any specific practical applications.

2.4.4 Control of dimensions during sintering

A major consideration in the production of sintered components is the control of dimensions and one of the advantages of the powder metallurgy process is the production of components within close dimensional tolerances, ±.0005 in/in (.01 mm/mm). It has been pointed out that a shrinkage is usually associated with sintering, ie pore shrinkage and elimination, but this can only be generally applied to single component systems. The introduction of alloying elements, when producing components from pre-mixed elemental powders, can have a significant effect on dimensional change and the general effects of additions of some of the more common alloying elements to iron are listed below:

- a Carbon - reduces shrinkage
- b Copper - promotes expansion
- c Nickel - promotes shrinkage

By careful control and "balancing" of alloying elements, close control of dimensions is possible.

A second major consideration in the control of dimensions is the as-pressed density. The shrinkage produced in the sintering of pure iron is inversely proportional to the as-pressed density since there is a reduction in the sintering driving force as density increases. Hence, density variations must be avoided, especially in multi-height components and in practice are usually limited to approximately ±1% in the as-pressed component.

10%

2.4.5 Sintering furnaces

a Basic requirements

The basic requirements of a sintering furnace are that it should heat the compact at a controlled rate to a specified temperature which is maintained for a specified time. A protective atmosphere,

Table 6

Comparison of Continuous and Batch Sintering Furnaces

Continuous Furnaces			Batch Furnaces
Mesh Belt	Roller Hearth	Pusher (Walking beam)	
The "green" components are either loaded directly onto the mesh belt or loaded into trays which are then placed on the belt. The endless mesh belt is usually made of a heat resisting alloy, eg 80% Ni, 20% Cr, and its speed can be closely controlled, thus regulating the speed at which compacts pass through the various zones of the furnace.	Green components are loaded in trays which are conveyed through the furnace by a series of driven rolls. Loading capacity is 4-7 times greater than mesh belt but the system is less reliable and being more complex requires more maintenance, etc.	Components are loaded into ceramic or heat resistant trays and are mechanically pushed through the furnace. This system is less reliable and more complex than mesh belts.	Furnace charge is staionary and hence there are no problems in conveying the furnace charge. Close control of heating and cooling rates is difficult and this introduces problems in the close control of dimensions.
Maximum operating temperature - 1150°C Maximum loading capacity - 10 lb/ft^2	Maximum operating temperature - approx 1150°C	Maximum operating temperature - approx 2000°C	Maximum operating temperature - approx 3000°C
Widely used for sintering iron, iron-carbon, iron copper and iron copper carbon alloys.	Applications are similar to those of mesh belt furnace.	Applications are mainly high temperature sintering of Fe-Ni and Fe-Ni-C alloys.	Furnace through put is slow compared with continuous furnaces and hence its limited use in the manufacture of sintered components. The main applications in the powder metallurgy industry are in the sintering of large parts, eg brake drums.

generally reducing, is also required for the production of iron base components. In modern plants, sophisticated instrumentation is required in order to achieve the close control of sintering time, temperature and atmosphere composition necessary for the manufacture of components within close dimensional specifications.

b Comparison of continuous and batch furnaces

Several types of sintering furnaces are available and the construction and factors involving the final choice for specific applications have been reviewed in several publications.[14,16] A brief comparison of continuous and batch furnaces is given in Table 6.

2.4.6 Sintering Atmospheres

A wide variety of protective atmospheres can be used for sintering iron and iron base components and the costs and applications of a number of these are outlined in Table 7.

The most widely used atmospheres in the commercial sintering of iron and iron base materials are:

a Endothermic Gas
b Exothermic Gas
c Cracked Ammonia

a Endothermic Gas

The products of combustion resulting from the burning of a hydrocarbon gas, eg natural gas, propane, and air in such a proportion that external heat is required to maintain the combustion reaction are known as endothermic gas. In the endothermic range, Figure 12, there is a significant increase in dew-point with increasing air/natural gas ratio, which is useful in controlling the carbon potential of the atmosphere since the higher the water vapour and carbon dioxide content the lower the carbon potential. This control of carbon potential is vital in sintering iron base components containing varying amounts of carbon and accounts for the wide use of endothermic gas in sintering such alloys.

b Exothermic Gas

The products of combustion resulting from the burning of a hydrocarbon gas and air in such proportions that heat is produced are known as exothermic gas. The richest exothermic gas is produced at air/natural gas ratios of $\sim$6:1, Figure 12, and its composition is such that it is generally reducing and can hence be used for sintering iron and iron copper components. However, its carbon dioxide content makes it unsuitable for sintering iron base components containing carbon.

Table 7

Applications and Cost of Various Protective Furnace Atmospheres

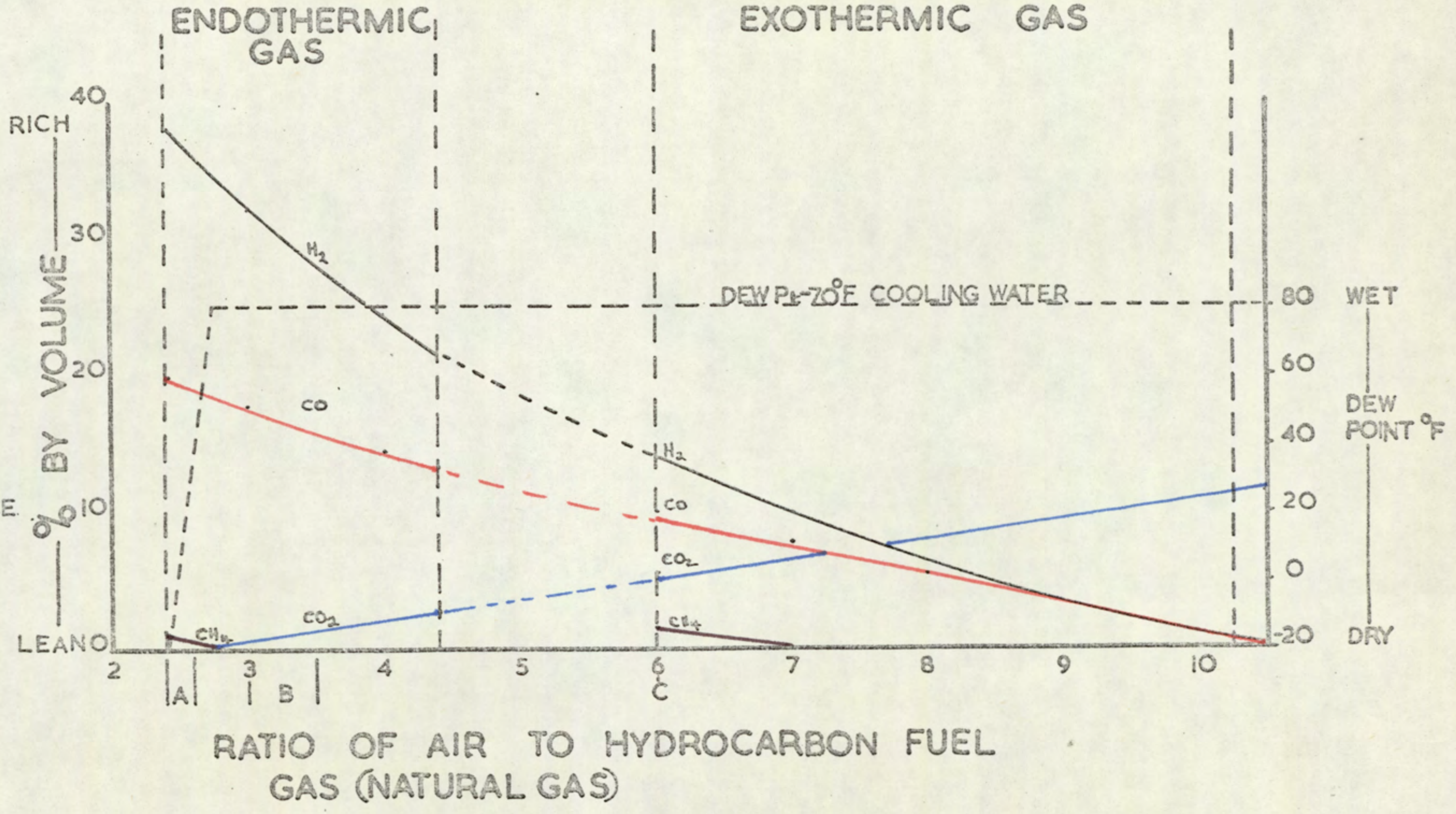
(Powder Metallurgy Equipment Manual)[14]

Atmosphere	Sintering Applications						Other Applications					Cost* £/1000 cuft
							Carburising	Heat Treatment		Infiltration		
	Fe	Fe-C	Fe-Cu-C	Fe-Cu	Steels C and Alloy	Others (Refractory metals etc)	Fe	Fe-C	Fe-Cu	Fe	Fe-C	
HYDROGEN												
Unpurified	+			+						+		0.30 - 5.00
HYDROGEN-NITROGEN MIXTURES												
1 Dissociated Ammonia Dry	+			+		Nickel Steels				+		0.60 - 1.80
2 Burned Dissociated Ammonia Rich Saturated	+			+						+		0.40 - 1.30
3 Direct Catalytic Conversion of Ammonia and Air. Rich	+			+						+		0.35 - 1.30
REFORMED HYDROCARBON GASES												
1 Exothermic Rich	+			+						+		0.04
2 Purified Exothermic												
a Rich	+	+	+	+	+			+	+		+	0.06
b Medium Rich	+	+	+	+	+		+∓	+	+	+	+	0.06
3 Endothermic Gas												
a Rich Dry		+	+		+		+	+	+		+	0.08
b Rich Fairly Dry		+	+		+		+∓	+	+	+	+	0.08
c Medium Rich Saturated	+			+						+		0.07
d Lean Saturated	+			+						+		0.07
ARGON	+					+						30.00 - 35.00
HELIUM	+					+						35.00 - 42.00
VACUUM						+						-

* Costs only include materials consumed, eg power, gas, water, etc

∓ Methane added

FIG.12 COMPOSITION OF ENDOTHERMIC & EXOTHERMIC GAS



A - Fe/C
Fe/C/Cu
LOW ALLOY
STEELS

B - Fe
Fe/Cu

C - Fe
Fe/Cu

N.B. NITROGEN (NOT SHOWN) IS BALANCE OF GAS COMPOSITION

c Cracked Ammonia

Cracked ammonia contains 75% hydrogen and 25% nitrogen and it is produced by dissociating ammonia over a heated catalyst (sponge iron at 520-550°C or nickel impregnated pellets). Its applications tend to overlap those of hydrogen and it is mainly used in the ferrous powder metallurgy industry for sintering iron nickel and iron nickel carbon alloys at temperatures of 1250-1350°C. Although some decarburisation problems arise when sintering alloys containing carbon, these can be reduced by refrigerating and/or chemically drying the gas before introduction into the sintering furnace.

3 THE MECHANICAL PROPERTIES OF IRON BASE POWDER METALLURGY MATERIALS

Tensile strength and elongation of sintered iron base alloys are widely reported in published literature and are used extensively in mechanical property specifications in the commercial production of sintered iron base components. Other mechanical properties are not extensively reviewed in published literature, but some limited fatigue data for a number of iron base alloys is reported.

The range of mechanical properties, viz tensile strength, percentage elongation and fatigue strength, reported in the literature reviewed are in histogram form Figures 13, 14, 15. respectively. Three main factors control the mechanical properties of iron base powder metallurgy components, viz

Density
Alloy additions
Heat treatment of alloy components

Separate sections of this review deal with each factor.

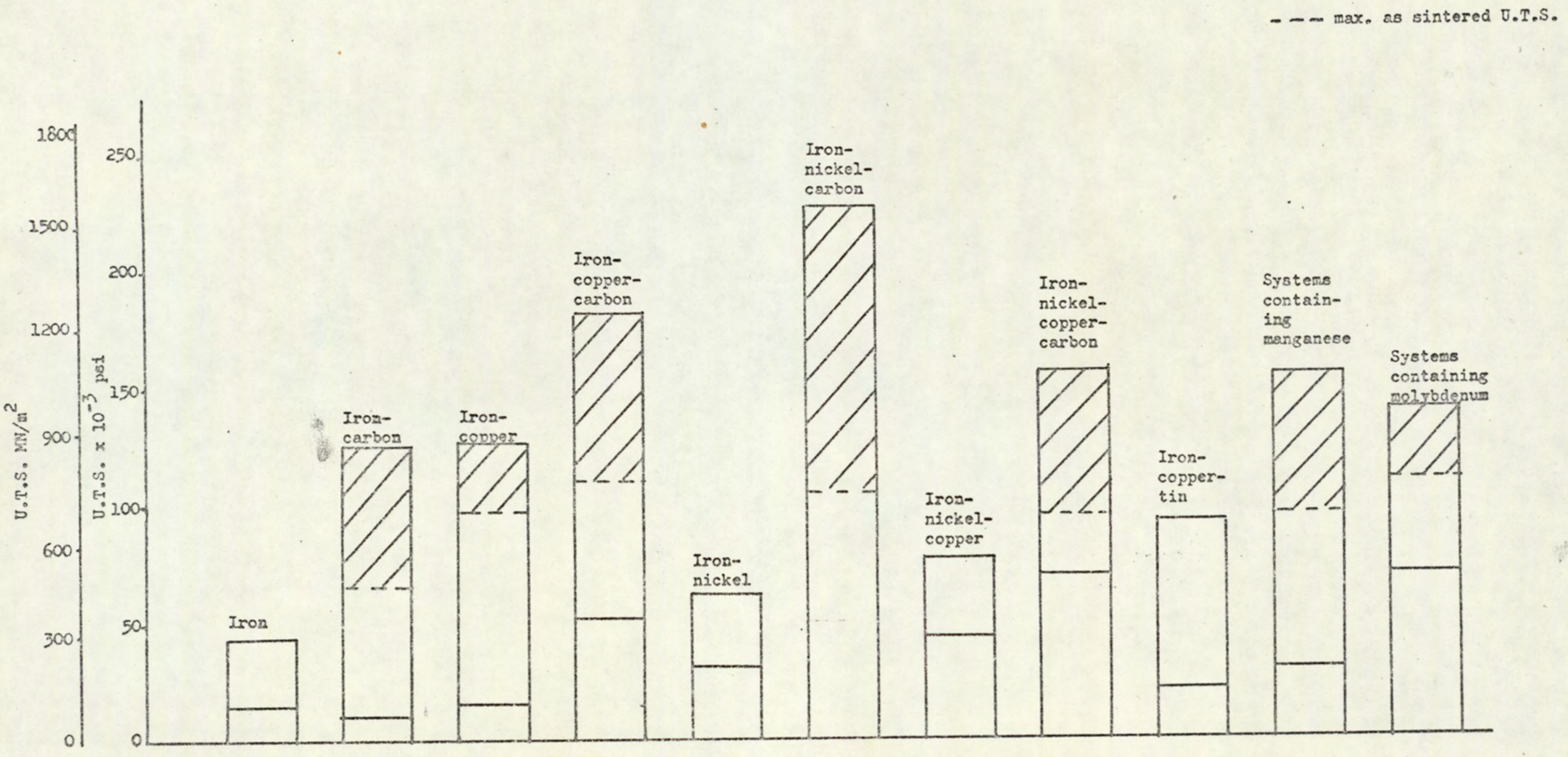
3.1 Density

3.1.1 Effect of Density on Mechanical Properties

a Strength

The strength of any sintered component depends upon the strength of the individual particles and also on the strength, size and number of inter-particle bonds, the former is governed by alloying and the latter by the chemistry and density of the system. Figure 16

Fig.13 Tensile strength of sintered iron and iron base alloys



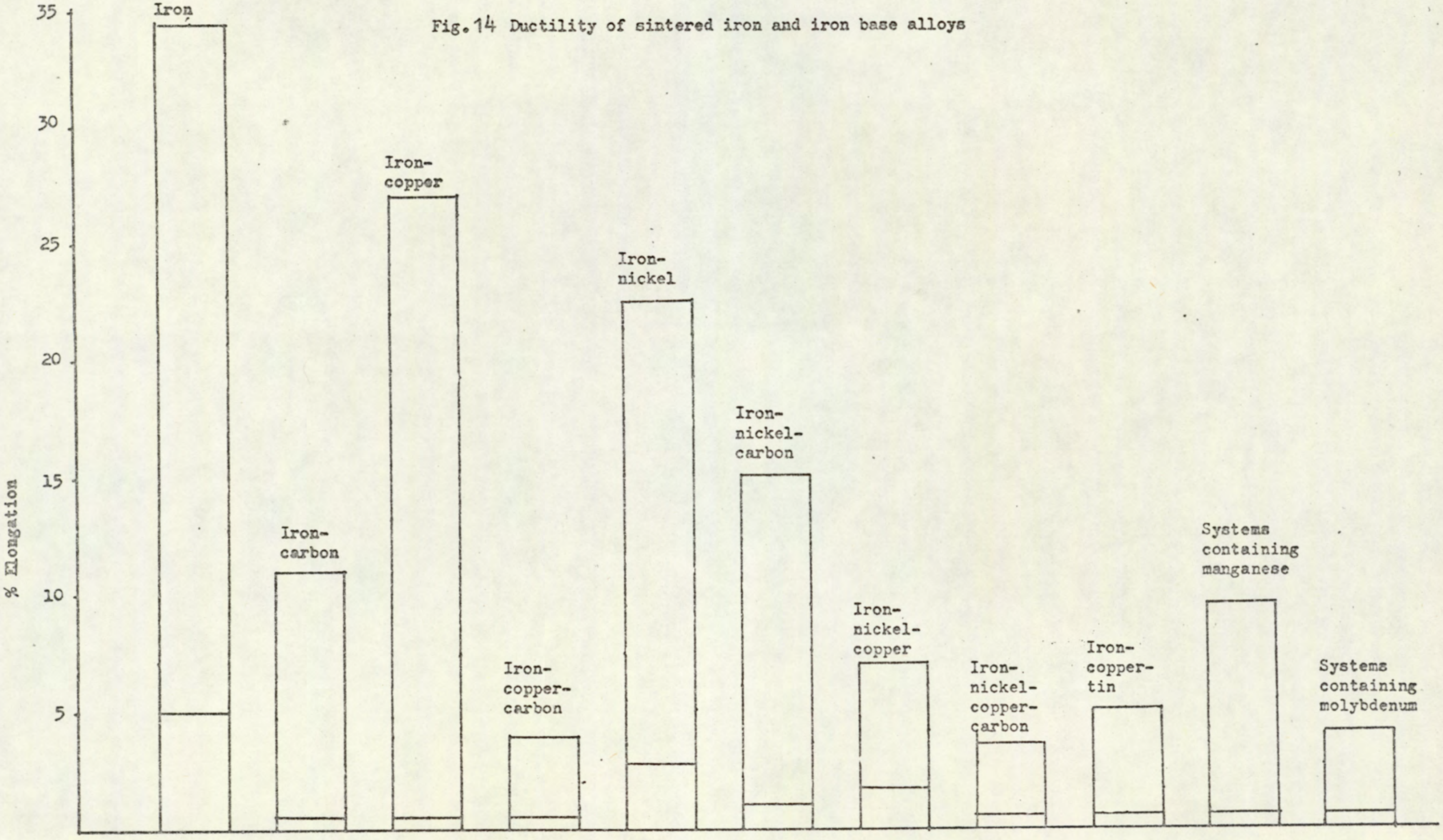


Fig. 14 Ductility of sintered iron and iron base alloys

Fig. 15 Fatigue strength of sintered iron and iron base alloys

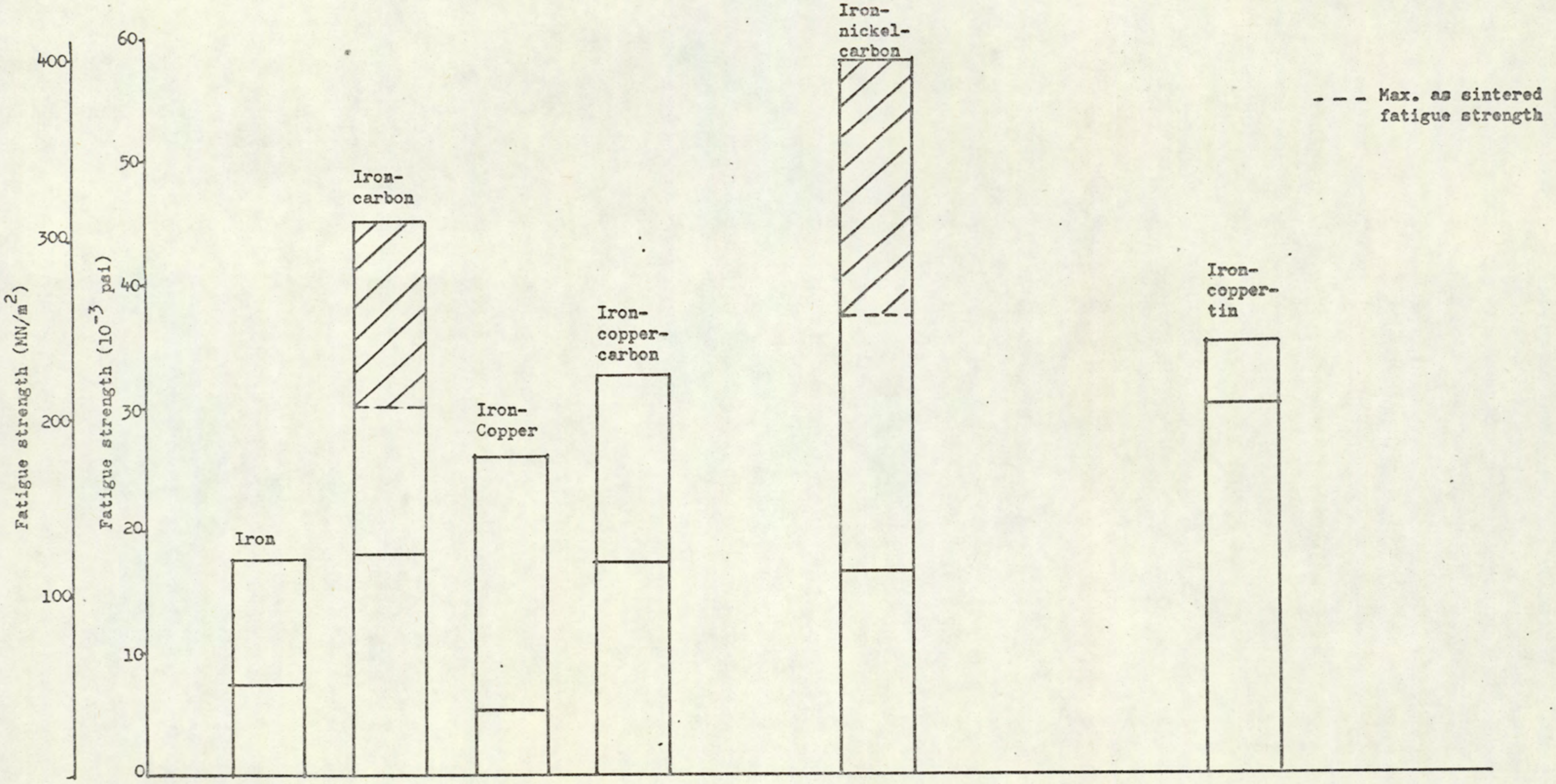
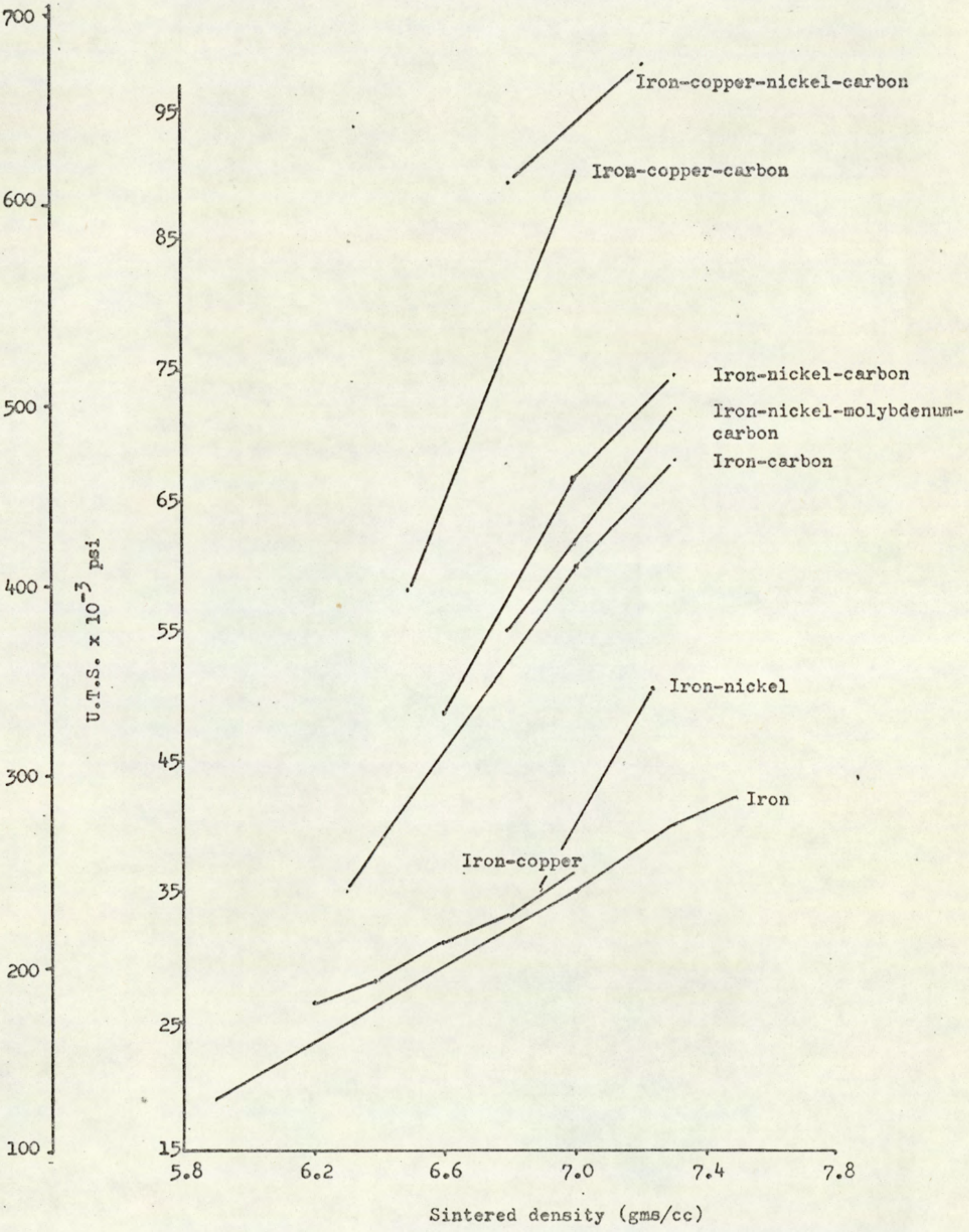


Fig. 16 Effect of density on as sintered strength of iron and iron base alloys



shows how the strength of iron and iron base powder metallurgy components increases with increasing density.

b Ductility

The effect of density on the ductility of iron and iron base powder metallurgy components is shown in Figure 17. The ductility of pure iron is significantly improved by increasing density, whereas that of alloy steel components is only slightly improved by increased density.

c Fatigue Strength

The limited information available concerning the fatigue properties of sintered iron and iron base alloys indicates that fatigue strength increases with increasing density (Figure 18).

3.1.2 Techniques Available for Increasing Density

a Improvements in Conventional Pressing Techniques

i Development of Larger Loading Capacity Presses

The green density of as-pressed components is governed by the load applied, the pressing characteristics of the powder and the lubrication system used. Hence, as the loading capacity of presses increases, the size and green density within other limits also increases. At the present time, presses of 100-400 tons are readily available and 1,500 ton presses are

Fig 17 Effect of density on ductility of sintered iron and iron base alloys.

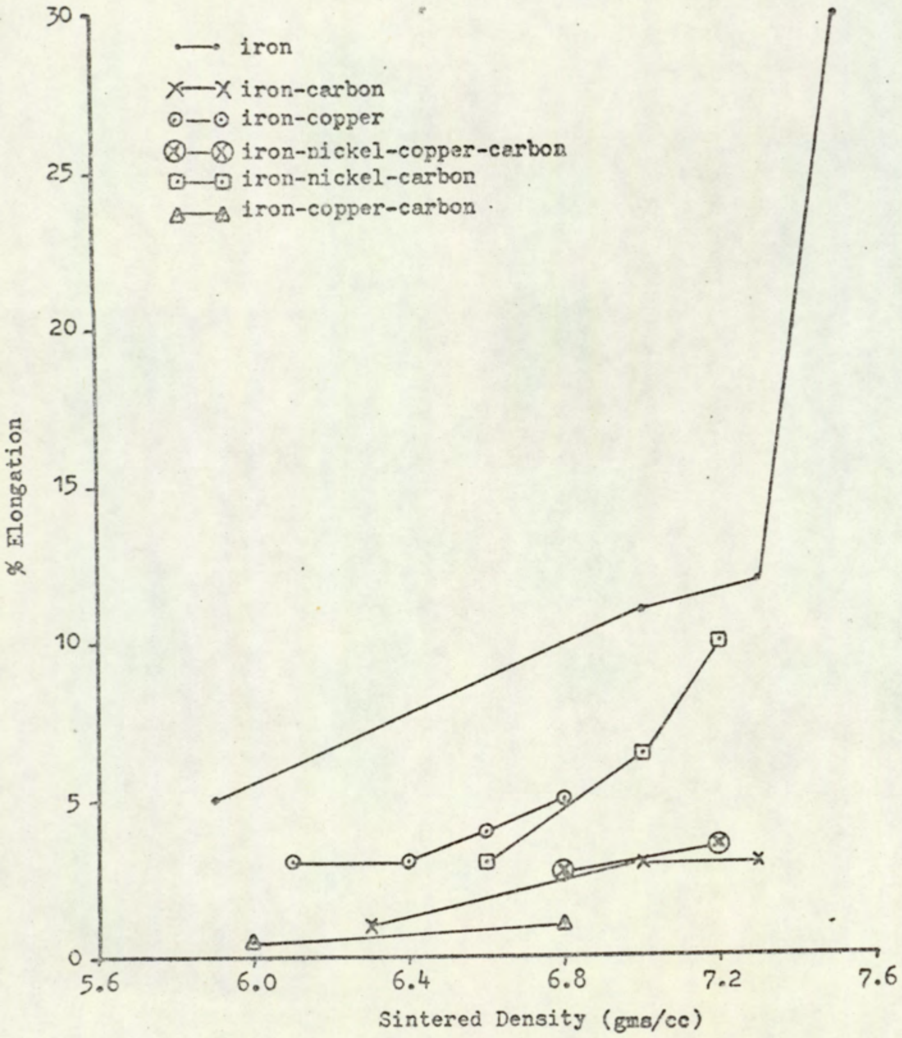
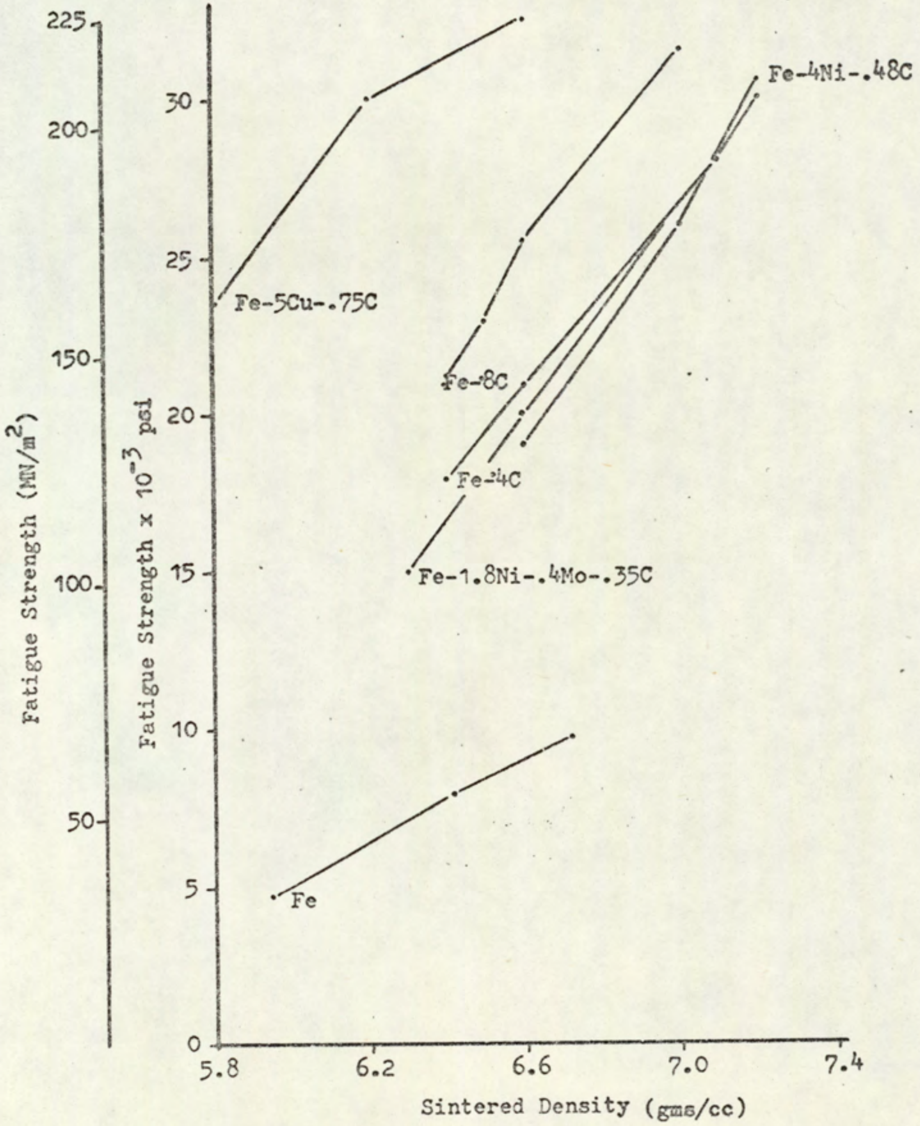


Fig 18 Fatigue strength v As sintered density



being manufactured. Some industry sources estimate a practical limit of $\sim$2,000 tons[18] for automatic mechanical presses, although one press of 2,800 tons is in operation in the USA. The major limitations in the application of such presses are the high capital investment required and the need to fully utilise the press. For instance, a 200 ton Cincinnati press costs $\sim$£60,000 and users in the UK are finding great difficulty in obtaining sufficient parts to fill the press.

ii Developments in Die Wall Lubrication

Two lubrication techniques can be used during die compaction of metal powders:

- admixed lubrication, ie lubricant intimately mixed with the powder prior to compaction
- die wall lubrication

The results of several investigations[15] have shown that die wall lubrication is superior to admixed lubrication in minimising the pressure required to achieve a given green density. Higher densities are achievable since there is no lubricant trapped between the powder particles hindering the collapse of pores as the pressing load increases. The absence of lubricant within the compact has a further advantage in that no dewaxing treatment is required.

At the present time, die wall lubrication is not widely used in the powder metallurgy industry, since no suitable lubricant injection system for use on modern presses with high production rates has yet been developed.

b Isostatic Pressing

Isostatic compaction is being applied to powder metallurgy and the process is based on the principle that when a pressure is applied to a fluid it propagates equally in all directions, thus producing a uniform isostropic force.

i Cold Isostatic Pressing

Cold isostatic pressing is usually carried out at room temperature using a liquid as the pressure transport medium. High uniform densities are produced and this in turn results in:

- a reduction in subsequent sintering time and/or temperature to produce specific mechanical properties
- uniform and controlled shrinkage which in turn reduces waste and final machining

The main disadvantages of the process are:

- low production rates compared with conventional techniques
- poor control of dimensions arising from problems in packing the powder to a uniform density

- the flexible rubber moulds used are not sufficiently rigid to maintain accurate shapes whilst being filled with powder

It appears that, as far as powder metallurgy is concerned, the major application of cold isostatic pressing will be in the preparation of dense slugs for further working, particularly in the manufacture of extrusion billets of refractory metals.

ii Hot Isostatic Pressing

The technique of hot isostatic pressing involves the application of pressure through a gaseous medium (usually argon or helium) at a temperature high enough to cause sintering. Hence sintering and compaction occur simultaneously. The pressure and temperature can be varied according to the powder used and the desired final properties. The process avoids some of the limitations of hot die compaction, eg long time cycle, expensive die materials, size limitations, etc, but there are a number of preliminary operations, eg enclosure of powder in a suitable container, which makes the process slow and expensive. At the present time, the technique has not been fully developed, but it appears that the major application will be in the field of refractory metals and ceramic materials.

c High Energy Rate Forming Techniques

High energy rate forming techniques are being used to a limited extent in closed die powder compaction and a number of methods are used to produce the energies required, eg pneumatic mechanical (eg Dynapak), explosive and spark discharge. The velocities of moving rams range from $\sim 10^3 - 2 \times 10^4$ fps and the pressures generated during compaction range from $10^6 - 15 \times 10^6$ psi. The major limitations of the techniques are:

- excessive punch and die wear due to high relative velocities and pressures
- relatively poor dimensional control
- low production rates
- the techniques are very dangerous and require extensive safety precautions

Hence, the techniques are not generally applied to materials which can be fabricated by conventional powder metallurgy techniques.

i Dynapak[19]

General Dynamics (USA) have recently developed the Dynapak machine. It is claimed that this machine produces high green densities and that parts with a surface area of four square inches can be produced at the rate of 25/30 per minute. A further advantage claimed is the ability of this machine to compact materials such as chips and coarse powders which are considered uncompressible by conventional techniques. An example of the capabilities of this process is its ability to compress a 13" diameter disc of iron weighing 20 lb to a density of 7.2 gm/cc using approximately 200 x 10^3 ft.lb of kinetic energy. A 7,000 ton press would be required to produce these properties by conventional compaction.

ii Explosive Compaction[20]

The process of explosive compaction is very simple and requires a small capital outlay compared with that required for more conventional techniques. Metal powders are sealed in mild steel containers and then wrapped in sheet explosive. On detonation, pressures of up to 5 x 10^6 psi and lasting only a few microseconds are generated, thus making the process significantly different from conventional techniques. The generation of pressures of this magnitude result in complete densification of some materials and there is almost no limitation to the size and weight of components that can be produced. A further advantage of the process is that no sintering is required.

This technique has been used for making billets and other simple shapes and also for the compaction of superalloy and refractory metal powders which are difficult to cold press.

iii Spark Sintering[19]

The process of spark sintering involves the application of a high energy spark discharge to a loose mass of powder. The powder is contained in an anodic graphite mould and the spark, which lasts for ~1 microsecond, is struck between this and a graphite cathode. During the discharge, ionisation occurs, removing oxide films and other surface contaminants and fusion occurs between adjacent particles. The discharge is followed by a resistive heating cycle and compaction takes place at elevated temperatures using a static load of ~14,000 psi. The operating cycle lasts approximately twelve to fifteen seconds and controllable densities between 65-99% theoretical are claimed.

At the present time, the technique is applied to materials which are difficult to fabricate by conventional powder metallurgy techniques, ie cold press and sinter, such as aluminium, tungsten carbide, refractory metals, etc.

d Forged Powder Preforms

This technique involves the hot forging of preforms, prepared by conventional powder metallurgy techniques, in a fully closed die system and is often referred to as "Sinter Forging", "Powder Metallurgy Forging" and "Powder Forging". The process produces high densities approaching 100% theoretical and hence a corresponding high level of mechanical properties (Figure 19). Apart from increasing density, the technique has many other advantages over conventional closed die forging, eg

- increased material utilisation
- reduced machining
- close weight control
- superior surface finish
- superior dimensional accuracy

The Haller Corporation (USA) has developed a sinter forging process known as HRC[21] and densities approaching 100% theoretical are claimed, together with tensile strengths of up to 300×10^3 psi with an elongation of 12%. However, no details of alloy systems used to achieve these properties are reported.

e <u>Vibratory Compaction</u>[9]

Vibratory compaction is very similar to conventional die compaction and simply involves the simultaneous application of pressure and vibration to a loose mass of powder within a rigid die. Although the technique produces higher densities for a given compacting load than static die compaction, additional fatigue problems arise which may result in a significant decrease in tool and machine life. A further problem at the present time is the application of the system to commercial tools and presses and it appears that the main appli-

Fig. 19 Schematic relationship between density and mechanical properties

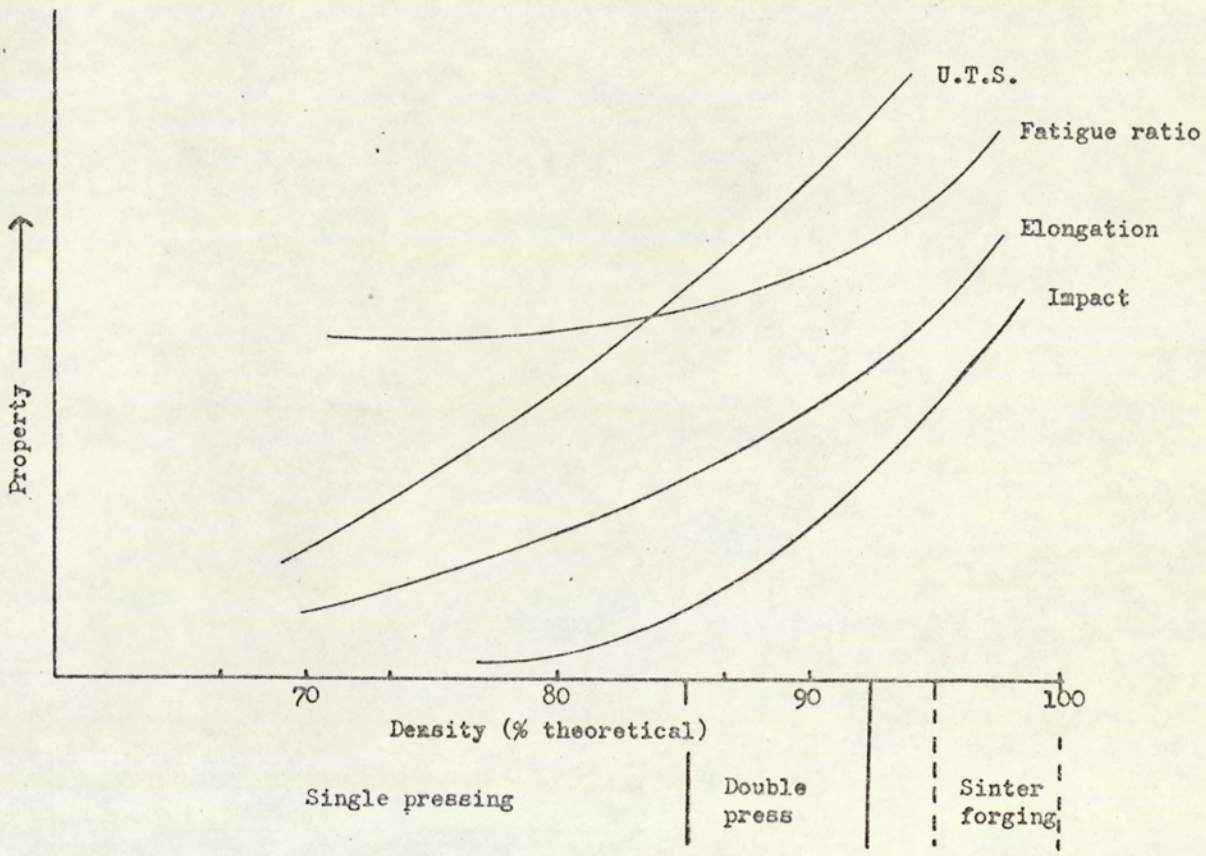
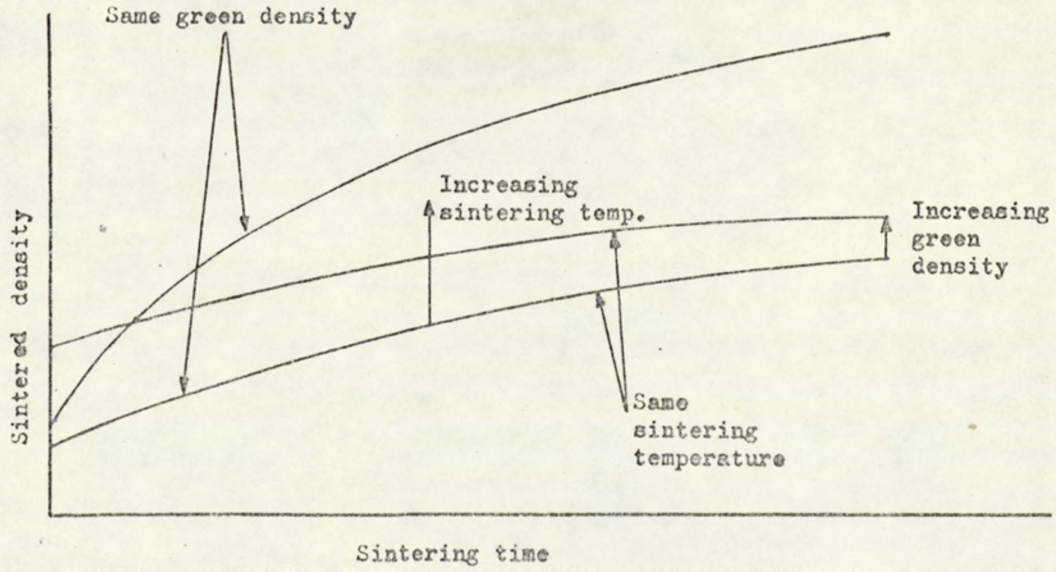


Fig. 20 Sintered density v sintering time



cations of the technique will be in fabrication of materials difficult to press by conventional techniques.

f Use of High Compressibility Powders

The density of powder metallurgy compacts is governed to a large extent by the characteristics of the powders used. One of the most significant properties of a metal powder is its compressibility which is defined as the amount by which a mass of powder can be compressed or increased in density.

The compressibility of a metal powder is enhanced by the following:

- the ability of the powder to deform plastically. This is reduced by the presence of impurities.
- a high internal porosity or a low apparent density, although due to its purity and therefore ability to deform plastically, electrolytic iron powder is an exception.
- a wide range of particle size.

A comparison of the green densities produced by various iron powders pressed at 40 tsi is given in Table 8.

Electrolytic iron powder is expensive (4 times the cost of sponge iron) and users of iron powders have tended to use mixtures of sponge and electrolytic to reduce costs. However, a number of powder manufacturers

Table 8

Green Densities of Various Iron Powders[22]

	Carbonyl	Sponge	Electrolytic	Hydrogen Reduced Mill Scale
Density (gm/cc)	6.60	6.63	7.06	6.38

are now attempting to produce powders with compressibilities similar to that of electrolytic powder but at a reduced cost.

g Control of Sintering Time and Temperature

During sintering, densification is achieved by increasing the size of interparticle bonds produced during compaction, ie elimination of porosity. This involves material transport and several mechanisms have been suggested:[17]

- Diffusion mechanisms - surface diffusion, grain boundary diffusion and volume (bulk) diffusion
- Plastic flow mechanisms
- Condensation and evaporation

All these processes are time and temperature dependent and hence the controlling effect of sintering time and temperature on the final density, Figure 20. However, it is very difficult to achieve complete densification during sintering since there is a decrease in driving force as the pores shrink and high densities are only achievable by using extreme sintering conditions, ie high sintering temperatures and long sintering times. This is uneconomical and in most systems adequate mechanical properties can be produced using sintering temperatures of 1120-1150°C and sintering times of 30-45 minutes. This temperature range is, in fact, the limit of most continuous mesh belt furnaces and higher temperatures involve the use of slower, more expensive conveyor systems, together with more expensive furnaces.

b Infiltration

The most widely used material for infiltration of iron base powder metallurgy components is a copper base alloy since:

- copper is molten at normal sintering temperatures ie 1120-1150°C
- copper has a limited solubility in iron
- no high melting point phases are produced between molten copper and iron
- its surface oxide is easily reduced under the conditions used to produce iron base components

Hence, at normal sintering temperatures, there is no hindrance to the flow of the molten copper alloy

The process involves conveying iron base components, which may or may not have been previously sintered, through a sintering furnace in contact with a copper base infiltrant material. The most widely used infiltrant is a Cu 5% Fe 5% Mn alloy which has been shown to prevent excessive erosion.[15] The furnace temperature is such that the infiltrant melts and is drawn into the pores by capillary action. The original iron base skeleton must contain interconnecting porosity to ensure good penetration, and high densities, eg 7.5-7.6 gm/cc are achieved since the liquid copper simply fills up the pores. Some alloying also occurs and this can produce improved mechanical properties. Infiltration has, however, some disadvantages:

- two heating operations are required
- ~20% copper is generally used, thus increasing the cost and decreasing the control of dimensions
- three pressing operations are often required to press the skeleton, the infiltrant and to repress (size) the final component

The technique is rapidly losing ground as a production process and is usually limited to applications where high strength and ductility are required and where manufacturers do not wish to use sintering temperatures greater than ~1150°C.

i Repressing and Sintering (Double Pressing)

This technique involves pre-heating the green components below normal sintering temperatures, followed by repressing and sintering at conventional temperatures (1120-1150°C). The pre-heating treatment removes any lubricant present, thus enabling densification during the second pressing operation. Further densification is then achieved during final sintering. Examples of increases in density and hence mechanical properties produced using this technique are given in Table 9.

Although this technique provides useful increases in density, it increases the cost of the components, since two compaction and two heat treatments are required.

j Repressing

The density of powder metallurgy components can be increased by repressing after sintering. However, a second die set is required and large compacting loads may be necessary when repressing strong alloy steel components. In practice, repressing is usually used as a final sizing operation where changes in dimensions and hence density are relatively small.

Table 9

Effect of Repressing and Sintering on Final As-Sintered Properties

Density gm/cc	UTS psi x 10^3 (MN/m^2)	Fatigue Limit psi x 10^3 (MN/m^2)	Elongation %	Alloy System	Production Technique
6.3-6.5	20.36 (141)	8.5 (58.5)	-	Fe	Pressed at 150 tonnes,[23] then: i pre-heat 20 minutes at 580°C ii sinter at 1120°C
6.9	41.35 (284)	14.9 (102)	-	Fe	As above. but repress after i at 150 tonnes
6.4-6.8	45 (309)	-	1.0	Fe - .4% C	a Pressed to 6.6 gm/cc, pre-heat[24] 24 minutes at 816°C, sinter 30 minutes at 1120°C. Quench from 950°C and temper at 240°C
6.8-7.2	72 (495)	-	.8	Fe - .4% C	b As a except repressed to required density after pre-heating
7.2-7.6	104 (715)	-	.2	Fe - .4% C	Treatment b
6.4-6.8	68 (466)	-	1.0	Fe - .45%C .4%Ni	Treatment a
6.8-7.2	120 (825)	-	1.0	Fe - .45%C .4%Ni	Treatment b
7.2-7.6	182 (1250)	-	2.0	Fe - .45%C .4%Ni	Treatment b

3.2 Alloying

3.2.1 Alloying Techniques

A variety of techniques are available for producing iron base alloy components and these are reviewed in Table 3. At the present time, the majority of alloy components are produced using pre-mixed elemental powders since pre-alloyed powders generally have inferior compressibilities and are only available in a limited composition range. Most of the published mechanical property data has been obtained using pre-mixed elemental powders.

3.2.2 Alloy Systems

a Iron-Carbon

i As sintered mechanical properties

Mechanical Strength

The relationship between mechanical strength and combined carbon for solid steel and sintered steels is shown in Figures 21 and 22 respectively. In the case of sintered steels, the sudden fall off in strength at .9% combined carbon is mainly due to the formation of intergranular cementite.

Ductility

The increase in strength of sintered steels with increasing carbon content is usually at the expense of ductility, Table 10.

Fig. 21 Combined carbon v tensile strength - Solid steel[26]

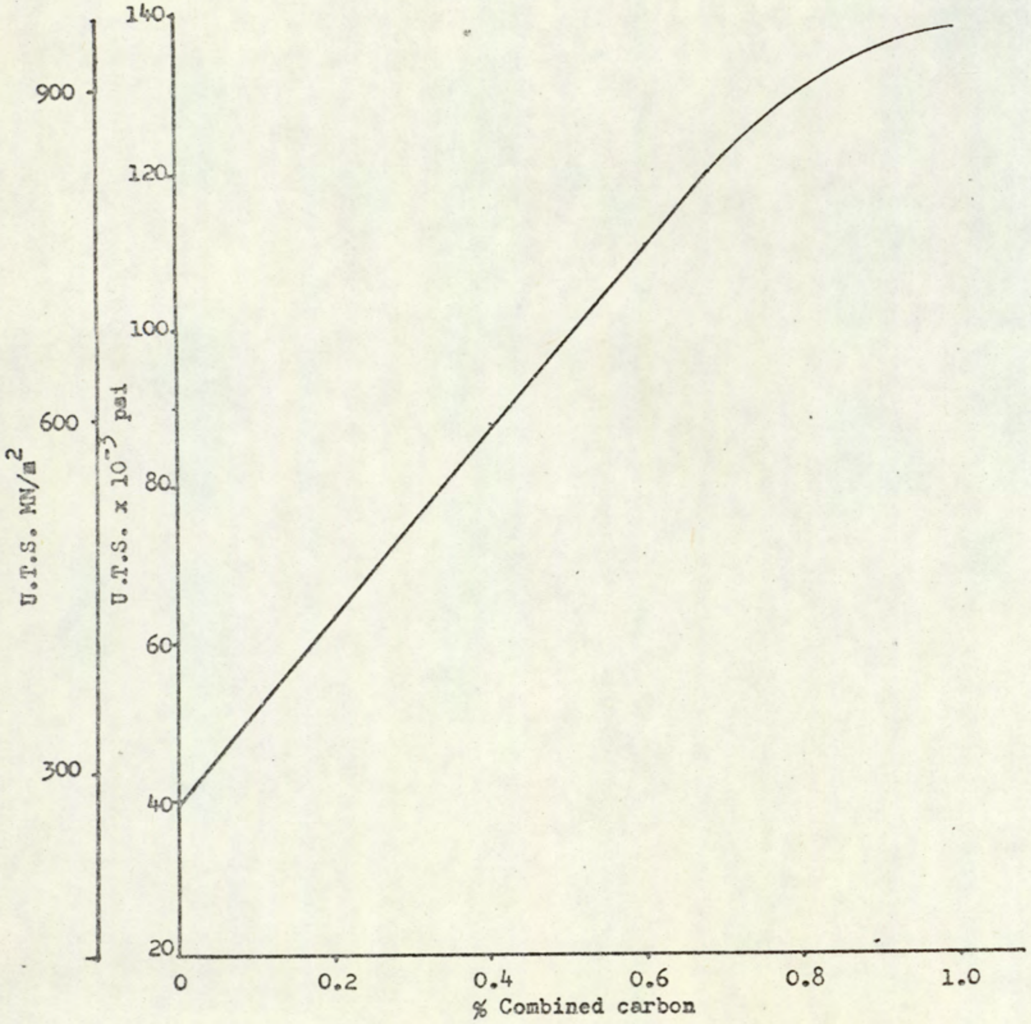


Fig. 22 Combined carbon v transverse rupture strength - Sintered steel[26]

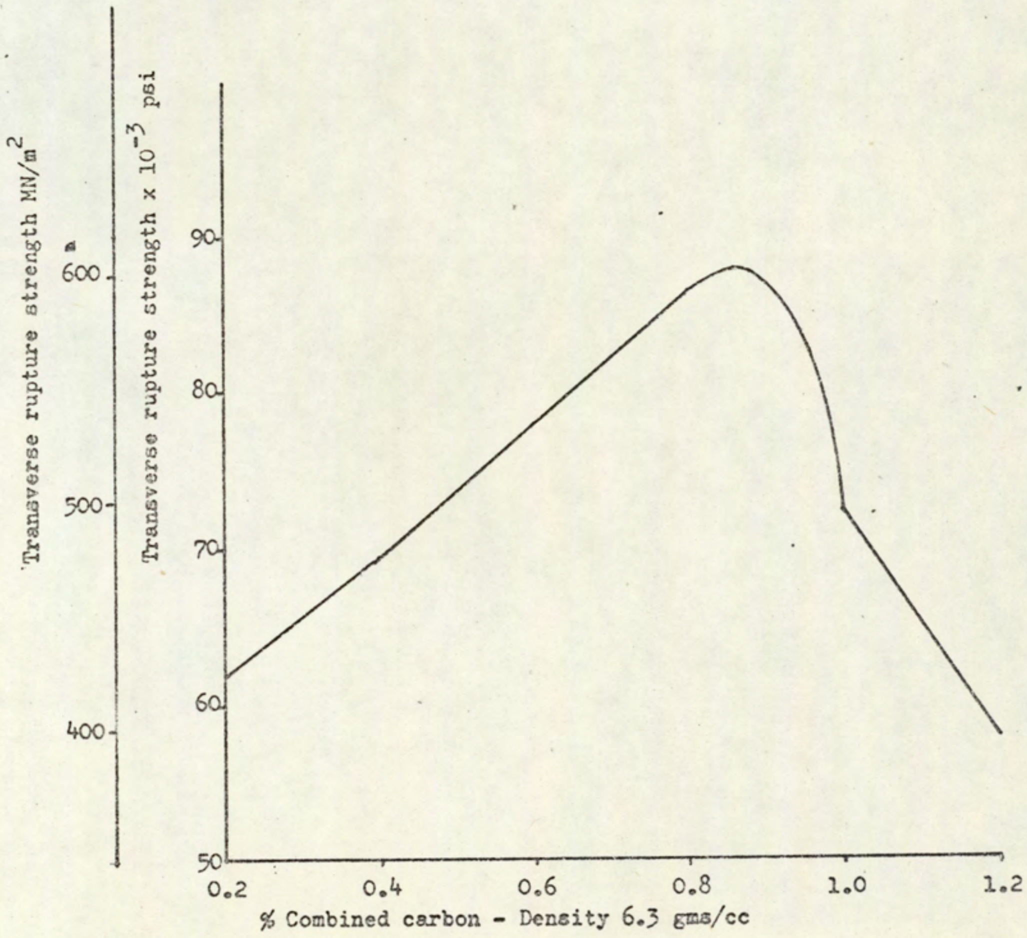


Table 10

Strength and Ductility of Sintered Iron-Carbon Compacts[27]

% C	Density gm/cc	UTS x 10^3 psi (MN/m^2)		% Elongation
0	6.78	26.4	(182)	20.0
.2	6.80	28.9	(199)	12.8
.5	6.78	50.1	(345)	5.0

Fatigue Strength

The limited fatigue data available showing the effect of increased carbon content on fatigue strength is shown in Figure 18. This data indicates that increased carbon content improves fatigue strength at a variety of density levels.

ii Alloying Mechanisms and Dimensional Stability

Alloying occurs during sintering by diffusion of carbon, from the various sources used, eg hydrocarbon atmosphere, solid carburising agent or premixed-graphite, into the iron lattice. The rate and extent of such carbon diffusion is dependent upon carbon concentration gradients, sintering time and temperature and hence the controlling effect of these parameters on mechanical properties, Figure 23.

Fig 23 Sintering Time v Transverse Rupture Strength[26]

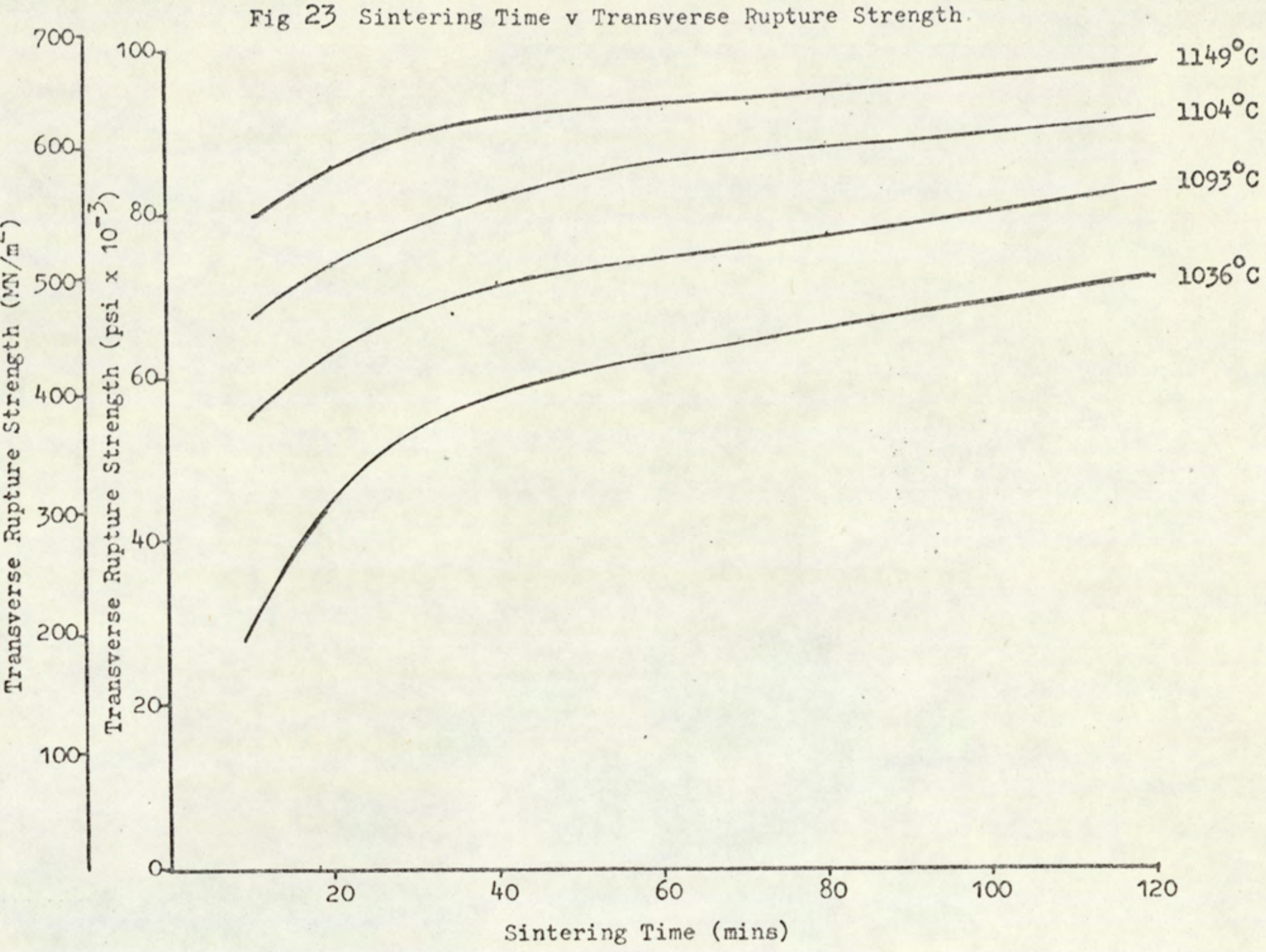
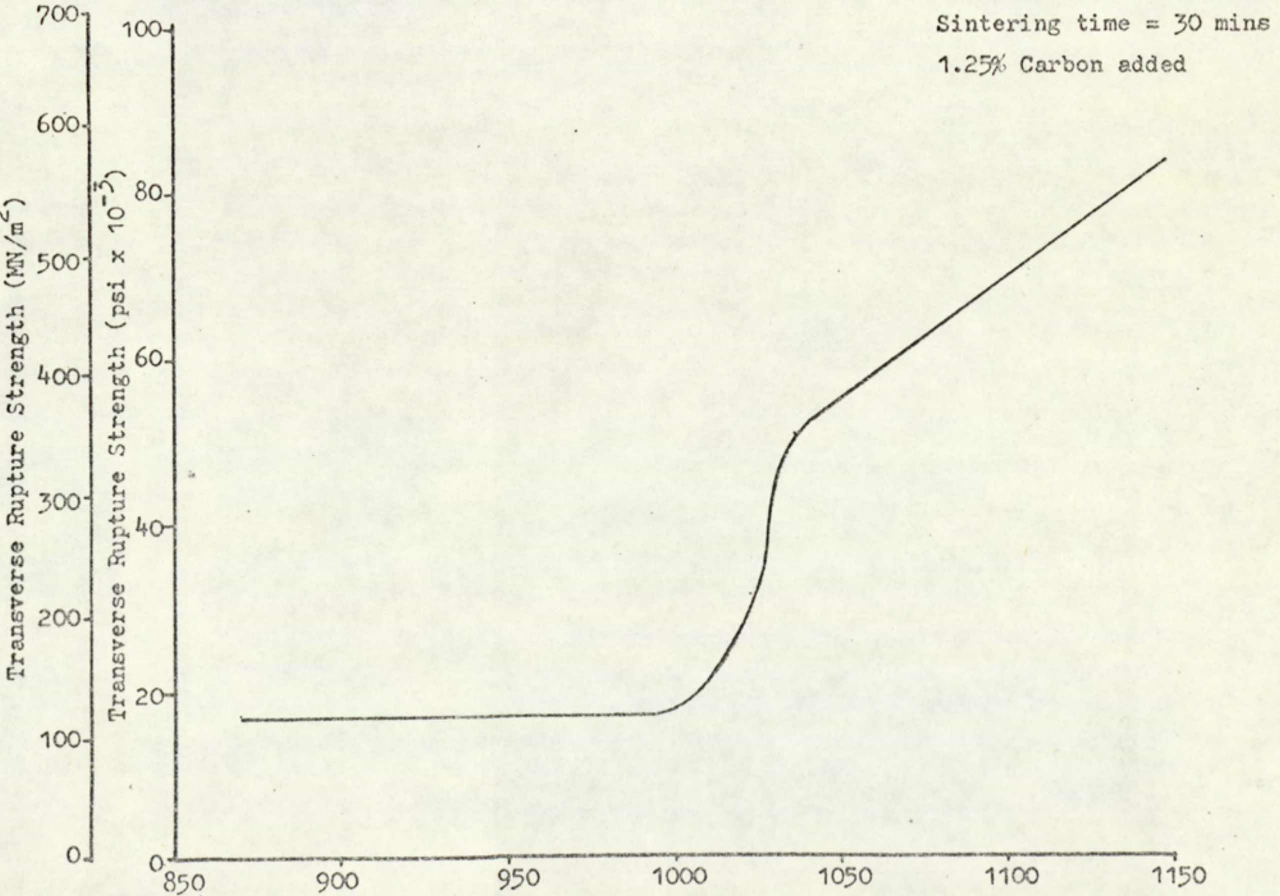


Fig 24 Sintering Temperature v Transverse Rupture Strength.[26]


Sintering time = 30 mins

1.25% Carbon added

Strengthening is achieved by the production of iron carbide (cementite) which appears in the form of pearlite in components which have been slowly cooled to room temperature. At sintering temperatures below ~1000°C very little increase in strength is achieved, Figure 24, since the rate of carbon diffusion and hence the formation of cementite is very slow. However, above a critical sintering temperature of ~1000°C rapid carbon diffusion results in increase in strength. At higher sintering temperatures, further increases in strength occur due to the conventional stages of sintering, ie spheroidisation and shrinkage of pores.

The dimensional stability of iron-carbon compacts depends not only on sintering time and temperature, Figure 25, but also on the type of iron and graphite powder used. In general, iron-carbon alloys show an expansion during sintering due to carbon diffusing into an interstitial position in the iron lattice. However, porosity is created by carbon diffusion and since this increases the total porosity, increased shrinkage due to pore spheroidisation and elimination may be produced. This increase tends to counteract some of the expansion due to interstitial carbon and accounts for the reduced expansion at higher sintering temperatures and longer sintering times.

Fig. 25 Growth of iron-1.5% graphite compacts

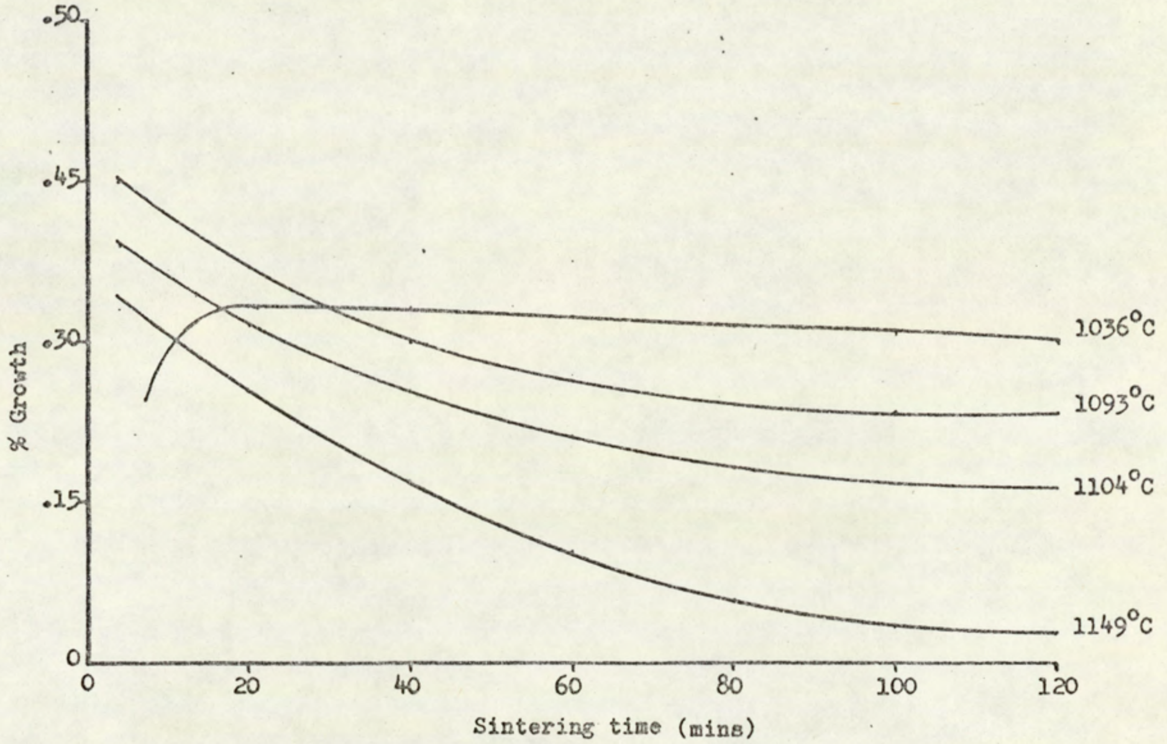
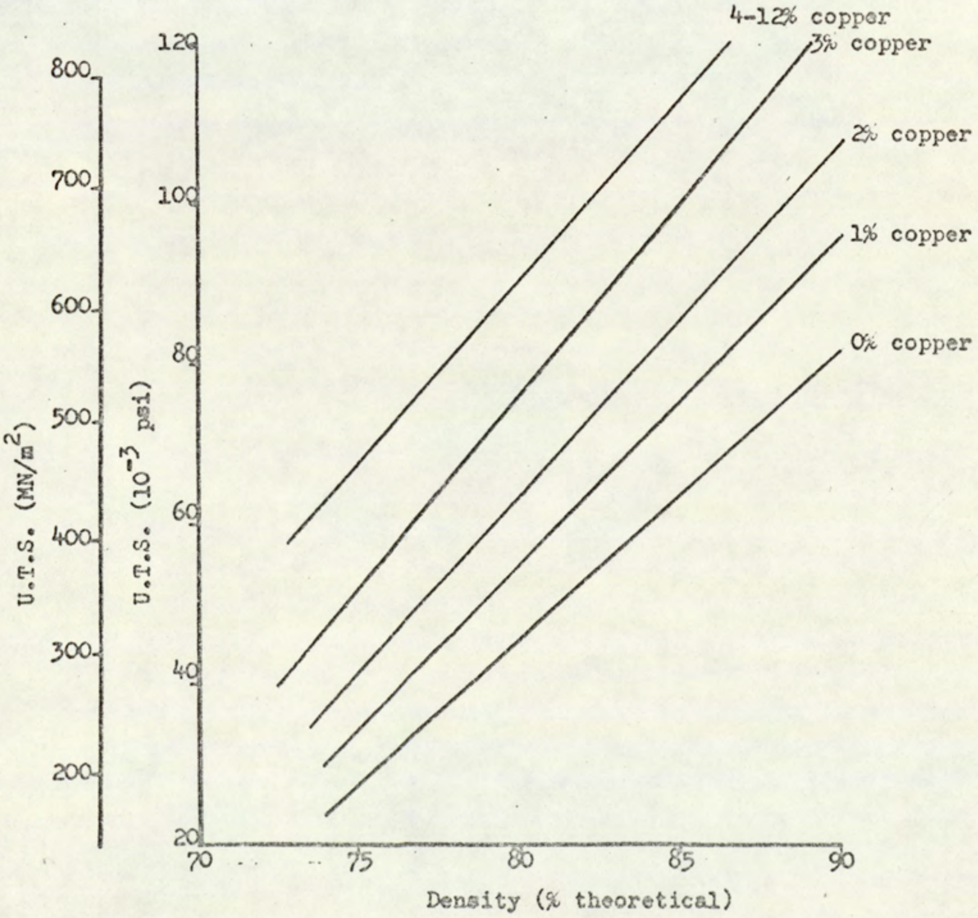


Fig. 26 Sintered strength v Density of iron-copper alloys[28]



b Iron-copper and Iron-copper-carbon

i As sintered mechanical properties

Mechanical Strength

Figure 26 summarises the results reported by Krantz[28] and shows that the tensile strength of iron-copper alloys increases with increasing copper content up to ~5% Cu. Beyond this level the strength is dependent on density and independent of copper content. Other investigators have reported similar effects at copper contents ranging from 4-25%.

The presence of carbon produces an expected increase in tensile strength of iron-copper alloys, Figure 27, and a maximum is produced at 5% copper.

Ductility

The elongation of sintered iron-copper alloys falls rapidly with increasing copper content, up to ~5% copper and beyond this level it increases slightly, Figure 28. The presence of carbon reduces the ductility of iron-copper compacts and there is little change in elongation with increasing copper content, Figure 27.

Fatigue Strength

Fatigue data for sintered iron-copper and iron-copper-carbon alloys is very limited and no detailed information concerning the direct effect of copper was found in the literature reviewed. However,

Fig. 27 Strength and ductility of sintered iron-copper and iron-copper + 1% graphite alloys[29]

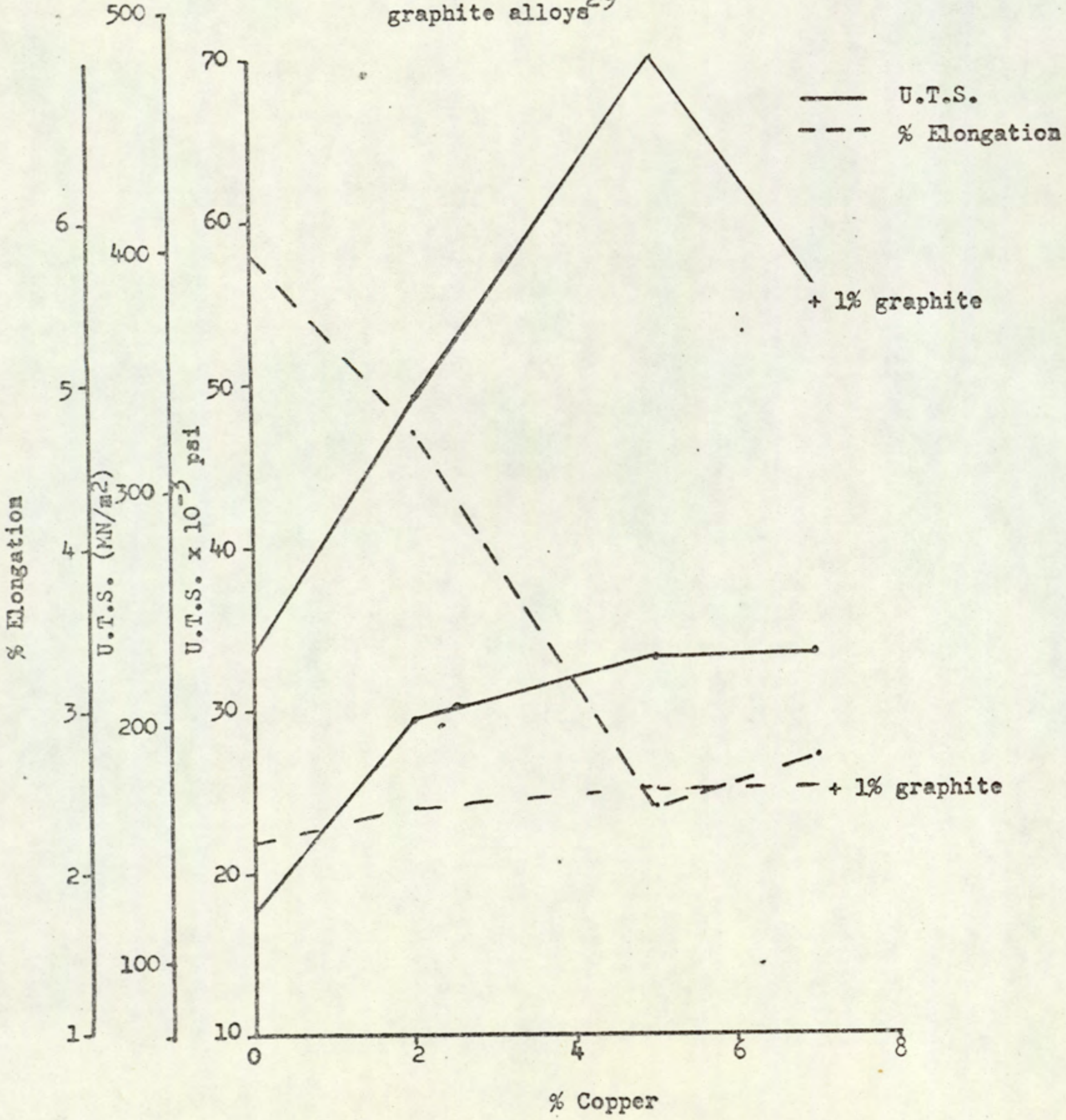
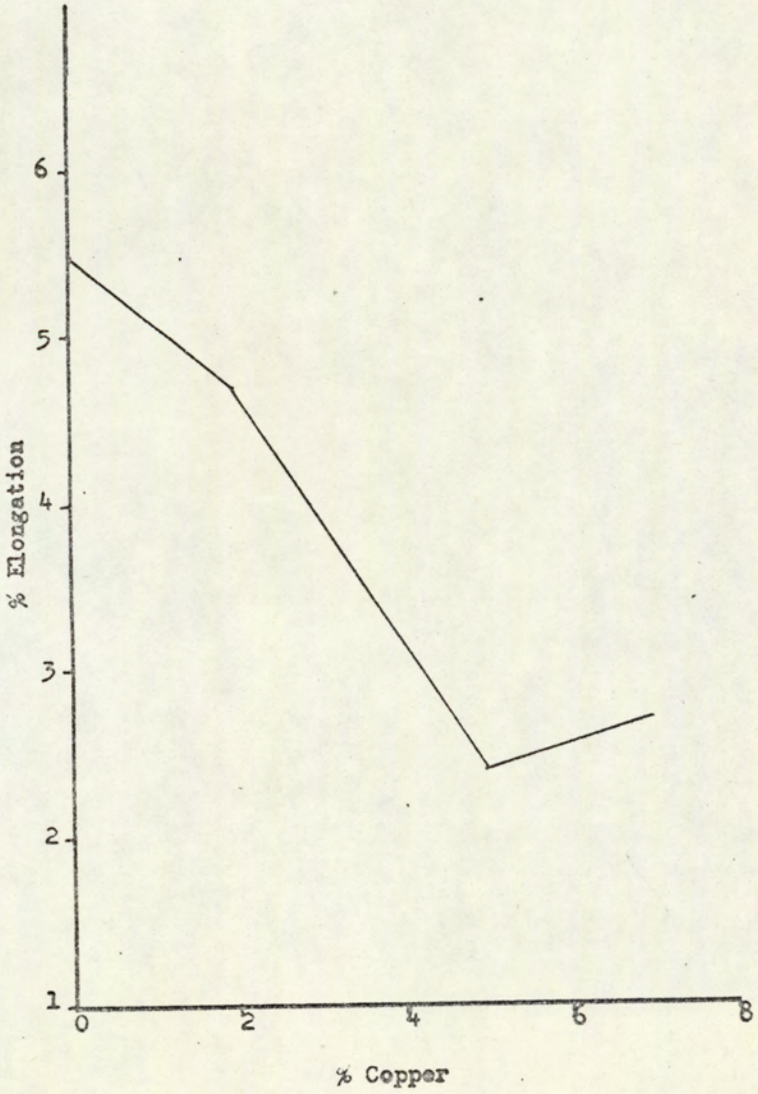


Fig. 28 Ductility of sintered iron-copper alloys[29]



since up to ~5% copper, with and without graphite additions, produces increased tensile strength, corresponding increases in fatigue strength should also be produced.

Figures 29 and 30 show S/N curves for sintered steels produced by infiltration and pre-mixed elemental powders respectively.

ii Alloying Mechanisms and Dimensional Stability

The sintering of iron copper alloys involves the following:

a solid bonding of iron to iron

b solid bonding of copper to iron

c melting of copper

d diffusion of copper

e solution and re-precipitation of iron in liquid copper

The instance at which each of these mechanisms occur and the extent of their occurance is extremely complex. At normal sintering temperatures copper is molten and diffuses rapidly into the iron particles. Iron is also soluble in molten copper and some iron dissolves and is re-precipitated.

The solubility limit of copper in iron is about 8% at normal sintering temperatures, viz 1120°C,

Fig. 29 S/n curves for as sintered 5.0% copper - 0.75% carbon steel[30]

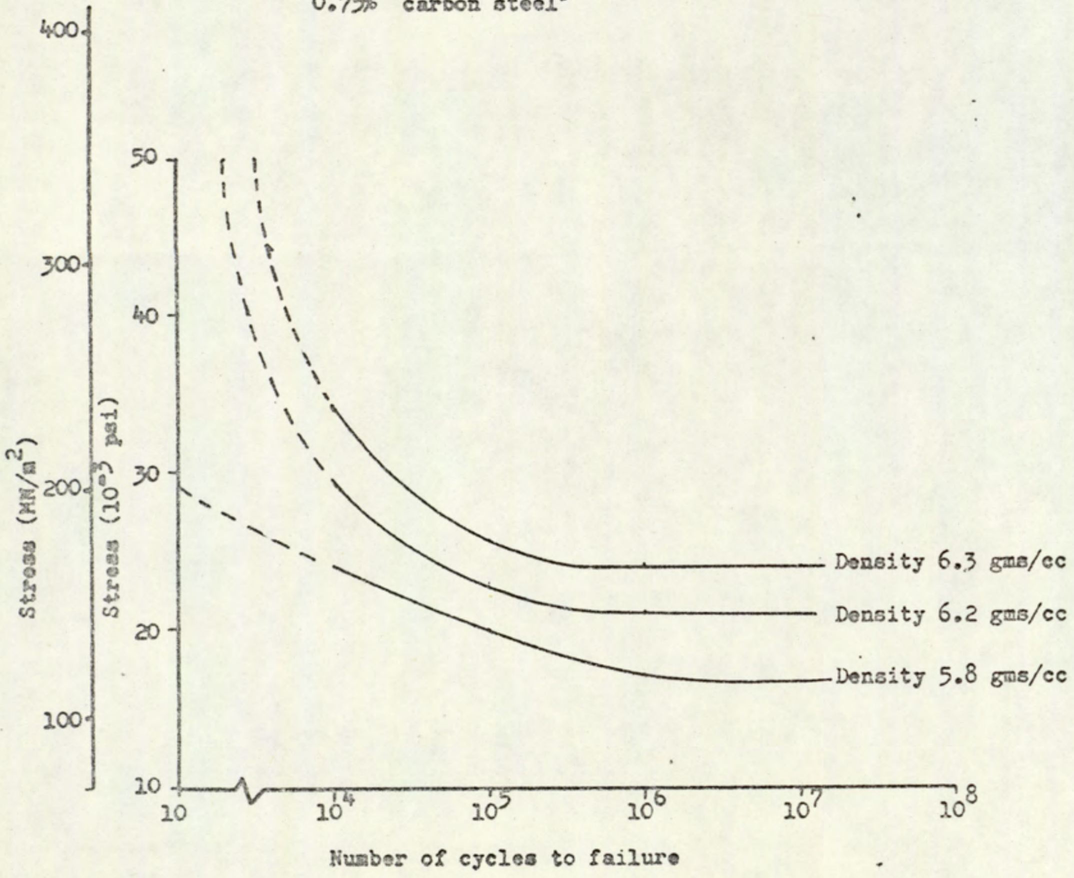
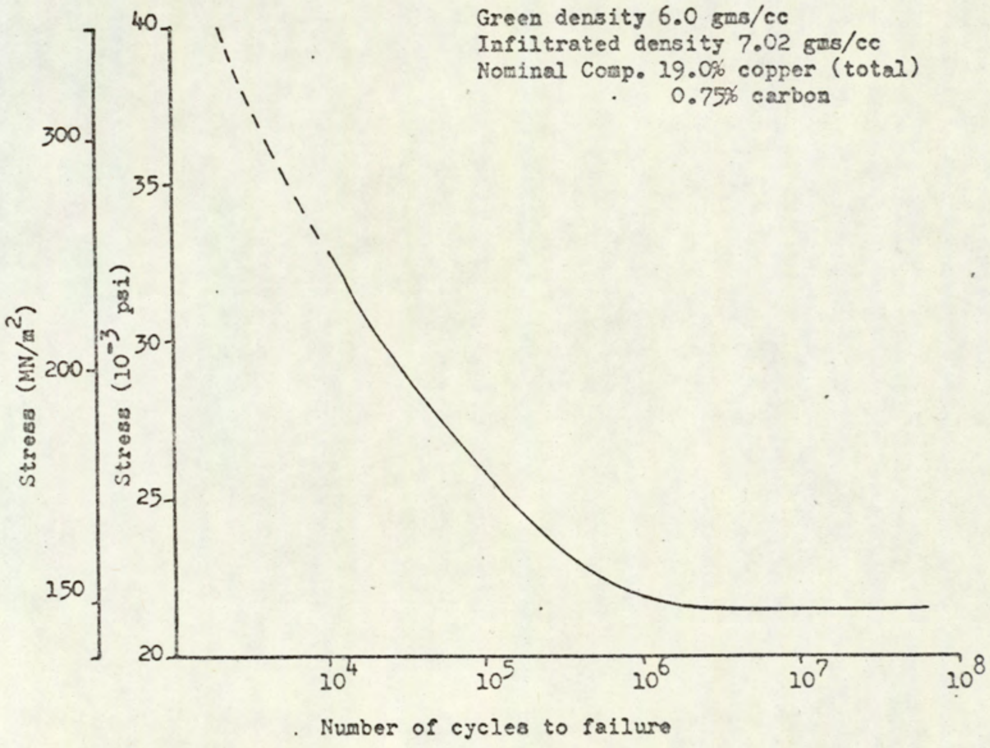


Fig. 30 S/n curve for as sintered copper infiltrated steel[30]



but this decreases rapidly to <.1% at room temperature, resulting in the production of a fully dispersed copper precipitate. The faster the cooling, the finer this precipitate.

Iron-copper alloys undergo considerable expansion during sintering due to the expansion of the iron lattice by the diffusion of copper. This expansion has been shown to be of maximum at 8% copper which is the approximate solubility limit of copper in austenite at 1120°C, Figure 31. However, iron powders are now available which do not show large expansion when used with pre-mixed copper,[9] eg hydrogen reduced (Pyron) iron powder, but as yet no explanation for this behaviour has been reported.

The presence of carbon markedly reduces the expansion described above and promotes strength increases due to the formation of cementite. The exact mechanism of growth inhibition is not fully understood but it has been suggested that carbon atoms segregate at grain boundaries, thus retarding copper diffusion.[28] It has also been suggested that the liquid iron-copper-carbon ternary eutectic formed during sintering increases the amount of liquid phase present and thus causes the sintered iron skeleton to yield under residual surface tension and hence undergo a sudden shrinkage.[8,9]

Fig 31 Growth of sintered iron-copper alloys.[8]

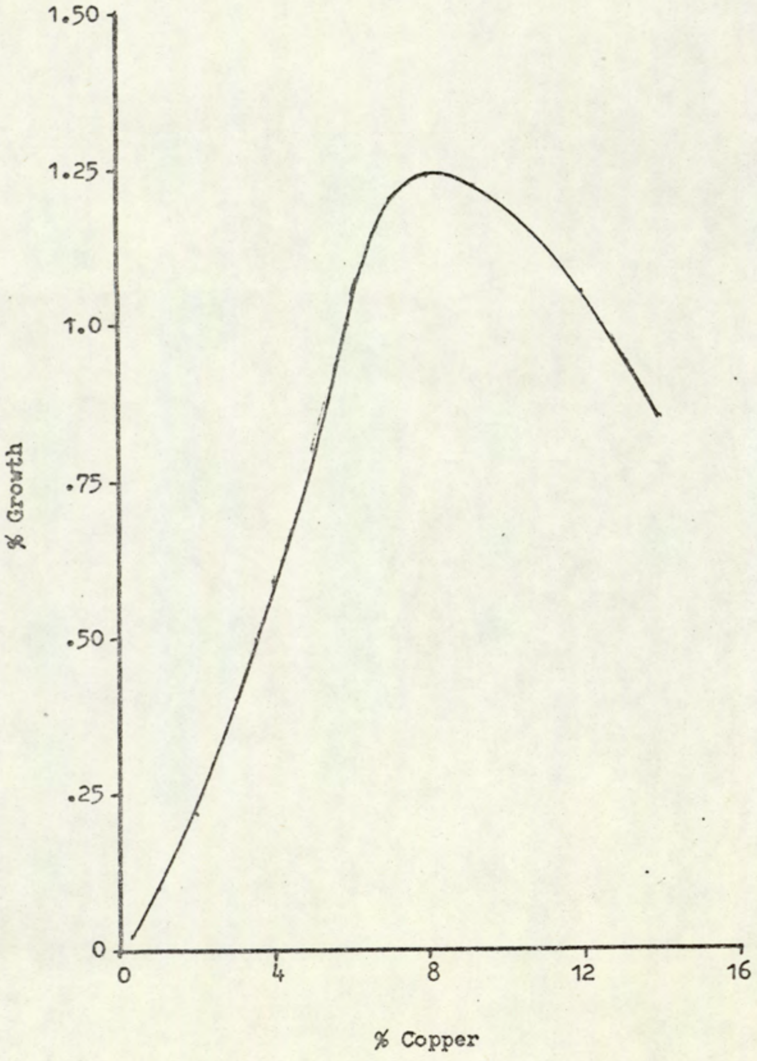
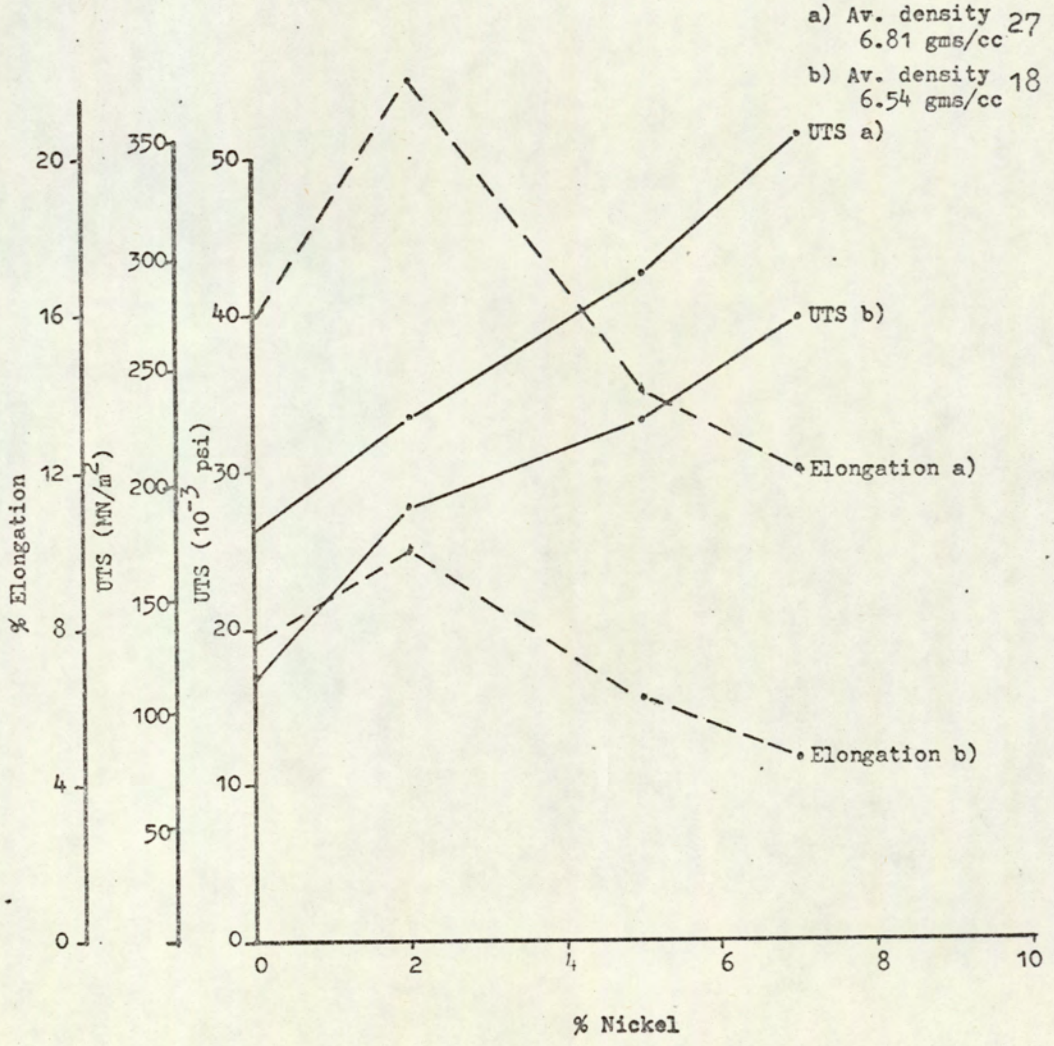


Fig 32 Effect of increased nickel content on the strength and ductility of sintered iron-nickel alloys.

a) Av. density 6.81 gms/cc[27]

b) Av. density 6.54 gms/cc[18]



Although the possible production of an iron-copper-carbon ternary eutectic has been suggested in several publications, no direct metallographic evidence has been reported in published literature.

c Iron-Nickel and Iron-Nickel-Carbon

i As sintered mechanical properties

Mechanical Strength

The tensile strength of sintered iron-nickel compacts increases with increasing nickel content in the range 0-7% nickel, Figure 32. Although sintered nickel steels are usually used in the quenched and tempered condition in order to obtain maximum benefit from the alloy content, increases in as sintered strength are produced with increasing nickel content (range 0-7%) and increasing carbon content up to .8%, Figures 33 and 34 respectively.

Ductility

The elongation of sintered iron-nickel alloys is a maximum at 2% nickel, Figure 32. The presence of carbon reduces ductility at all levels of nickel and removes the maximum observed in simple iron-nickel alloys, Figures 33 and 34.

Fatigue Strength

No fatigue data was found in the literature reviewed for sintered iron-nickel alloys. However, since fatigue strength generally increases with

Fig. 33 Effect of increased nickel content on mechanical properties of sintered iron-nickel - .5% carbon alloys[31]

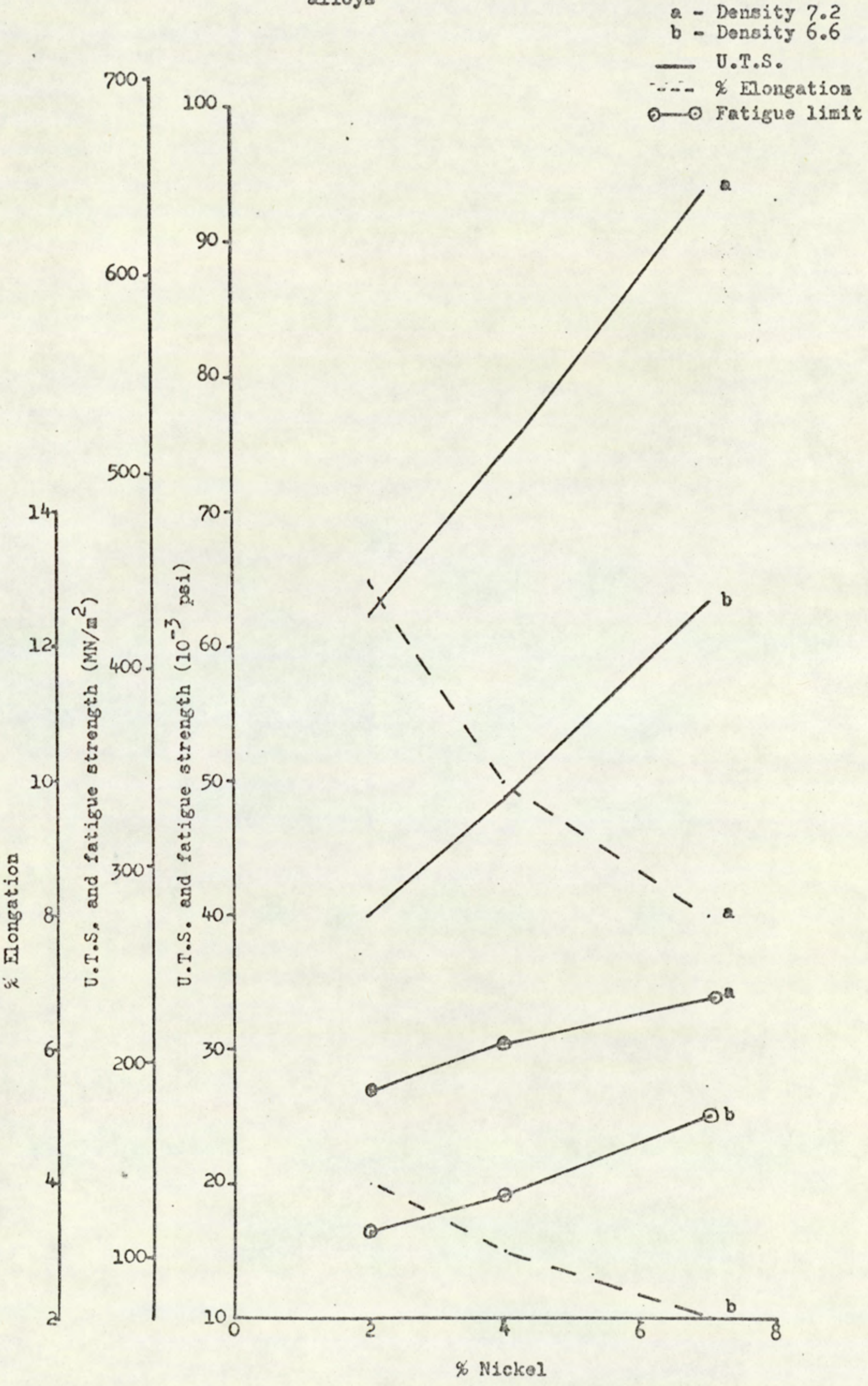
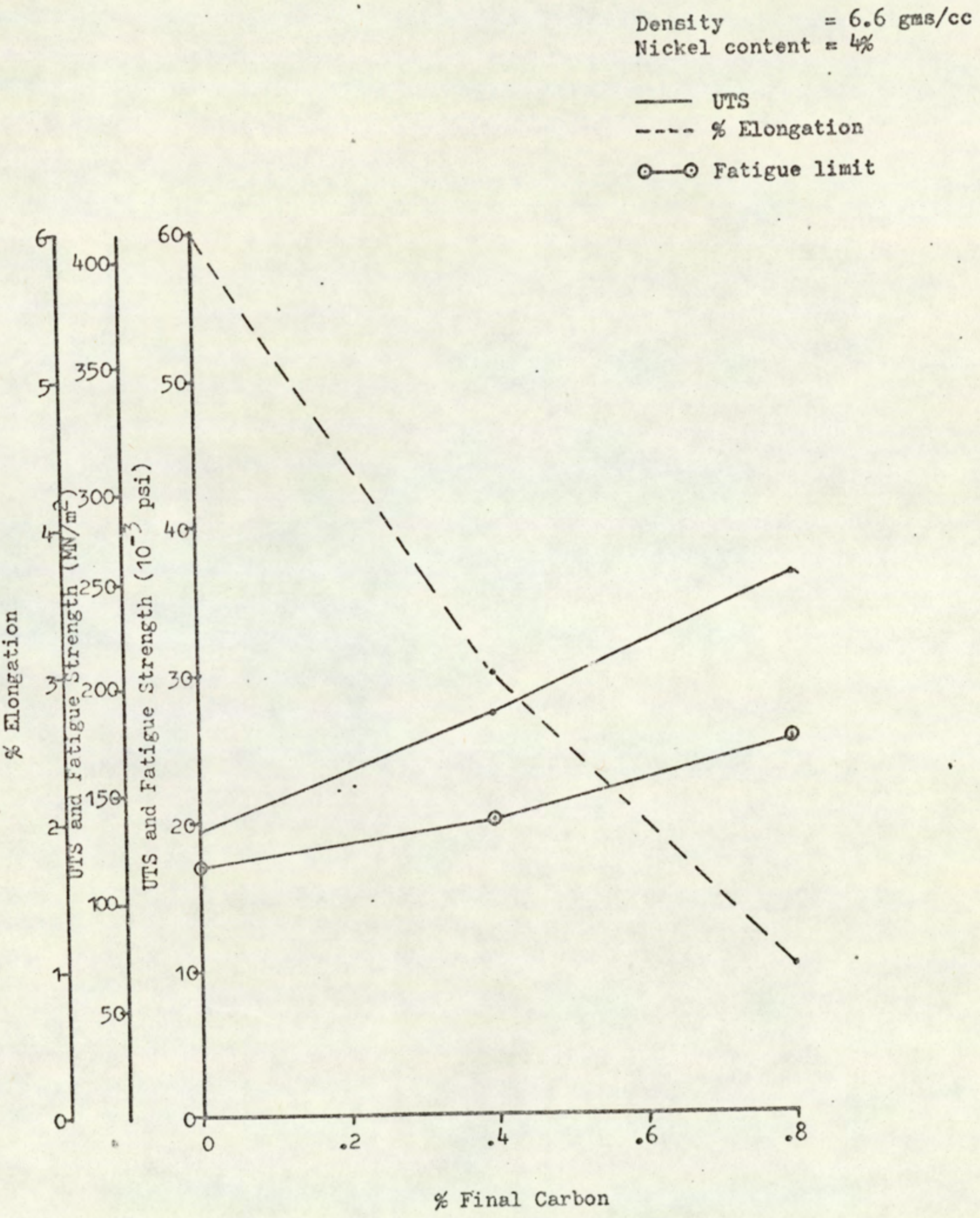


Fig 34 Effect of final carbon content on mechanical properties of sintered iron-nickel-carbon alloys.[31]



Density = 6.6 gms/cc
Nickel content = 4%

increasing tensile strength, the fatigue strength of as sintered iron-nickel alloys should increase with increasing nickel content.

The fatigue properties of sintered iron-nickel-carbon alloys have been examined in detail and Figures 33 and 34 respectively indicate that fatigue strength increases with increasing nickel and carbon content.

The relationship between the fatigue strength and tensile strength of sintered nickel steels (ie fatigue ratio) is shown in Figure 35 and is the same as that produced for sintered iron ie $\sim$.4. The results used to obtain this curve indicate that the fatigue ratio is independent of alloy content, heat treatment and density.

ii Alloying Mechanism and Dimensional Stability

Alloying in both systems is achieved by solid state diffusion. It has been suggested that due to the mechanism outlined below nickel activates sintering and thus produces increased strength. It is also possible that solution of the nickel leads to a solution strengthened structure producing an addition of increase in strength. However, the rate of diffusion of nickel into iron is very slow and fine powders (average) particle size $\sim 5\mu$) together with

Fig 35 Relationship between fatigue strength and tensile strength of sintered iron-nickel-carbon alloys[31]

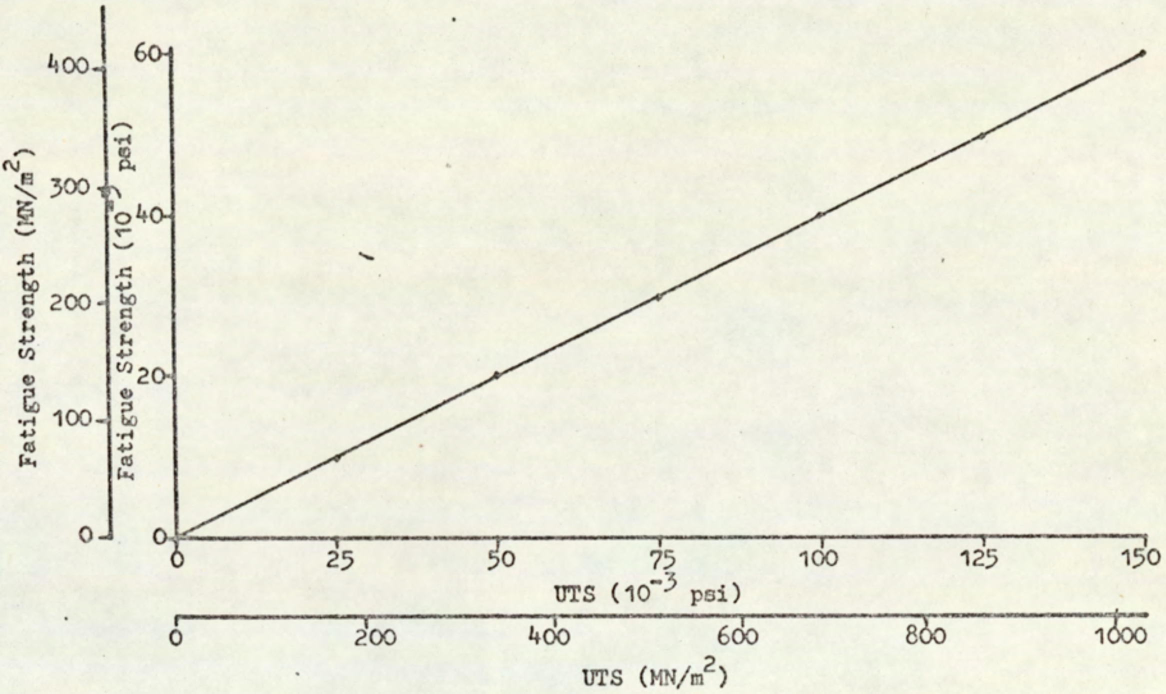
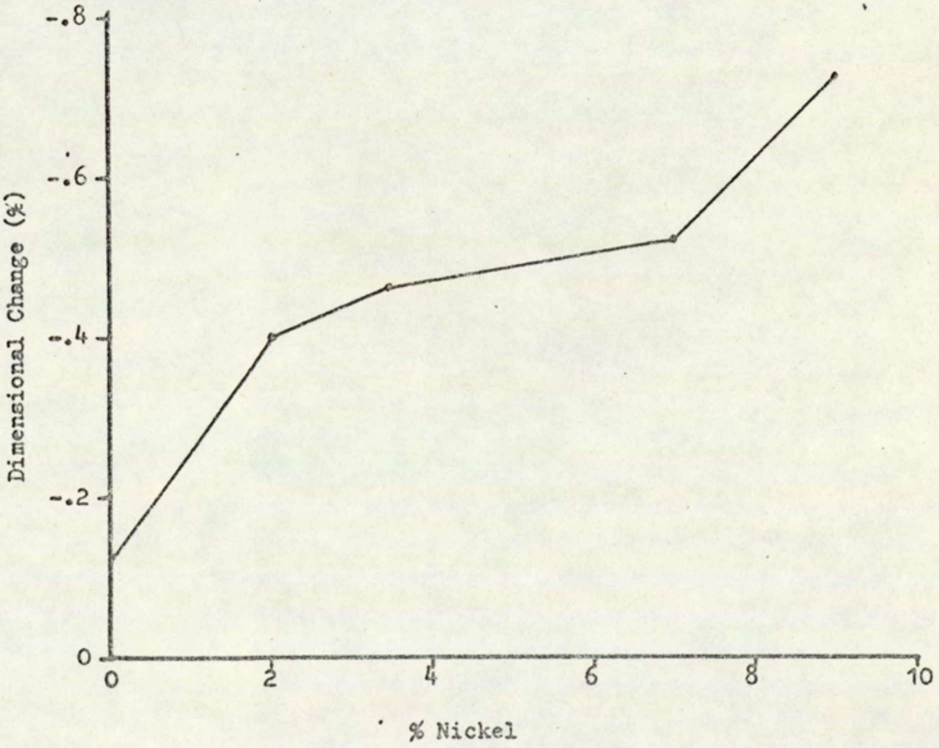


Fig 36 Effect of increased nickel content on the dimensional stability of sintered iron-nickel alloys.[33]



a high sintering time (1-2 hours) are required. Carbon enhances strength as in plain carbon steels.

During sintering iron-nickel alloys undergo excessive shrinkage and the extent of shrinkage is mainly dependent on the nickel content, Figure 36, although sintering time and temperature have a controlling effect. Attempts have been made to explain the mechanism by which such shrinkage occurs, and it has been suggested that it is a result of diffusion porosity, created by nickel diffusing into iron, being eliminated during sintering.[32] The effect of carbon on such shrinkage in iron-nickel alloys is not well established and it has been reported independently that carbon additions both reduce[29] and increase shrinkage.[33]

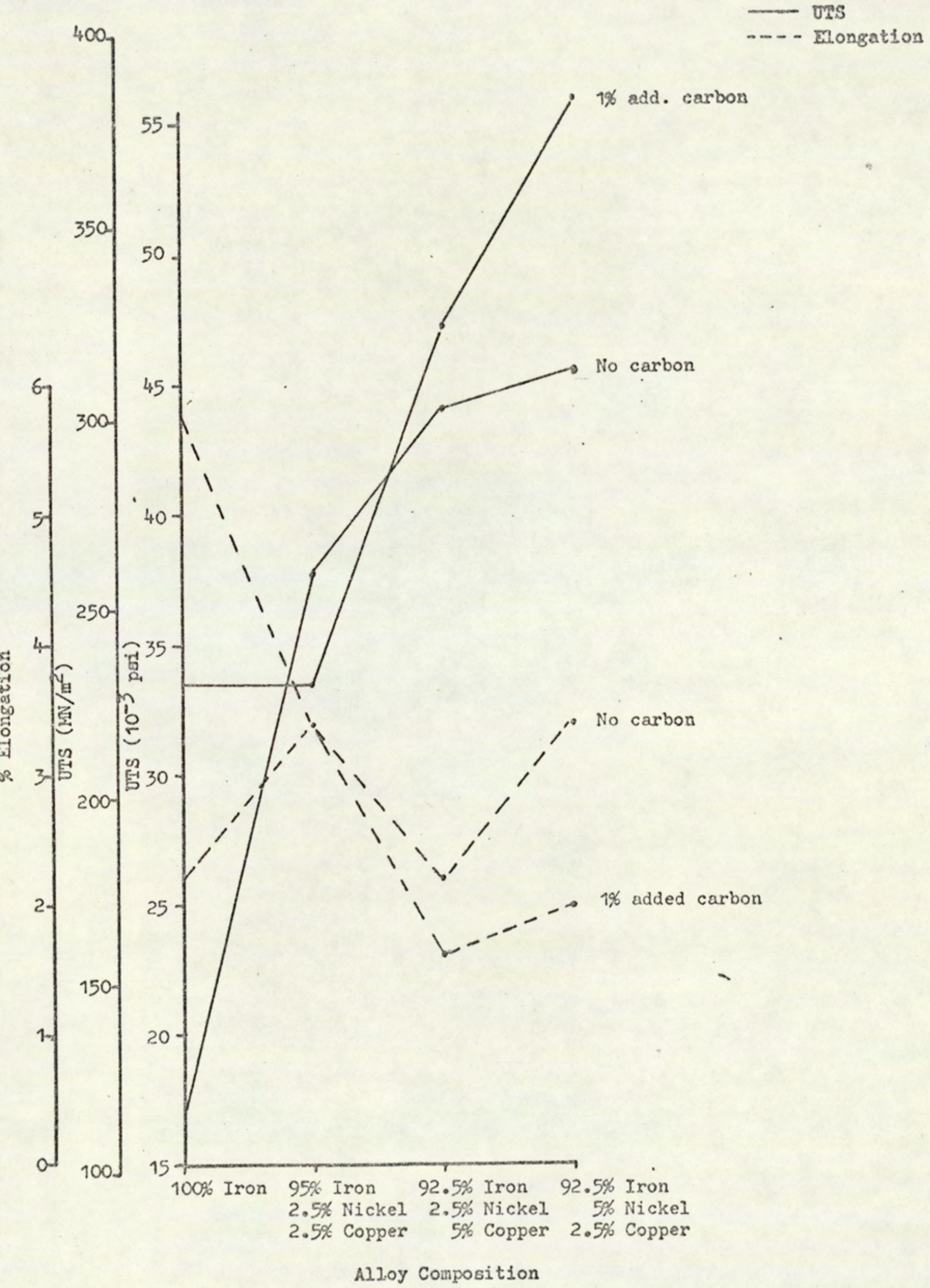
d Iron-Nickel-Copper and Iron-Nickel-Copper-Carbon

i As sintered mechanical properties

Mechanical Strength and Ductility

The tensile strength and elongations of various iron-copper-nickel alloys with and without graphite addition are shown in Figure 37. The strengthening effect of copper and nickel is claimed to be more than additive but the presence of copper impairs ductility, eg 5% Cu - 2.5% Ni and 5% Ni - 2.5% Cu have similar tensile strengths but the higher nickel alloy has a superior ductility, ie 3.4% compared with 2.2%.

Fig 37 UTS and elongation of sintered iron-nickel-copper and iron-nickel-copper-carbon alloys.



The addition of carbon to these alloys produces an expected increase in tensile strength but at the expense of ductility.

Fatigue Strength

No fatigue data was found in the literature reviewed for these alloys but since the presence of such alloying elements increases tensile strength, fatigue strength should increase accordingly.

ii Alloying Mechanisms and Dimensional Stability

Alloying occurs by diffusion and without carbon additions strengthening is achieved by the production of a solution hardened structure. Carbon further increases strength due to the production of cementite. Alloying is enhanced by the presence of liquid copper and good mechanical properties can be produced by sintering at 1120-1150°C for ~30 minutes.

Iron-nickel-copper-carbon alloys show a wide range of dimensional change during sintering and this can vary, depending on the alloy composition and sintering conditions used, from an expansion to a shrinkage. By careful control of alloy composition and sintering conditions, it is possible to produce compacts which show no dimensional change during sintering. It has been suggested that in such systems, the expansion due to copper is simply balanced by the shrinkage due to nickel[8] but no attempts have been made to predict or investigate the precise mechanisms involved.

e Iron-Copper-Tin

i As Sintered Mechanical Properties

Figures 38, 39 and 40 summarise the results of the limited work that has been carried out to investigate the factors controlling the mechanical properties of sintered iron-copper-tin alloys. The results obtained by Robins,[34] Figure 38, indicate a maximum strength at a copper-tin ratio of 60:40 and a total alloy content of 5%. The results obtained by Kothari,[35] Figure 39, indicate maximum strength at an alloy content of 10% using a 9:1 Cu:Sn ratio, Figure 39 also shows that superior properties are produced at this alloy level when using elemental copper and tin compared with a pre-alloyed bronze powder.

ii Alloying and Strengthening Mechanism

Published results[34] indicate that strengthening in this system is achieved at low sintering temperatures ~1000°C and short sintering times ~10 minutes. The mechanisms involved are not fully understood but it has been suggested that the presence of tin, in solid solution in iron, influences surface energy and hence the rate of self-diffusion

Fig. 38 Effect of simultaneous tin and copper additions in ratio 40:60 on the strength of sintered iron compacts[34]

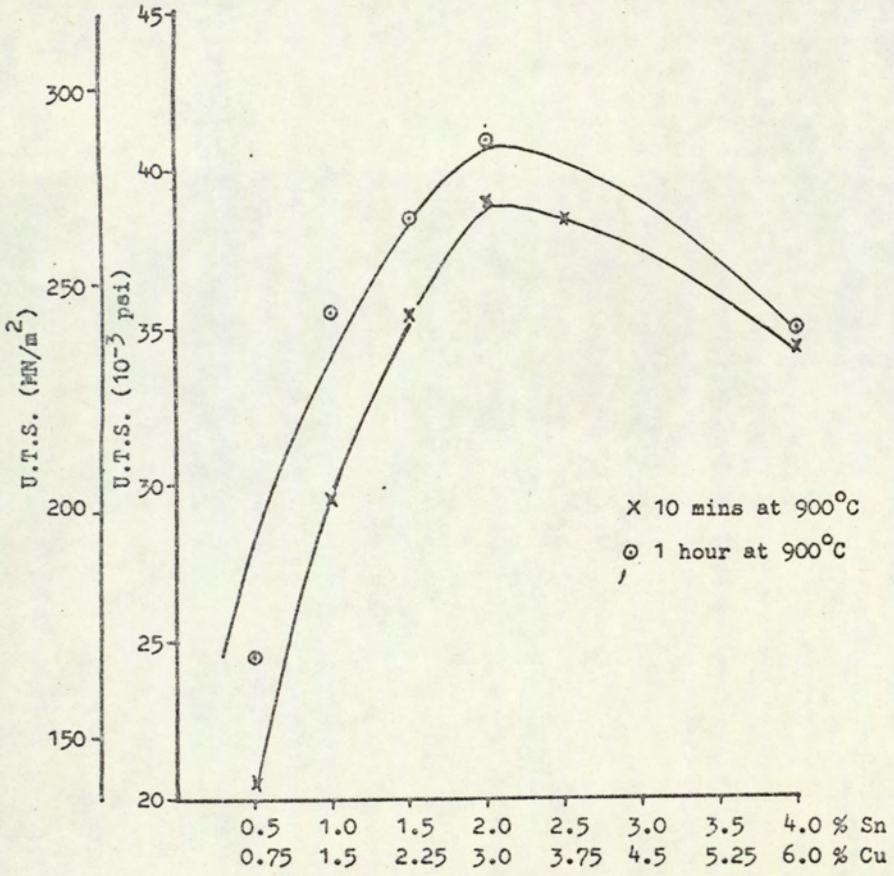


Fig. 39 Effect of elemental and pre-alloyed bronze on the strength of high density sintered iron base compacts[35]

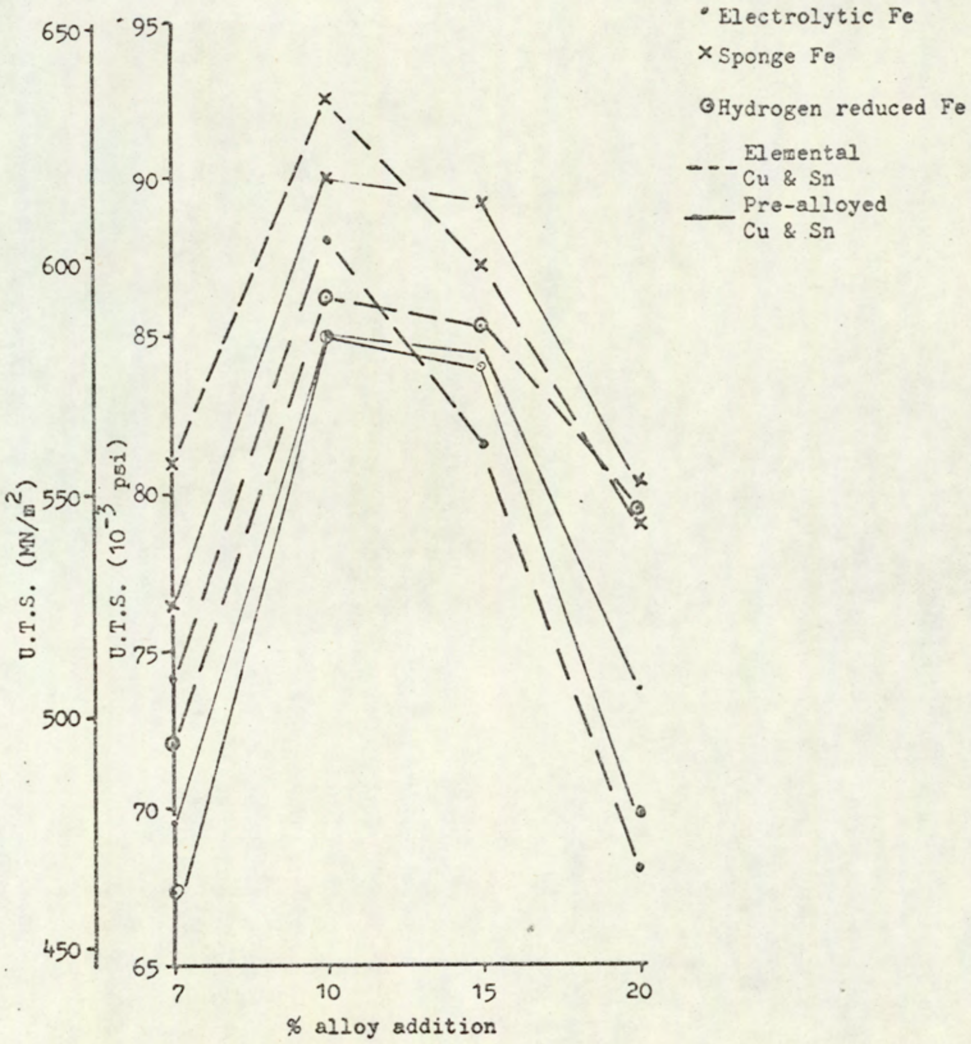


Fig. 40 Effect of sintering temperature on U.T.S. of iron-copper and iron-copper-tin alloys[34]

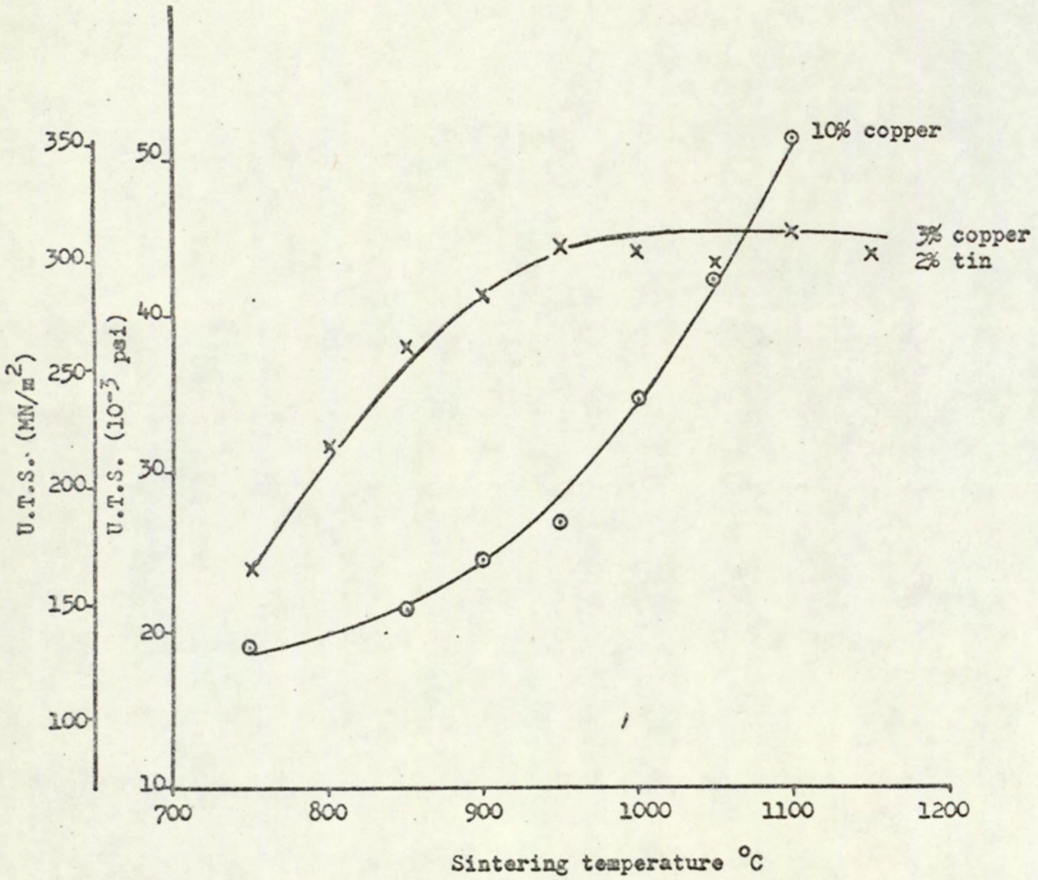
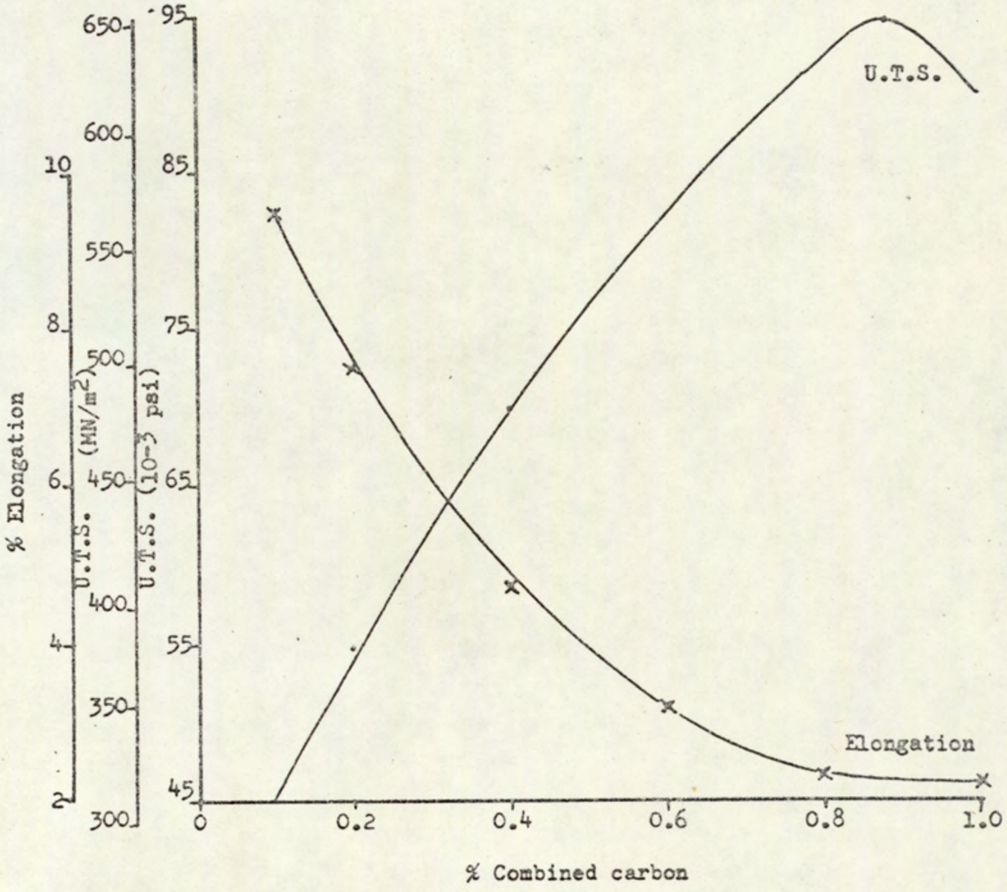


Fig. 41 Strength and ductility of as sintered Ancoloy S.D. as a function of combined carbon[37]



of iron.

The limited information concerning dimensional change indicates an initial expansion which decreases with increasing sintering time. The expansion of Fe/Cu/Sn is due to solution of Cu/Sn and the reduction in expansion at extended sintering time is due to increased shrinkage due to normal pore size reduction contracting the solution expansion effect.

f Systems Containing Manganese

Although manganese is widely used in the production of solid low alloy steels, its use in the manufacture of sintered alloy steels has not been widely reported and only two systems are reported in detail:

i Iron-copper-manganese-carbon, produced using pre-mixed elemental powders

ii Iron-nickel-manganese-carbon, produced using a partially pre-alloyed powder

i Iron-copper-manganese-carbon

As Sintered Mechanical Properties

The limited mechanical property data available for this system is summarised in Table 11 below.

Table 11

Tensile Strength of Various Fe-Cu-Mn-C Alloys

Composition (weight %)				Density (gm/cc)	UTS x 10^3 psi	(MN/m^2)
Cu	Mn	B	C			
4.4	2.4	.04	.4	6.70 6.20	75.3 51.2	(518) (352)
1.3	2.4	.04	.4	6.70 6.22	62.5 48.4	(430) (332)
4.4	4.4	.04	.4	6.40 5.94	36.9 31.3	(254) (215)
4.4	2.4	.16	.4	6.52 6.08	69.6 48.4	(478) (333)

Compacts sintered at 1150°C for 30 minutes

Production Technique and Strengthening Mechanism

Manganese is not normally used in the form of a pre-mixed elemental powder since it has a high affinity for oxygen resulting in a viscous, stable, oxide film which prevents diffusion of manganese into iron. The oxide film is stable even when sintering is carried out above the fusion point of manganese. However, it has been claimed[36] that if a fluxing agent such as a borate or boric acid is mixed with the elemental powders, this oxide film is stripped off at conventional sintering temperatures, thus facilitating diffusion. Strengthening is achieved in the as-sintered condition by the production of a solution strengthened structure containing cementite in the form of pearlite.

No information concerning the dimensional stability of this system was reported in the patent specification.

ii Iron-Nickel-Manganese-Carbon

As Sintered Mechanical Properties

This system has been prepared using a partially pre-alloyed powder, ie Hoganas - Ancoloy SD - 1.25% Mn .5% Ni, mixed with the desired quantity of graphite prior to pressing and sintering. Limited mechanical property data has been published, and this is summarised in Figure 41 which shows tensile strength and elongation as a function of combined carbon content.

Alloying Mechanism and Dimensional Stability

Alloying occurs by diffusion and strengthening is mainly due to the presence of carbon, although some increases are produced by the presence of nickel and manganese in solid solution in the iron. Since manganese produces a slight expansion during sintering, nickel is added in such a proportion that no dimensional change occurs during sintering under specified conditions.

g Systems Containing Molybdenum

i As Sintered Mechanical Properties

Several molybdenum bearing systems have been reported and the limited mechanical property data available is shown in Tables 12 and 13 below. These systems can be prepared using homogeneous or partially pre-alloyed powders, but the homogeneous pre-alloyed powder has a poor compressibility and produces inferior fatigue properties.

Table 12

Strength and Ductility of Molybdenum Bearing Systems Produced Using Homogeneous Pre-Alloyed Powder[38]

Composition	Sintered Density gm/cc	UTS x 10^3 psi (MN/m^2)	Elongation %
Fe - .60%C - 2.0%Ni - .50%Mo	6.5	71 (487.5)	5
	6.8	80 (550.0)	9
Fe - .60%C - 2%Cu - .50%Ni - .80%Mo	6.5	75 (515.0)	4
	6.8	85 (584.0)	6

Table 13

Comparison of Mechanical Properties of Molybdenum Bearing Systems Produced Using Homogeneous Pre-Alloyed Powder and Partially Pre-Alloyed Powder[37]

	Partially Pre-Alloyed Powder	Homogeneous Pre-Alloyed Powder
Composition	Cu 1.66% Ni 1.76% Mo .53% C .51%	Cu 1.51% Ni 1.87% Mo .72% C .53%
Density gm/cc	7.17	7.15
UTS x 10^3 psi (MN/m^2)	110 (756)	112 (770)
% Elongation	2.6	1.4
Fatigue limit x 10^3 psi (MN/m^2)	22 (137.5)	18 (123.8)
Compressibility at 60,000 psi compacting load (gm/cc)	6.6	6.0

ii Alloying and Strengthening Mechanisms

Alloying is achieved by diffusion and the major strengthening element is carbon. The maximum benefit from the alloying elements is obtained in the quenched and tempered condition and molybdenum is useful in reducing the susceptibility to temper embrittlement[38] and hence promotes ductility at high strength levels. When used in a homogeneous pre-alloyed powder molybdenum enhances the diffusion of carbon.[38] No information concerning the general effects of molybdenum on dimensional stability was reported in the literature reviewed.

h Iron-Phosphorus

The use of sintered iron-phosphorus alloys is not widespread since extensive shrinkage occurs during sintering due to the formation of a low melting point iron-phosphorus eutectic. However, a technique has recently been patented[39] which claims tensile strengths of ~56 x 10^3 psi (385 MN/m^2) with low shrinkage from an iron-phosphorus system. These properties are achieved by using a mixture of low phosphorus iron powder, ie $<$.03% P and a high phosphorus iron powder, ie up to 3% phosphorus. It has been suggested that the low shrinkage during sintering arises from the fact that no eutectic is formed and the phosphorus simply diffuses into the iron producing a solution strengthened structure.

3.3 Heat Treatment

Sintered steels have a similar potential for heat treatment as solid steels and there are basically two types of treatment that can be carried out:

a Case hardening

b Through hardening

3.3.1 Case Hardening

The object of case hardening is to increase surface hardness and wear resistance whilst retaining to a large extent as sintered mechanical properties.

This can be achieved by increasing the case carbon content to ~.8%, ie case carburising, or by introducing both carbon and nitrogen into surface layers, ie carbonitriding.

Case carburising of sintered parts presents many problems in the control of case depths. At densities below ~6.4 gm/cc virtually no control is achievable and complete through hardening occurs. Between 6.4-7.0 gm/cc case depth is highly dependent upon density and it does not become completely independent until density levels of 7.0-7.2 gm/cc are achieved.[8]

3.3.2 Through Hardening

The object of through hardening is not only to increase wear resistance but also to increase the strength of the whole section. Two basic through hardening techniques can be applied to iron base powder metallurgy components:

- a quench hardening of carbon bearing systems
- b precipitation hardening of iron-copper alloys

a Quench Hardening

i Problems associated with the porous nature of sintered steels

Quench hardening of sintered steels is carried out in a similar manner to that applied to solid steels, ie austenitisation, quench and temper. However, their porous nature introduces some problems:

Thermal Conductivity

Sintered steels have a much lower thermal conductivity than solid steels and hence in plain carbon steels severe quenching agents are required to achieve the critical cooling velocities necessary for the production of martensite. However, sintered steels also have a poor thermal shock resistance preventing the use of severe quenching agents and in practice plain carbon sintered steels can only be produced with a fine pearlitic as quenched structure. However, martersitic structures can be obtained in sintered steels by using alloying elements such as manganese and nickel which reduce the critical cooling velocity required for martensite formation, thus enabling quenching oils to be used.

$D = \frac{\text{Th-C}}{\rho \times \text{S.H.}}$,

Retention of Quenching Media

Since sintered steels are porous, they retain some of the quenchant used and hence components quenched in corrosive media require careful washing and drying. In this respect, the use of quenching oil can be advantageous since it can simultaneously serve as a corrosion protection treatment. However, oil absorption can present problems if components require tempering after quenching.

ii Effect on Mechanical Properties

In general, through hardening increases tensile strength and fatigue strength at the expense of ductility, Figures 42 and 43, but final mechanical properties are dependent upon tempering temperatures, Figure 44.

b Precipitation Hardening of Iron-Copper Alloys

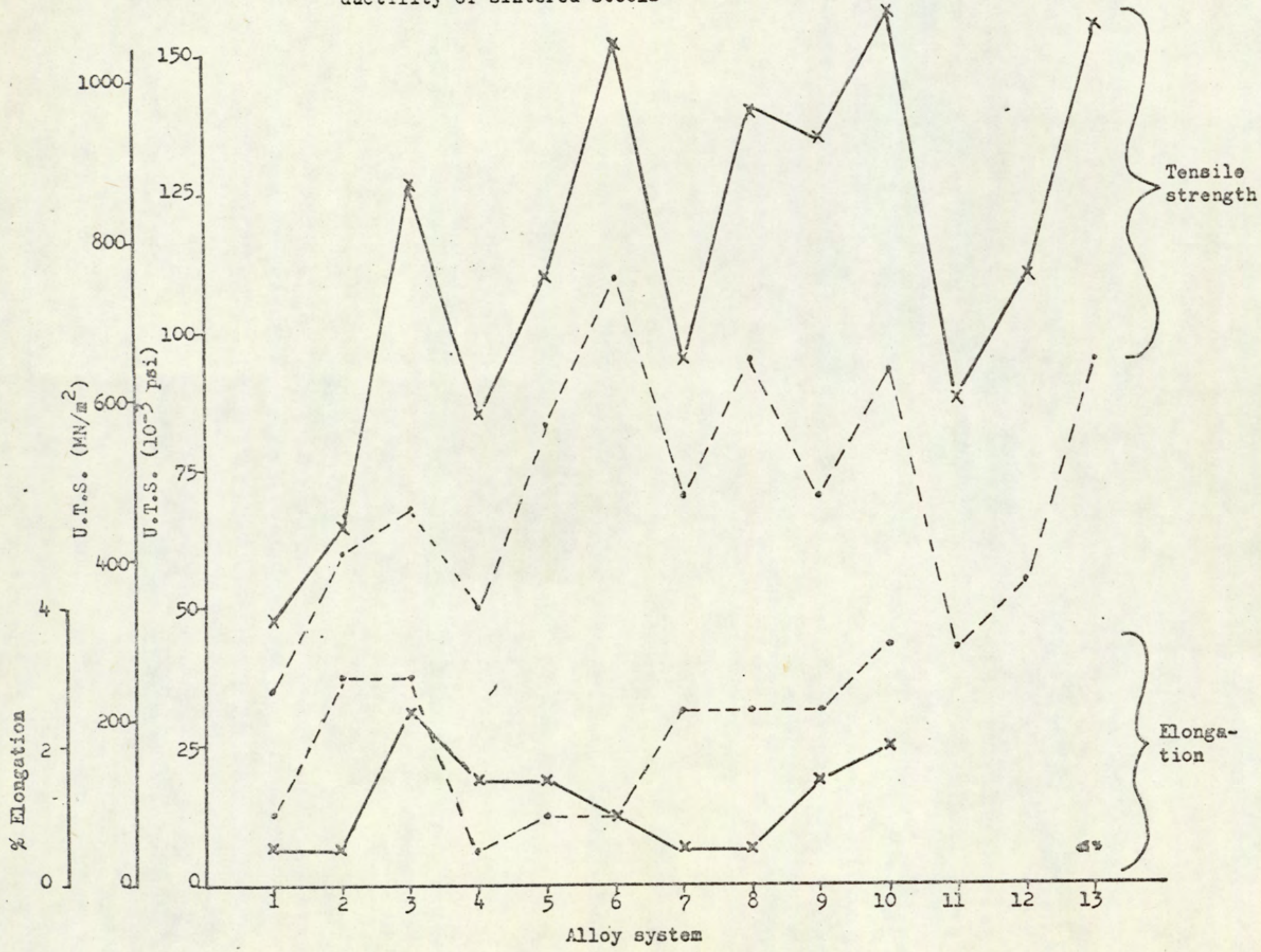
i Hardening Technique

The solubility of copper in α iron falls from $\sim$1.4% at 850°C to $\sim$.3% at room temperature, Figure 45, resulting in the precipitation of an iron/copper solid solution, ie ϵ which can, if its precipitation is closely controlled, improve strength. For maximum strengthening, alloys are quenched from $\sim$980°C, producing a supersaturated solid solution and then reheated, ie aged, under controlled conditions thus producing a fine grain boundary precipitate.

ii Effect on Mechanical Properties

Precipitation hardening of iron-copper alloys has been examined by Harrison and Dixon[40] and Figures 46 and 47 show optimum copper content of 3-4% and an optimum ageing temperature of $\sim$500°C. Figure 48 summarises the results published in the Hoganas Handbook and they indicate maximum strengthening at 5% copper.

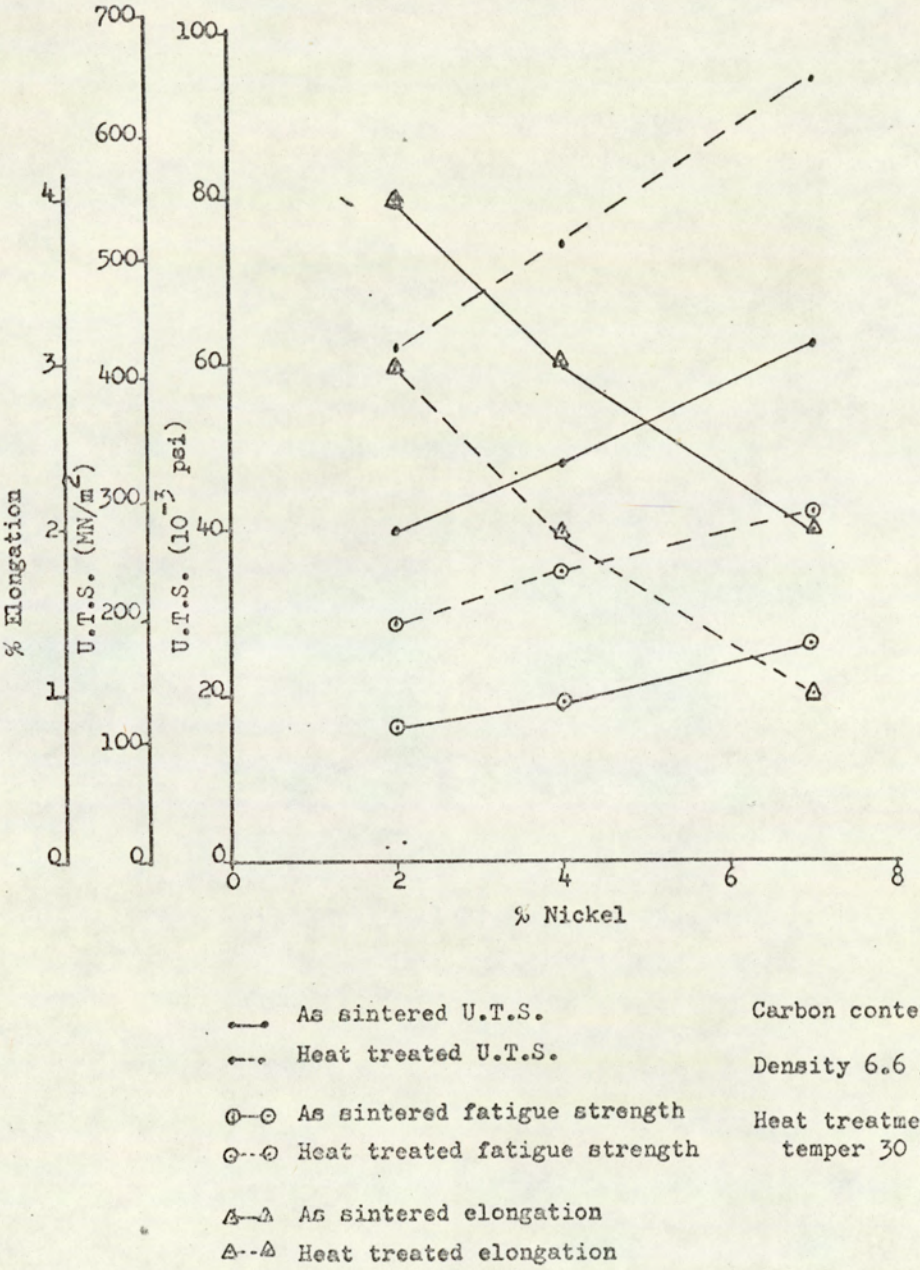
ductility of sintered steels



Alloy systems

1) 99% iron-1% carbon Density 6.1 - 6.5 gms/cc
2) 99% iron-1% carbon Density 7.0 gms/cc
3) 99% iron-1% carbon Density 7.3 gms/cc
4) Iron-7% copper-1% carbon Density 5.8-6.2 gms/cc
5) Iron-7% copper-1% carbon Density 6.8 gms/cc
6) Iron-20% copper-1% carbon Density 7.1 gms/cc
7) Iron-1.5 nickel-.5% molybdenum-0.6% carbon Density 6.8 gms/cc
8) Iron-1.5% nickel-.5% molybdenum-0.6% carbon Density 7.2 gms/cc
9) Iron-2% copper-1% carbon Density 6.8 gms/cc
10) Iron-2% copper-1% carbon Density 7.2 gms/cc
11) Iron-12% chromium-.15% carbon Density 5.9 gms/cc
12) Iron-12% chromium-.15% carbon Density 6.4 gms/cc
13) Iron-1.25% manganese-0.5% nickel-.8% carbon

Fig. 43 Effect of heat treatment on mechanical properties of sintered iron-nickel-carbon alloys[31]

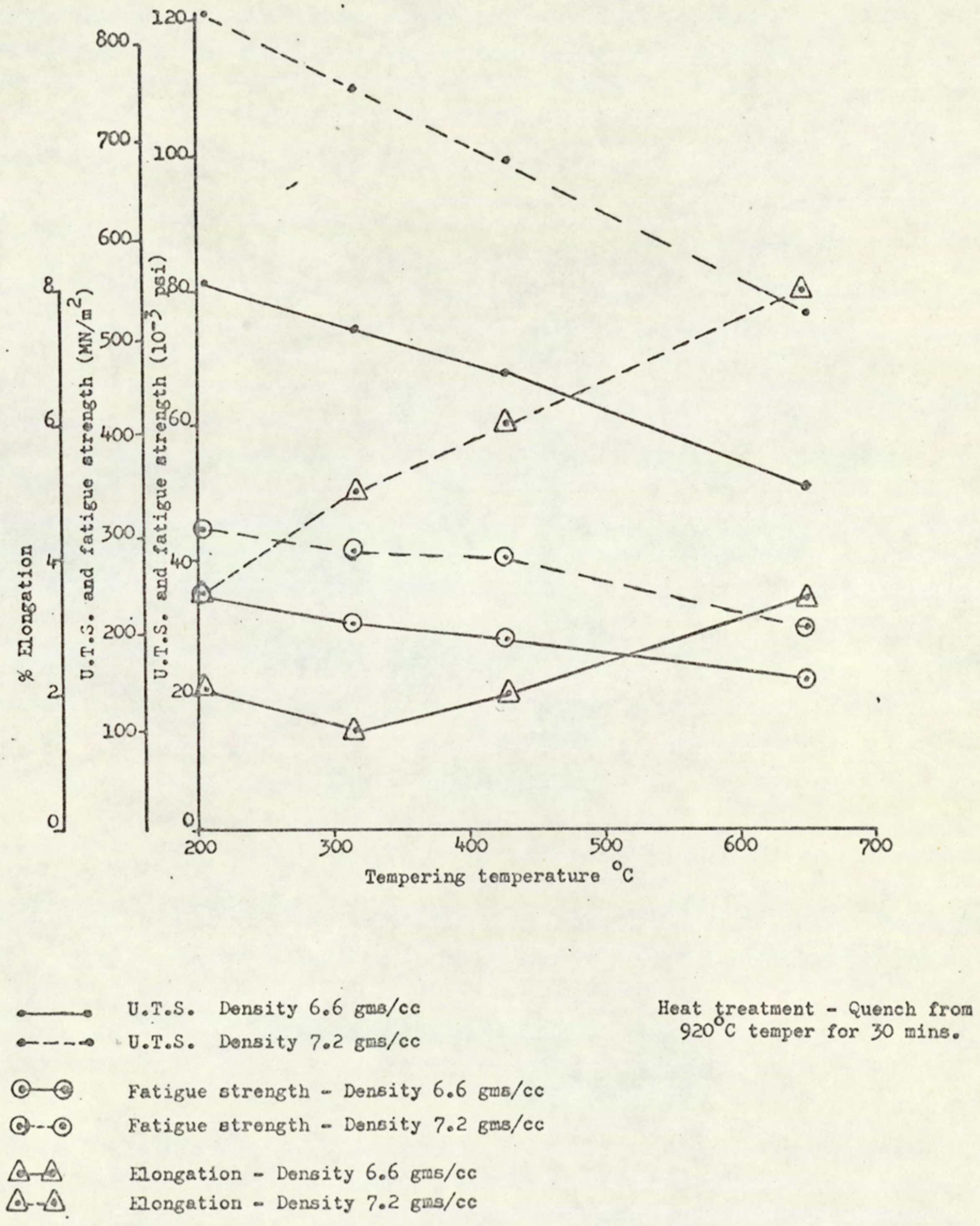


Carbon content - 0.5%

Density 6.6 gms/cc

Heat treatment - quench from 920°C
temper 30 mins. at 250°C

Fig. 44 Effect of tempering temperature on mechanical properties of oil quenched iron-4% nickel-0.4% carbon alloys[31]



Heat treatment - Quench from 920°C temper for 30 mins.

Fig. 45 Iron rich end of iron-copper equilibrium diagram[40]

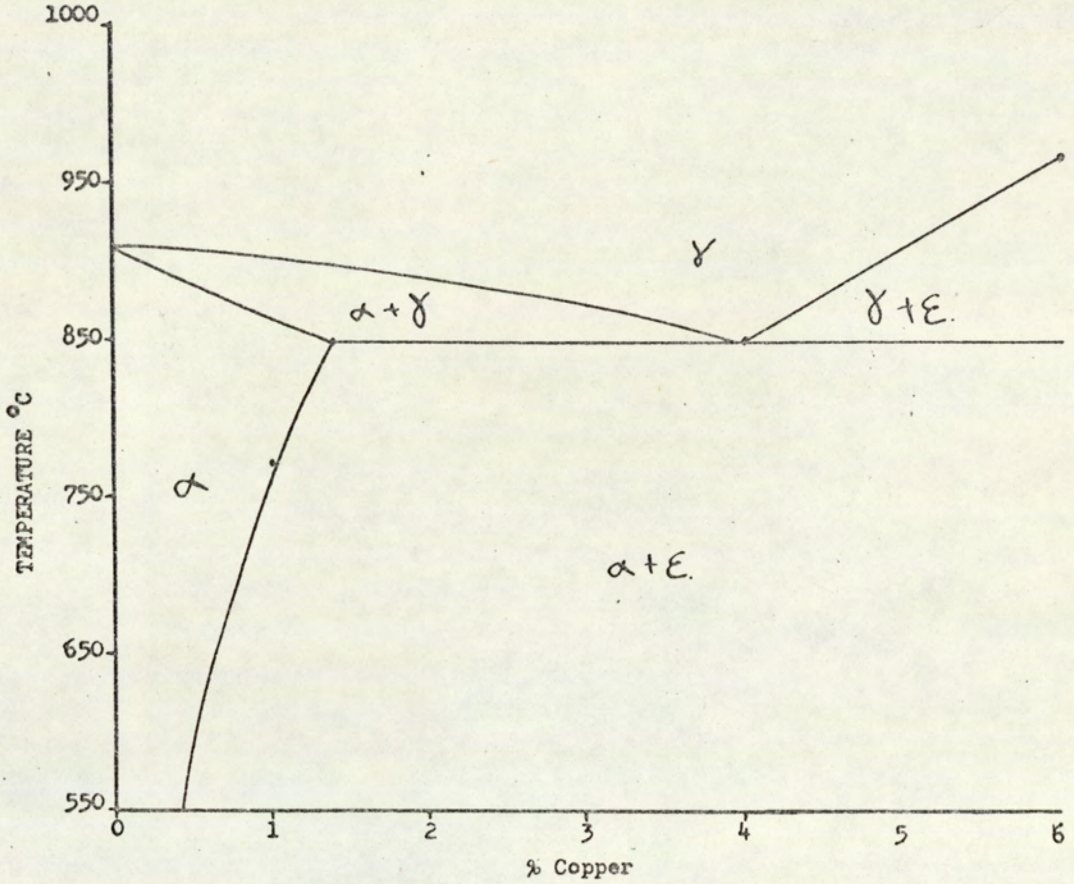
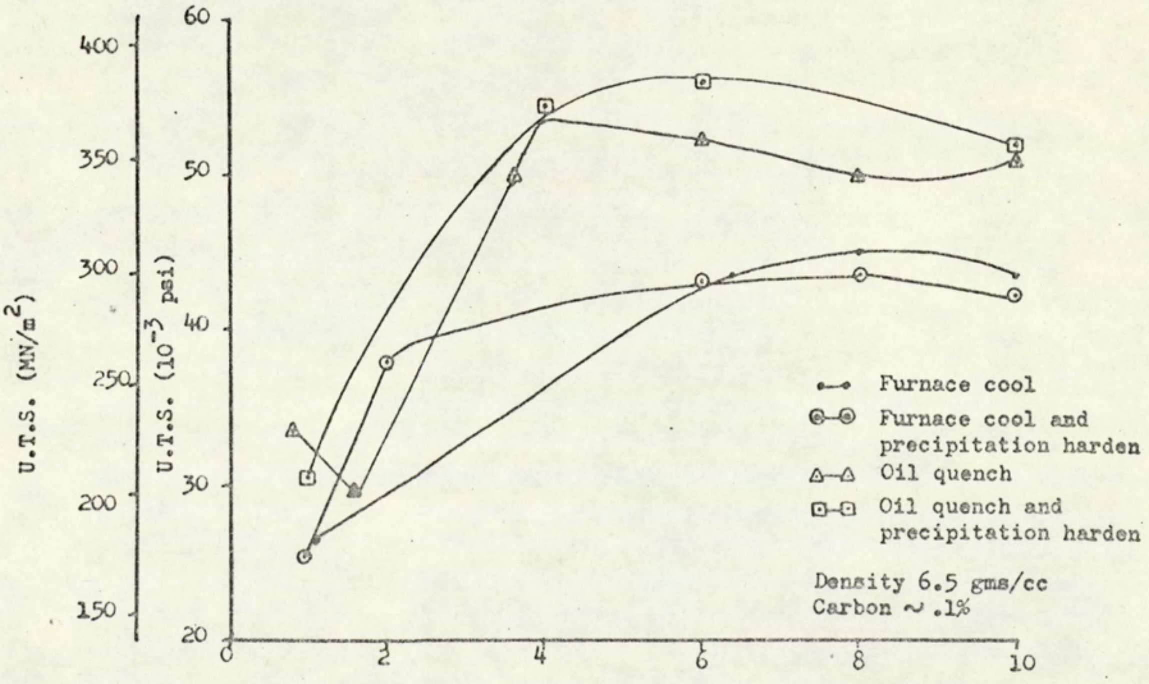


Fig. 46 Effect of copper content on the response of sintered iron-copper-carbon alloys to various age hardening treatments[40]



Density 6.5 gms/cc
Carbon ~ .1%

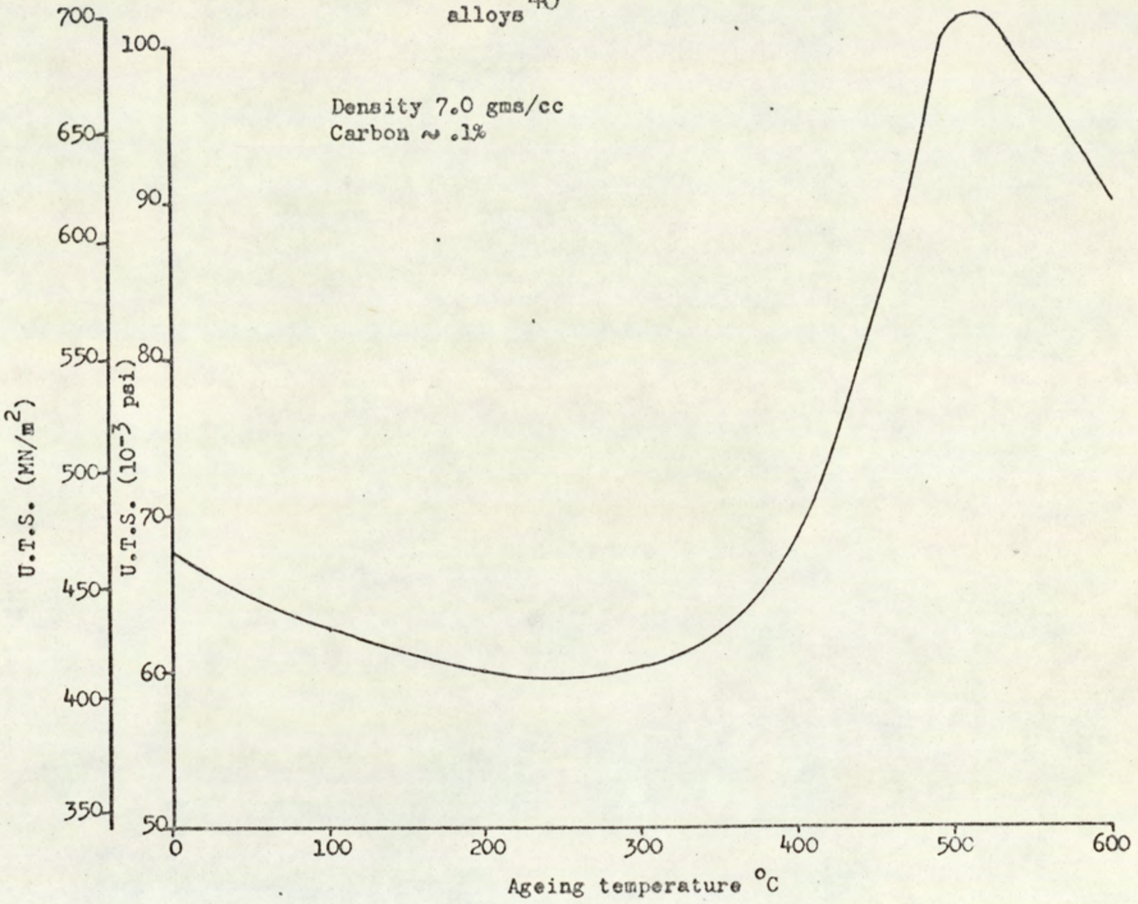


Fig. 47 Effect of ageing temperature on the strength of sintered iron-copper-carbon alloys[40]

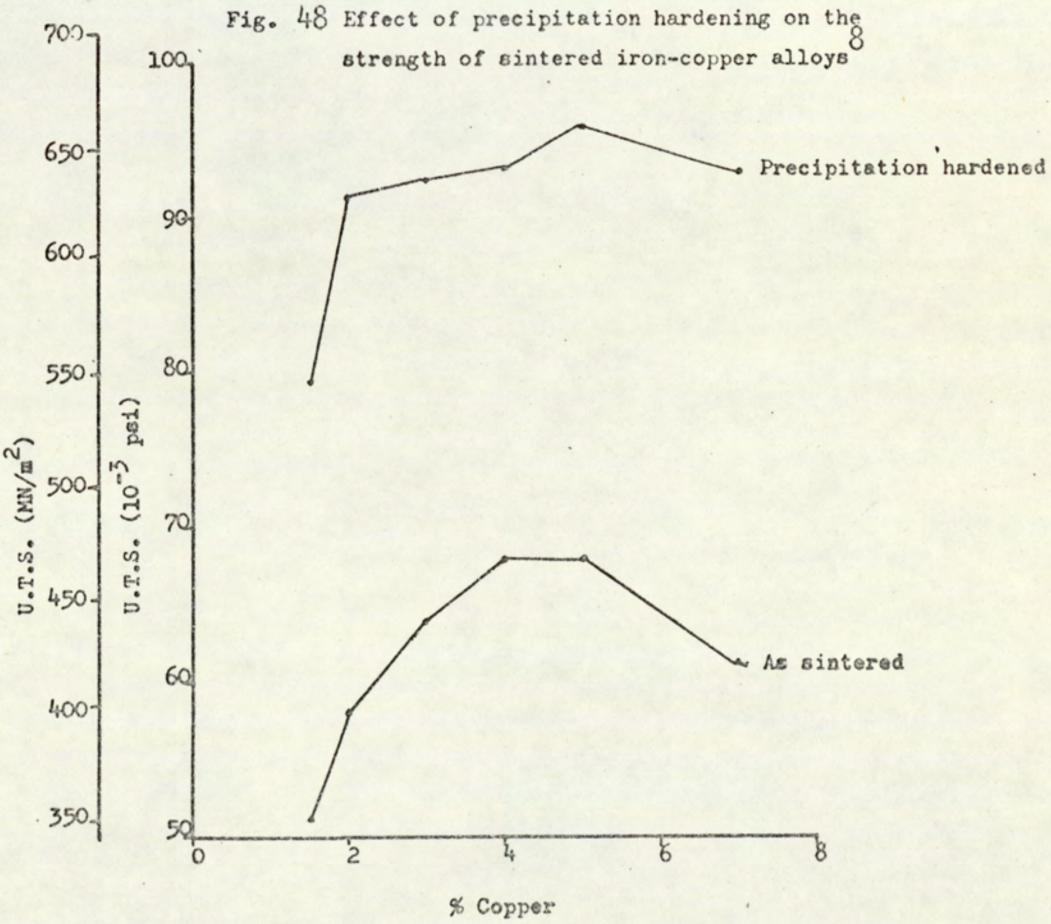


Fig. 48 Effect of precipitation hardening on the strength of sintered iron-copper alloys[8]

3.4 Summary

a The mechanical strength, ductility and fatigue strength of iron base powder metallurgy components increase with increasing density.

b A variety of techniques can be used for increasing density but many are limited to materials which are difficult to compact conventionally. Techniques such as double pressing, repressing and infiltration are useful but they are expensive and at the present time there is a large demand within the component industry for cheaper high compressibility powders.

c A number of techniques are available for producing iron base alloy components but the majority are produced using pre-mixed elemental powders.

d The introduction of alloying elements generally increases tensile strength and fatigue strength at the expense of ductility. The main strengthening element is carbon and this, together with copper and nickel, is the most widely used alloying element.

e The addition of alloying elements has a significant effect on the dimensional stability of iron base components during sintering. In the iron-nickel copper-carbon system it is possible, by careful control of the alloy content, to produce expansion, shrinkage or no dimensional change during sintering.

f Sintered steels have a similar potential for heat treatment as solid steels but their porous nature presents some problems. As in solid steels, nickel and manganese are widely used to increase hardenability.

4 COST ANALYSIS

4.1 Introduction

Developments in powder metallurgy technology outlined in Section 1 of this thesis, have resulted in direct competition between sintered components and components produced by more conventional techniques, eg precision casting, machining, forging, etc. In some limited applications, sintered components also compete with plastic parts. A detailed technico-economic comparison between the powder metallurgy process and other numerous competitive processes would require a relatively lengthy review which would be inappropriate in this thesis. However, many of the major advantages of the powder metallurgy process can be outlined by considering a cost comparison such as the one presented in this section.

4.2 Component

Details of the component, a latch plate bush, are given in Figures 49, 50. Originally, the bush was machined from mild steel bar stock on a capstan lathe and hence costs involved using this technique are compared with those incurred by using the powder metallurgy route.

4.3 Machining Cost

A breakdown of the estimated costs for producing the bush by machining from mild steel bar stock is given in Table 14

Figure 49

Latch Plate Bush

Figure 50

Dimensions of Latch Plate Bush

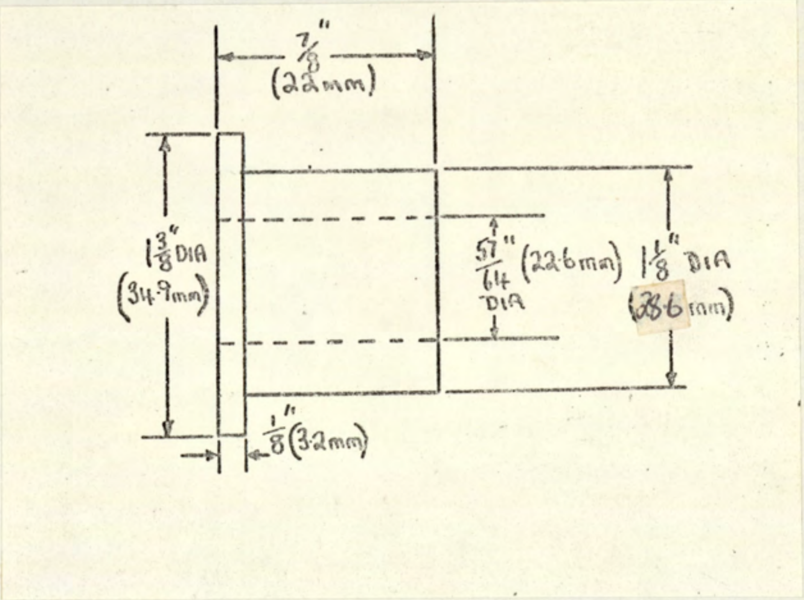


Table 14

Machining Costs

Scrap?

		Cost (p each)
1	Materials	2.170
2	Labour	4.711
3	Power	.100
4	Depreciation	.510
5	Overheads	5.889
6	Tools	.106
		13.486
	Total cost per piece -	13.5p

The basic assumptions used in calculating these costs are outlined below:

1 Batch size - 25 000

2 Total weight of bar stock required for each part - 222 gm

3 Labour cost - 90p/hour (including 14% allowance for national insurance, holiday pay, etc)

4 Raw material - $1\frac{3}{8}$" diameter black bar @ £81/ton

5 Capital cost of equipment (Ward Capstan Lathe) - £2803.55 depreciated at $6\frac{2}{3}$% pa

6 Power rating - 5.5 hp

7 Tools - Parting off tools £0.75 each 2000 parts/tool
Turning tool £1.25 each 25×10^3 parts/tool
Drills £0.5 each 800 parts/tool

8 1 shift system - 1920 hours/year

9 Overheads - 125% of direct labour

A detailed breakdown of the various operations and times involved in machining is given in Table 15.

Table 15

Breakdown of Machining Operations

(Works Study Department - Stanton & Staveley)

Operation	rpm	Feed /rev	Time (Minutes)
Release collet			.02
Bar to stop			.09
Close collet			.02
Index turret for R/bcx			.04
Turn $1\frac{1}{8}$" diameter	150	.010	.56
Index turret + change speed			.08
Centre drill			.09
Index turret for drill			.04
Drill 57/64" dia hole $\frac{7}{8}$" deep	250	.007	.56
Turn $1\frac{3}{8}$" dia change speed	150	.010	.29
Index tool post			.04
Clear shoulder	150	Hand	.08
Part of bush (rear tool post)	150	.005	.45
Face bar for next bush	150	Hand	.25
Actual time			2.61
Contingencies			.522
Total Time			3.132
Setting up for machining			40.000

4.4 Powder Metallurgy Costs

The estimated costs involved in producing the bush as a sintered component are outlined in Table 16.

Table 16

Sintering Costs

		Cost (p each)	
1	Materials	.502	
2	Labour	.187	
3	Depreciation	.159	
4	Power	.038	
5	Overheads	.414	540 — 250%
6	Tools	.540	
		1.840	1.84

Total cost per piece - 1.8p

Since these costs have been calculated using a system developed for a production unit, a detailed breakdown of the costing procedure used cannot be presented. However, some limited information is given below:

a Batch size - 25 000

b Material - iron, 1% C

c Density - ~6.4 gm/cc

d Compacting press - Stokes 100 ton 280G

e Compacting rate - 1000/hour

f Sintering - 18" furnace using endothermic atmosphere

g Tool cost - ~£540 and tool life - ~100 x 10^3 parts

h One shift system

4.5 Discussion of Costs

a Materials

Although the basic cost of the iron powder mix used in the sintering process is considerably greater than that of mild steel bar stock, viz 5.28 p/lb compared with 3.62 p/lb, the actual material cost per component is much lower. This is a result of

- i a high material utilisation in the powder metallurgy route, ie ~95% compared with ~28% by machining
- ii a density of ~82% theoretical in the sintered component, reducing the weight of material required and hence the material cost

The powder mix used in producing this part as a sintered component, viz Fe/1%C at a density of 6.4 gm/cc, produces a UTS of ~14 tsi (216 MN/m^2), which is more than adequate for the prevailing service conditions. However, if a strength equivalent to that of mild steel is required, a more expensive powder mix, eg Fe 5%Cu 1%C, is required, resulting in a material cost/component of ~.695p. Even greater strengths ~38 tsi (586 MN/m^2), require the use of a partially pre-alloyed powder, resulting in a material cost/component of ~.912p. However, all these costs are considerably less than that incurred during machining from mild steel bar stock.

b Labour

The production of the bush by the powder metallurgy route involves the use of highly sophisticated, automated equipment which produces parts more rapidly than the machining set up chosen, viz 1000/hour compared with 19/hour, and does not require the constant manning of the machining set up. The nett result is a much lower labour cost/unit cost via the powder metallurgy route which in the comparison outlined amounts to a reduction of 4.524p/piece.

c Depreciation and Power

Although the total capital investment and power consumption is much greater using the powder metallurgy route, depreciation and power costs per unit cost are considerably less than those incurred during machining. This reduced cost is again directly related to the superior production rates of the powder metallurgy process.

d Overheads

Since the machining route has a high direct labour content and a low production rate, overhead costs/unit cost are considerably greater than those incurred using the powdeter metallurgy route.

e Tools

Powder metallurgy compacting tools are expensive since they are made to extremely close tolerances, viz ±.0001" (±.003 mm), but relatively good tool lives result in tool costs/unit cost being a minor consideration provided orders are large enough to cover the life of the tool. In the cost comparison, it was assumed that orders were sufficient to cover a conservative tool life of 100×10^3 parts, but this could possibly be extended to $250 - 300 \times 10^3$ by regrinding punch faces which, provided orders are large enough to cover this extra tool life, would bring the tool cost/unit cost, via the powder metallurgy route, to the same order as that incurred during machining.

4.6 Summary

In this cost analysis, the reduced manufacturing costs by the powder metallurgy route are a direct result of high production rates, minimum labour content and good material utilisation. The comparison also shows how the apparent disadvantage of a high raw material cost is greatly reduced by a material utilisation approaching 95%.

The high cost of powder metallurgy tools can be off-set by good tool lives, but production orders must be large enough to cover this life so that tool costs/unit cost can be kept to a minimum. In order to minimise tool changes and hence reduce press "down time", production batches below $\sim 10 - 20 \times 10^3$ parts per annum are not usually considered economically viable. However, much depends on the shape, size and properties required from the final part and in many cases, especially those where extensive machining required during production by more conventional routes can be eliminated, smaller runs can be economically viable.

The limitations imposed by the powder metallurgy process on component shape are not highlighted in the cost comparison. Features such as undercuts, reverse tapers, threads, holes normal to the pressing direction, etc, cannot be produced directly during powder compaction and if required the components must be machined after sintering. Although this increases production costs, in many cases the savings produced using the powder metallurgy route more than compensate for this extra cost.

5 EXPERIMENTAL WORK

5.1 Introduction

The main considerations in the production of sintered materials, excluding cost, are the achievement of a specified level of mechanical and physical properties, tensile strength in particular, whilst maintaining a minimum and known dimensional change during sintering. It has already been shown that the factors which have a significant effect on mechanical strength are:

i density
ii alloy composition
iii heat treatment

and of these, i and ii also have a significant effect on dimensional change during sintering under specified sintering conditions, viz time, temperature and atmosphere. Hence, the variables chosen for analysis in this study are alloy composition and density and the parameters to be measured are mechanical strength and dimensional change during sintering. Although heat treatment has a major beneficial effect on mechanical strength, it has not been included since:

a For economic reasons, the majority of sintered materials are used in the as-sintered condition b The porous nature of sintered materials presents some major problems in maintaining close dimensional tolerances and avoiding distortion during thermal treatments, and thus in practice heat treatment is usually avoided whenever possible

The majority of iron base alloys are produced from pre-mixed elemental powders and the most widely used alloying elements are carbon, copper and nickel. The qualitative dependence of the mechanical strength and dimensional stability on each of these alloying elements and the as-pressed density, when using relatively simple systems, viz Fe/C Fe/Cu, Fe/Cu/C, Fe/Ni and Fe/Ni/C, has been reasonably well reported in published literature. However, few attempts have been made to quantitatively analyse the effect of alloy composition and density on mechanical strength and dimensional stability. Hence, the object of this investigation is to examine the effect of varying density and alloy composition in the iron-copper-nickel-carbon system and to:

i Establish which variables and combinations of variables have a significant effect on mechanical strength and dimensional change during sintering, and

ii Produce a quantitative assessment of the magnitude of these significant effects

5.2 Experimental Design

a Comparison of Statistical (eg Factorial and Classical Experimentation

Statistically designed experiments are gradually becoming more widely used in technological research, particularly in multi-factor (variable) systems. One of the most useful techniques is the factorial design, and a brief comparison of this technique and the classical type of experimentation is given below:

i Classical Experimentation

The traditional or classical method of investigating the effect of a number of factors on one or more measurable properties is to vary one factor at a time whilst holding the remainder constant. Although this is useful in indicating the general effects of each variable, it can be extremely time consuming and costly, since a large number of experiments are usually required when dealing with a number of factors, eg in an investigation involving n factors at x levels, a total of x^n experiments are required to cover all combinations, whereas in one involving n + 1 factors, a total of x^{n+1} experiments are required. A further disadvantage is that there is no indication of what happens when more than one factor at a time varies, which is usually the case in most industrial situations, nor is there any assessment of interactions between factors.

ii Factorial Experimentation

This technique involves allowing all factors to vary simultaneously in a planned and controlled manner. The main advantages of the factorial system are:

- the maximum amount of information arises from the minimum number of experiments,
- detailed information is obtained about interactions between the factors

These advantages are better outlined by considering the following hypothetical experiment which is based on the examples reviewed by Duckworth[41] and Brownlee.[42] Consider a system in which we wish to investigate the effect of varying three factors, A, B, and C, from one level, A_1, B_1, C_1, to a second level, A_2, B_2, C_2, on a dependent variable x.

Classical experimentation involves initially a control experiment using level $A_1\ B_1\ C_1$ producing a value of x denoted by $(A_1\ B_1\ C_1)x$. This is then followed by increasing A to A_2 keeping B and C constant, resulting in $(A_2\ B_1\ C_1)x$. Factor B is then increased to B_2, keeping A and C constant, resulting in $(A_1\ B_2\ C_1)x$, and finally factor C is increased to C_2, keeping A and B constant, resulting in $(A_1\ B_1\ C_2)x$. Hence, the effect of increasing each variable can be denoted as shown below:

1 effect of increasing A = $(A_2\ B_1\ C_1)x - (A_1\ B_1\ C_1)x$

2 effect of increasing B = $(A_1\ B_2\ C_1)x - (A_1\ B_1\ C_1)x$

3 effect of increasing C = $(A_1\ B_1\ C_2)x - (A_1\ B_1\ C_1)x$

Clearly, these experiments must be repeated at least once in order to obtain a reliable estimate of experimental error. Without this, it is not possible to say whether the effect is real or simply due to errors in sampling, measurement, etc. Hence, a minimum of eight experiments is required and to obtain the main effects the mean of two observations is compared with the mean of two other observations. No information concerning interactions between factors is available and hence it is not possible to predict any difference in increasing one factor at different levels of the other two factors.

A factorially designed investigation of the above situation involves carrying out eight separate experiments in order to cover all possible combinations of A, B, and C, resulting in eight values of x as shown below:

Experiment	Factors (Variable)			Result
	A	B	C	
1	A_1	B_1	C_1	x_1
2	A_2	B_1	C_1	x_2
3	A_1	B_2	C_1	x_3
4	A_2	B_2	C_1	x_4
5	A_1	B_1	C_2	x_5
6	A_2	B_1	C_2	x_6
7	A_1	B_2	C_2	x_7
8	A_2	B_2	C_2	x_8

The main effects are then given by the following:

i Main effect of increasing A =

$$\frac{(x_2 + x_4 + x_6 + x_8)}{4} - \frac{(x_1 + x_3 + x_5 + x_7)}{4}$$

ii main effect of increasing B =

$$\frac{(x_3 + x_4 + x_7 + x_8)}{4} - \frac{(x_1 + x_2 + x_5 + x_6)}{4}$$

iii main effect of increasing C =

$$\frac{(x_5 + x_6 + x_7 + x_8)}{4} - \frac{(x_1 + x_2 + x_3 + x_4)}{4}$$

From this, it can be seen that the main effects, viz effect of increasing A, B, and C, are obtained by comparing the average of four experiments with the average of four other experiments. In the classical design, these effects were obtained by comparing the average of two experiments with the average of two other experiments. Hence, the factorial is twice as accurate as the classical. In order to obtain the same level of accuracy in the classical design, sixteen separate experiments are required, viz twice as many as in the factorial.

It is quite possible that the effect of varying one of the factors, eg A at the low levels of B and C, is different from the effect of varying A at the high levels of B and C. Such an effect is known as an interaction and can be readily detected and assessed using the factorial approach, Table 17. However, interactions are rarely discovered using the classical approach unless a large number of experiments is used, and even then it is possible that some interactions may be missed.

Table 17

Two Factor Interactions

Interaction		Result	
1 Interaction between A and B		Level of B	
		B_1	B_2
Level of A	A_1	$\frac{x_1 + x_5}{2}$	$\frac{x_3 + x_2}{2}$
	A_2	$\frac{x_2 + x_6}{2}$	$\frac{x_4 + x_8}{2}$
2 Interaction between A and C		Level of C	
		C_1	C_2
Level of A	A_1	$\frac{x_1 + x_3}{2}$	$\frac{x_2 + x_4}{2}$
	A_2	$\frac{x_5 + x_7}{2}$	$\frac{x_6 + x_8}{2}$

The statistical significance of the main effects and interactions deduced from the factorial design can be readily established using either simple or discriminating significance tests. The use of duplicated factorial experiments considerably increases the accuracy of the results which often outweighs the disadvantage of increased experimentation, particularly when there is little or no published information concerning the system under investigation.

Although the analysis of the hypothetical experiment outlined earlier is relatively simple, a complete analysis of all main effects and interactions of even a simple factorial design such as this is time consuming. Hence, the analysis of factorial experiments is usually carried out using a computer which is essential when dealing with complex systems, viz more factors and/or more levels.

b Application of Factorial Design to Experimental Objectives

The system chosen for investigation involves varying four factors:

- i as pressed density
- ii % C
- iii % Cu
- iv % Ni

and measuring the resultant mechanical strength and dimensional change during sintering. The limited published information concerning this system indicates that interaction between these factors could have a significant effect on mechanical strength and dimensional stability, and hence the necessity for a factorial design. The range of alloy additions chosen, viz:

% C	.3	.6	.9
% Cu	2	5	8
% Ni	2	5	8

are designed to cover the range of alloy additions used commercially, and the three levels are used in order to detect any nonlinearity. The three levels of density chosen, viz 80%, 84% and 88% of the theoretical wrought density, cover the general density range employed in the production of sintered components using a basic sponge iron powder, viz Hoganas NC 100 24. Hence the system consists of a full factorial examination of four factors at three levels, resulting in the necessity for 3^4, viz 81 experiments. Accuracy can be further increased by carrying out a complete duplicate, which is necessary since little published information is available, thus increasing the total number of experiments to 162.

The majority of factorial experiments discussed in published literature involve the use of a varying number of factors at two levels only and the presence of a third level greatly increases the complexity of the analysis. Although text books outline the deduction of both the magnitude and significance of the results from factorial designs for variables at two levels, no details of the analysis of variables at three levels is given, and computer programmes readily available are only designed to carry out an analysis of significance, usually an analysis of variance. Hence, this investigation consists of a duplicated factorial design of four factors at three levels from which the magnitude of main effects and interactions shown to be significant, using an F test, are deduced using multiple-regression analysis.

5.3 Design of Test Piece

The dimensions of the test piece used to determine mechanical strength and dimensional change during sintering are shown in Figure 51. This is not a standard test piece suitable for the determination of UTS and would require extensive and costly machining in order to produce a suitable shape. However, mechanical strength in terms of transverse rupture strength, was considered to be more meaningful in this type of project, and can be quickly and easily determined using a three point bend test. Transverse rupture strength is widely used in the mechanical property specifications of sintered materials, a factor which also influenced the choice.

The determination of dimensional change simply involves measuring thickness, width and length, using a micrometer.

5.4 Experimental Procedure

a Powders Used

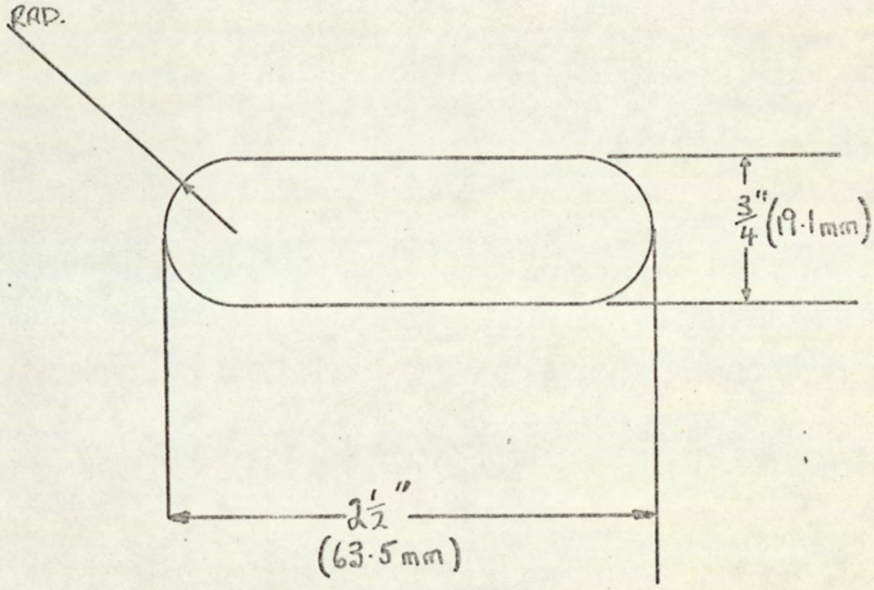
The iron powder used, Hoganas NC 100 24, is one of the most widely used powders for producing sintered, iron base, alloys from pre-mixed elemental powders. The type of copper, nickel and graphite powder used is listed below:

Copper - 100 mesh electrolytic -
supplied by Berk Limited

Nickel - Carbonyl type 123 -
supplied by International Nickel

Graphite - OA Grade -
supplied by T. S. Wilson & Co.

FIG. 51 DIMENSIONS OF TEST PIECE



APPROX. SURFACE AREA - 2 s.q. ins.

b Blending

The powders were mixed for 40 minutes in batches of $1\frac{1}{2}$ kg to the desired compression, which is given in Table 18 The container used was a glass jar attached to the driving shaft of a double cone blender. One per cent by weight of zinc stearate (Fissons technical grade) was added to all powder mixes.

Table 18

Composition of Powder Mixes

Powder Mix	Composition	Powder Mix	Composition
1	Fe .3%C 2%Cu 2%Ni	15	Fe .9%C 5%Cu 5%Ni
2	Fe .6%C 2%Cu 2%Ni	16	Fe .3%C 8%Cu 5%Ni
3	Fe .9%C 2%Cu 2%Ni	17	Fe .6%C 8%Cu 5%Ni
4	Fe .3%C 5%Cu 2%Ni	18	Fe .9%C 8%Cu 5%Ni
5	Fe .6%C 5%Cu 2%Ni	19	Fe .3%C 2%Cu 8%Ni
6	Fe .9%C 5%Cu 2%Ni	20	Fe .6%C 2%Cu 8%Ni
7	Fe .3%C 8%Cu 2%Ni	21	Fe .9%C 2%Cu 8%Ni
8	Fe .6%C 8%Cu 2%Ni	22	Fe .3%C 5%Cu 8%Ni
9	Fe .9%C 8%Cu 2%Ni	23	Fe .6%C 5%Cu 8%Ni
10	Fe .3%C 2%Cu 5%Ni	24	Fe .9%C 5%Cu 8%Ni
11	Fe .6%C 2%Cu 5%Ni	25	Fe .3%C 8%Cu 8%Ni
12	Fe .9%C 2%Cu 5%Ni	26	Fe .6%C 8%Cu 8%Ni
13	Fe .3%C 5%Cu 5%Ni	27	Fe .9%C 8%Cu 8%Ni
14	Fe .6%C 5%Cu 5%Ni		

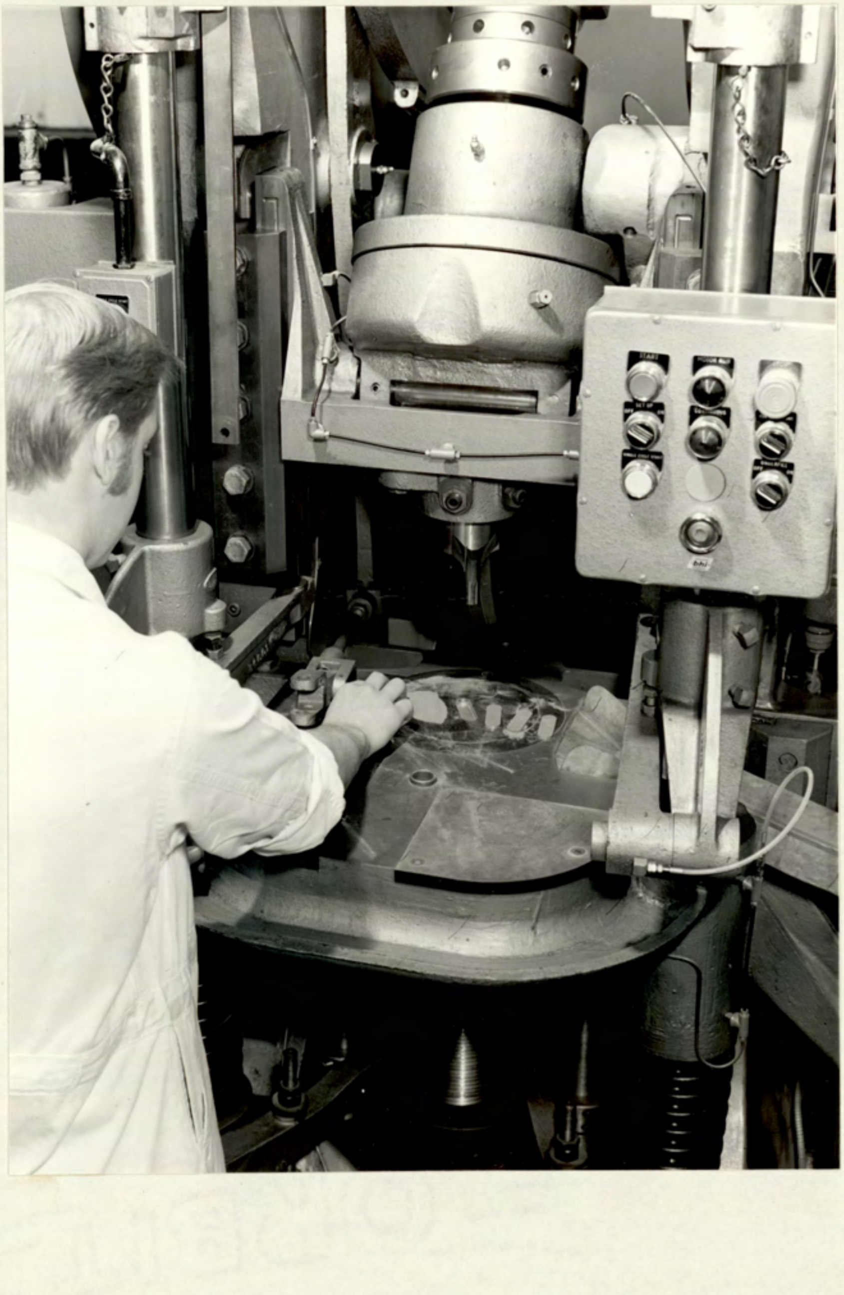
c Compaction

Five test pieces, $\frac{1}{8}$" thick, were compacted at each of the desired levels, viz 80%, 84% and 88% theoretical, from each of the above powder mixes using a Stokes 100 ton 280G press, Figure 52. From each batch of five test pieces, two were measured, viz thickness h, breadth b, length l, and numbered prior to sintering. A green density check was carried out using one further test piece leaving two spares in case of damage prior to sintering.

Figure 52

Compaction of Test Pieces on

Stokes 280 G Press



d Sintering

The test pieces were sintered in an FHD 6" continuous mesh belt furnace for half an hour at 1120°C using endothermic atmosphere. Since three different carbon levels were used, sintering was carried out in three batches and the dew-point of the endothermic atmosphere was adjusted to provide a carbon potential equivalent to each carbon level, viz:

.3%C - dew point required - +10 to +16°C
.6%C - dew point required - 0 to +4°C
.9%C - dew point required - -7 to -5°C

After sintering, all the test pieces were measured and the percentage change in dimensions was calculated. A check was also made on the carbon content of selected samples from each of the three batches.

e Mechanical Testing

All the sintered test pieces were subjected to a three point bend test using two supports $1\frac{1}{2}$" (37.5 mm) apart, and a centrally applied load provided by an Avery testing machine. The load at fracture was recorded and the transverse rupture strengths were calculated using the dimensions determined in d in the following formula:

$$\mathrm{TRS} = \frac{3}{2}\,\frac{PL}{bh^2}$$

where: P = load at fracture
L = distance between supports
b = width of test piece
h = thickness of test piece

f Computer Analysis

The analysis of variance of the mechanical strengths and dimensional changes determined as described in e, was carried out at Aston University in Birmingham using the Stats Package programme on an ICL 1900 computer. The mechanical property and dimensional change data were then prepared for multiple regression analysis and the magnitude of the significant effects and interactions was determined, together with square effects, viz $(\text{density})^2$, $(\text{carbon})^2$, $(\text{copper})^2$, $(\text{nickel})^2$ at 99% significance and 5% significance levels.

g Metallography

Sections of test pieces from each alloy system and at each density level were prepared for conventional optical metallography. Preparation of these samples was difficult since their porous nature resulted in retention of the polishing lubricant and the etchant. Hence, thorough degreasing after polishing was required, together with extreme care during etching. All the samples were etched in Picral.

Selected samples, viz the highest strength system and those showing unusual microstructures, were examined on a Stereoscan scanning electron microscope, where an attempt was made to produce photographs showing both the fracture surface and the adjacent microstructure. This involved fracturing a polished and etched sample across the etched face and mounting, prior to examination, so that the electron beam was incident on both the polished and fractured surface.

These selected samples were also examined on a Microscan microprobe analyser, and X-ray patterns showing the distribution of copper and nickel in randomly selected areas were produced.

5.3 Experimental Results

a Strength and dimensional change data

Full details of all transverse rupture strengths and dimensional changes determined during the investigation, are given in Table 19.

b Computer analysis of mechanical strength and dimensional change data

i Analysis of variance

Table 20 shows the results of the analysis of variance.

Table 20

Analysis of Variance

Effect	Variance Ratio for each Dependent Variable			
	TRS	% L	% B	% Y
Nickel	116.1	37.8	99.3	49.6
Copper	179.8	1655.4	2681.1	1075.7
Carbon	-	95.6	335.0	156.1
Density	407.5	8.5	97.8	40.4
Nickel x Copper	78.8	68.0	117.1	54.1
Nickel x Carbon	28.1	11.4	7.9	6.5
Nickel x Density	-	3.9	4.6	-
Copper x Carbon	24.3	21.4	67.0	28.4
Copper x Density	4.85	-	4.2	-
Carbon x Density	-	-	4.5	-
Nickel x Copper x Carbon	4.14	9.1	6.9	4.9

All ratios are significant at 5% and 1% significance levels

Table 19

Experimentally Determined Transverse Rupture Strength and Dimensional Change

Sample No.		Green Density (% theoretical)	%C	%Cu	%Ni	Transverse Rupture Strength psi x 10^3 (MN/m^2)		Dimensional Change					
								% △ H		% △ B		% △ L	
1	1A	80	.3	2	2	64.2 (442)	61.4 (423)	0	0	+.13	+.13	+.16	+.13
2	2A	84	.3	2	2	83.8 (576)	82.9 (571)	0	0	+.17	+.17	+.20	+.14
3	3A	88	.3	2	2	95.0 (655)	97.2 (670)	0	0	+.17	+.17	+.14	+.20
4	4A	80	.6	2	2	63.1 (435)	69.1 (476)	+.10	+.10	+.03	+.03	+.12	+.14
5	5A	84	.6	2	2	88.5 (610)	88.5 (610)	-.10	0	+.07	+.07	+.09	+.13
6	6A	88	.6	2	2	107.3 (739)	108.0 (744)	0	-.10	+.10	+.10	+.09	+.12
7	7A	80	.9	2	2	83.1 (574)	93.1 (643)	-.20	0	-.13	-.10	-.05	-.03
8	8A	84	.9	2	2	108.9 (750)	111.9 (771)	-.10	-.20	+.05	0	+.09	+.03
9	9A	88	.9	2	2	129.7 (893)	129.1 (891)	-.26	0	+.08	+.07	+.11	+.14
10	10A	80	.3	5	2	80.5 (555)	74.8 (526)	+1.58	+1.58	+1.20	+1.36	+1.26	+1.22
11	11A	84	.3	5	2	98.7 (681)	99.4 (686)	+1.70	+1.50	+1.33	+1.38	+1.29	+1.37
12	12A	88	.3	5	2	113.5 (783)	117.5 (810)	+1.78	+1.98	+1.46	+1.53	+2.17	+2.16
13	13A	80	.6	5	2	83.6 (576)	86.1 (594)	+1.46	+1.29	+.96	+.99	+1.01	+.91
14	14A	84	.6	5	2	109.0 (751)	109.0 (751)	+1.60	+1.80	+.93	+1.01	+1.13	+1.04
15	15A	88	.6	5	2	128.2 (884)	128.2 (884)	+1.79	+1.10	+1.16	+1.16	+1.07	+1.07
16	16A	80	.9	5	2	92.8 (640)	97.8 (664)	+1.80	+.70	+.47	+.42	+.11	+.09
17	17A	84	.9	5	2	116.3 (802)	114.5 (789)	+.90	+.80	+.57	+.48	+.55	+.60
18	18A	88	.9	5	2	137.8 (956)	146.8 (1010)	+.69	+.80	+.77	+.75	+.79	+.75

Table 19 (Continuation 1)

19	19A	80	.3	8	2	74.5 (514)	78.2 (539)	+2.78	+2.88	+1.99	+2.13	+2.01	+1.98
20	20A	84	.3	8	2	98.2 (676)	100.5 (691)	+2.69	+2.69	+2.06	+2.13	+2.01	+2.01
21	21A	88	.3	8	2	114.7 (732)	112.2 (704)	+2.59	+2.68	+2.04	+2.06	+2.01	+2.01
22	22A	80	.6	8	2	86.6 (597)	85.5 (589)	+1.69	+1.89	+1.20	+1.20	+1.15	+1.15
23	23A	84	.6	8	2	108.9 (750)	106.8 (737)	+1.77	+1.88	+1.33	+1.26	+1.26	+1.30
24	24A	88	.6	8	2	123.1 (849)	124.0 (855)	+1.79	+1.48	+1.65	+1.69	+1.35	+1.35
25	25A	80	.9	8	2	79.3 (546)	79.3 (546)	+.60	+1.00	+.17	+.23	+.20	+.22
26	26A	84	.9	8	2	98.9 (681)	96.8 (668)	+.80	+.69	+.62	+.58	+4.1	+.41
27	27A	88	.9	8	2	123.5 (851)	116.0 (800)	+.60	+.60	+.70	+.70	+.68	+.71
28	28A	80	.3	2	5	67.9 (468)	73.1 (504)	+.19	0	-.13	-.13	-.02	-.03
29	29A	84	.3	2	5	95.1 (656)	92.6 (638)	-.30	-.39	-.03	-.03	-.02	-.03
30	30A	88	.3	2	5	111.1 (766)	106.6 (736)	+.20	+.10	+.17	+.13	+.05	+.05
31	31A	80	.6	2	5	77.9 (536)	74.6 (514)	-.30	-.30	-.27	-.27	-.17	-.17
32	32A	84	.6	2	5	99.1 (684)	96.1 (663)	0	-.20	0	0	-.02	-.03
33	33A	88	.6	2	5	123.0 (849)	124.0 (855)	-.40	-.20	+.03	+.03	0	-.02
34	34A	80	.9	2	5	78.4 (540)	89.5 (616)	-.40	-.30	-.27	-.27	-.25	-.27
35	35A	84	.9	2	5	98.8 (682)	108.5 (748)	-.20	-.20	-.17	-.17	-.17	-.20
36	36A	88	.9	2	5	126.1 (870)	124.1 (856)	0	0	0	-.05	0	-.03
37	37A	80	.3	5	5	69.7 (480)	73.0 (503)	+1.39	+1.09	+.93	+.93	+.91	+.90
38	38A	84	.3	5	5	93.5 (644)	94.3 (650)	+1.39	+1.59	+1.01	+.93	+.96	+.93
39	39A	88	.3	5	5	107.2 (739)	108.1 (745)	+1.78	+1.78	+1.30	+1.16	+1.01	+.99

Table 19 (Continuation 2)

40	40A	80	.6	5	5	67.6 (466)	67.4 (464)	+1.29	+1.39	+.53	+.56	+.63	+.61
41	41A	84	.6	5	5	86.1 (594)	83.5 (576)	+1.19	+1.19	+.95	+.81	+.80	+.85
42	42A	88	.6	5	5	97.5 (674)	102.5 (706)	+1.99	+1.59	+1.08	+.99	+.99	+.96
43	43A	80	.9	5	5	70.0 (482)	69.7 (480)	+1.59	+.89	+.27	+.33	+.30	+.39
44	44A	84	.9	5	5	89.4 (616)	91.3 (629)	+1.00	+1.20	+.63	+.60	+.64	+.64
45	45A	88	.9	5	5	96.1 (663)	97.7 (674)	+1.39	+1.30	+.92	+.97	+.91	+.91
46	46A	80	.3	8	5	59.4 (409)	57.1 (393)	+4.15	+3.87	+2.72	+2.92	+2.72	+2.61
47	47A	84	.3	8	5	83.2 (572)	89.7 (618)	+3.57	+3.79	+2.99	+2.92	+2.79	+2.74
48	48A	88	.3	8	5	96.9 (667)	96.9 (667)	+3.87	+3.56	+2.96	+2.96	+2.89	+2.89
49	49A	80	.6	8	6	52.7 (363)	48.9 (337)	+2.78	+2.98	+2.10	+2.03	+2.00	+1.95
50	50A	84	.6	8	5	65.5 (451)	63.5 (437)	+2.80	+2.80	+2.16	+2.16	+2.06	+2.05
51	51A	88	.6	8	5	66.0 (454)	66.8 (461)	+2.98	+2.97	+2.37	+2.31	+2.33	+2.33
52	52A	80	.9	8	5	42.4 (293)	47.4 (317)	+2.39	+1.76	+1.26	+1.36	+1.27	+1.24
53	53A	84	.9	8	5	56.8 (392)	55.2 (380)	+2.41	+2.22	+1.50	+1.46	+1.49	+1.46
54	54A	88	.9	8	5	65.5 (451)	68.6 (472)	+2.59	+2.39	+1.84	+2.20	+1.70	+1.67
55	55A	80	.3	2	8	76.7 (524)	81.7 (570)	+.10	+.10	-.33	+.27	-.31	-.31
56	56A	84	.3	2	8	103.5 (713)	103.5 (713)	-.30	-.10	-.20	-.15	-.20	-.19
57	57A	88	.3	2	8	124.3 (856)	125.2 (862)	0	-.39	0	0	-.06	-.06
58	58A	80	.6	2	8	87.5 (602)	86.1 (594)	-.10	-.40	-.47	-.47	-.42	-.42
59	59A	84	.6	2	8	108.0 (744)	114.2 (766)	-.30	-.50	-.33	-.32	-.31	-.31
60	60A	88	.6	2	8	127.0 (875)	130.8 (902)	-.30	0	-.15	-.20	-.14	-.19

Table 19 (Continuation 3)

61	61A	80	.9	2	8	76.5 (526)	79.3 (546)	-.28	-.40	-.66	-.66	-.61	-.60
62	62A	84	.9	2	8	102.8 (707)	102.8 (707)	-.80	-.62	-.23	-.23	-.46	-.49
63	63A	88	.9	2	8	117.7 (811)	121.3 (936)	-.40	-.40	-.28	-.30	-.25	-.25
64	64A	80	.3	5	8	67.9 (467)	69.4 (477)	+.79	+1.10	+.53	+.53	+.50	+.50
65	65A	84	.3	5	8	96.0 (661)	96.2 (662)	+.80	+1.00	+.75	+.77	+.68	+.68
66	66A	88	.3	5	8	118.1 (814)	115.2 (794)	+1.00	+1.20	+.86	+.83	+.88	+.91
67	67A	80	.6	5	8	67.1 (463)	70.8 (488)	+.60	+1.10	+.27	+.30	+.25	+.25
68	68A	84	.6	5	8	89.0 (613)	84.0 (579)	+1.20	+.80	+.48	+.43	+.46	+.40
69	69A	88	.6	5	8	95.1 (656)	95.6 (659)	+1.19	+1.69	+.73	+.76	+.66	+.69
70	70A	80	.9	5	8	72.9 (502)	66.9 (461)	0	+.29	-.07	-.07	-.08	-.08
71	71A	84	.9	5	8	80.4 (554)	86.4 (596)	+.40	+.80	+.03	+.09	+.05	+.16
72	72A	88	.9	5	8	91.2 (628)	95.6 (659)	+1.10	+1.20	+.43	+.33	+.39	+.35
73	73A	80	.3	8	8	65.5 (452)	63.5 (438)	+2.70	+2.89	+1.87	+1.87	+1.76	+1.75
74	74A	84	.3	8	8	81.1 (558)	78.0 (537)	+2.70	+3.10	+2.03	+2.03	+1.95	+1.93
75	75A	88	.3	8	8	91.7 (633)	90.0 (650)	+3.26	+2.97	+2.40	+2.40	+2.06	+2.06
76	76A	80	.6	8	8	45.6 (314)	47.6 (326)	+2.69	+2.89	+1.46	+1.46	+1.40	+1.38
77	77A	84	.6	8	8	59.8 (413)	53.8 (372)	+2.60	+2.60	+1.66	+1.73	+1.20	+1.64
78	78A	88	.6	8	8	64.4 (444)	64.4 (444)	+3.19	+3.29	+2.16	+2.19	+2.05	+2.03
79	79A	80	.9	8	8	44.6 (308)	43.6 (300)	+2.69	+2.40	+.93	+.93	+.88	+.90
80	80A	84	.9	8	8	52.0 (359)	47.7 (329)	+2.90	+2.59	+1.43	+1.35	+1.35	+.123
81	81A	88	.9	8	8	59.8 (413)	63.6 (438)	+3.29	+3.21	+1.89	+1.88	+1.81	+1.78

ii Multiple Regression Analysis

Final multiple regression equations at 5% significance level are given below:

1 $\mathrm{TRS} = -397.2 + 5.51\,(\text{density})^{*} + 37.20\,(\%\,\mathrm{Cu}) + 45.55\,(\%\,\mathrm{C}^2) + .81\,(\%\,\mathrm{Ni}^2) - 1.49\,(\%\,\mathrm{Cu} \times \%\,\mathrm{Ni}) - 5.64\,(\%\,\mathrm{C} \times \%\,\mathrm{Ni}) - 5.80\,(\%\,\mathrm{Cu} \times \%\,\mathrm{C}) - .26\,(\text{Density} \times \%\,\mathrm{Cu}) - .84\,(\%\,\mathrm{Cu}^2)$

Multiple correlation coefficient = .959

Residual error = 6.63

2 $\%\,\Delta \mathrm{H} = 50.4 + 1.002\,(\%\,\mathrm{C}) + .596\,(\%\,\mathrm{Cu}) + .066\,(\%\,\mathrm{Ni} \times \%\,\mathrm{Cu} \times \%\,\mathrm{C}) + .003\,(\%\,\mathrm{Ni} \times \text{Density}) + .007\,(\text{Density}^2) - .037\,(\%\,\mathrm{Ni}^2) - .566\,(\%\,\mathrm{C} \times \%\,\mathrm{Cu}) - .146\,(\%\,\mathrm{C} \times \%\,\mathrm{Ni}) - 1.246\,(\text{Density})$

Multiple correlation coefficient = .969

Residual error = .315

3 $\%\,\Delta \mathrm{B} = -1.074 + .009\,(\text{Density} \times \%\,\mathrm{C}) + .006\,(\text{Density} \times \%\,\mathrm{Cu}) + .003\,(\text{Density} \times \%\,\mathrm{Ni}) + .035\,(\%\,\mathrm{Ni} \times \%\,\mathrm{Cu} \times \%\,\mathrm{C}) - .032 \times (\%\,\mathrm{Ni})^2 - .420\,(\%\,\mathrm{Cu} \times \%\,\mathrm{C}) - .087\,(\%\,\mathrm{C} \times \%\,\mathrm{Ni})$

Multiple correlation coefficient = .970

Residual error = .223

4 $\%\,\Delta \mathrm{L} = -2.218 + .0003\,(\text{Density}^2) + .4199\,(\%\,\mathrm{Cu}) + .1205\,(\%\,\mathrm{Ni}) + .0312\,(\%\,\mathrm{Ni} \times \%\,\mathrm{Cu} \times \%\,\mathrm{C}) - .0265\,(\%\,\mathrm{Ni}^2) - .3533\,(\%\,\mathrm{Cu} \times \%\,\mathrm{C})$

Multiple correlation coefficient = .957

Residual error = .255

* i.e. 80-88

A graphical representation (based on these equations) showing the effect of varying density, % copper, % carbon and % nickel on the four parameters measured, viz TRS, % ΔH, % ΔB and % ΔL is given on Figures 53 - 68. Maximum and minimum points predicted by squared relationships were calculated by differentiating partially with respect to each specific variable and equating to zero. This value of the independent variable was then substituted in the relevant equation and maximum or minimum values of the dependent variable were calculated.

c Metallography

i Optical Metallography

Table 21 summarises the microstructure of the samples examined and the corresponding photomicrographs at 84% theoretical density level are shown in Figures 69 - 99. There was no significant variation in microstructure with varying density apart from reduced porosity.

ii Stereoscan Examination

Figures 100 - 103 show the fracture surface and adjacent microstructure of the four samples containing transformation products, and Figure 104 shows a similar view of the system showing the highest experimentally determined transverse rupture strength, viz Fe .9%C 5%Cu 2%Ni, 88% density.

FIG 53 T.R.S. v AS PRESSED DENSITY

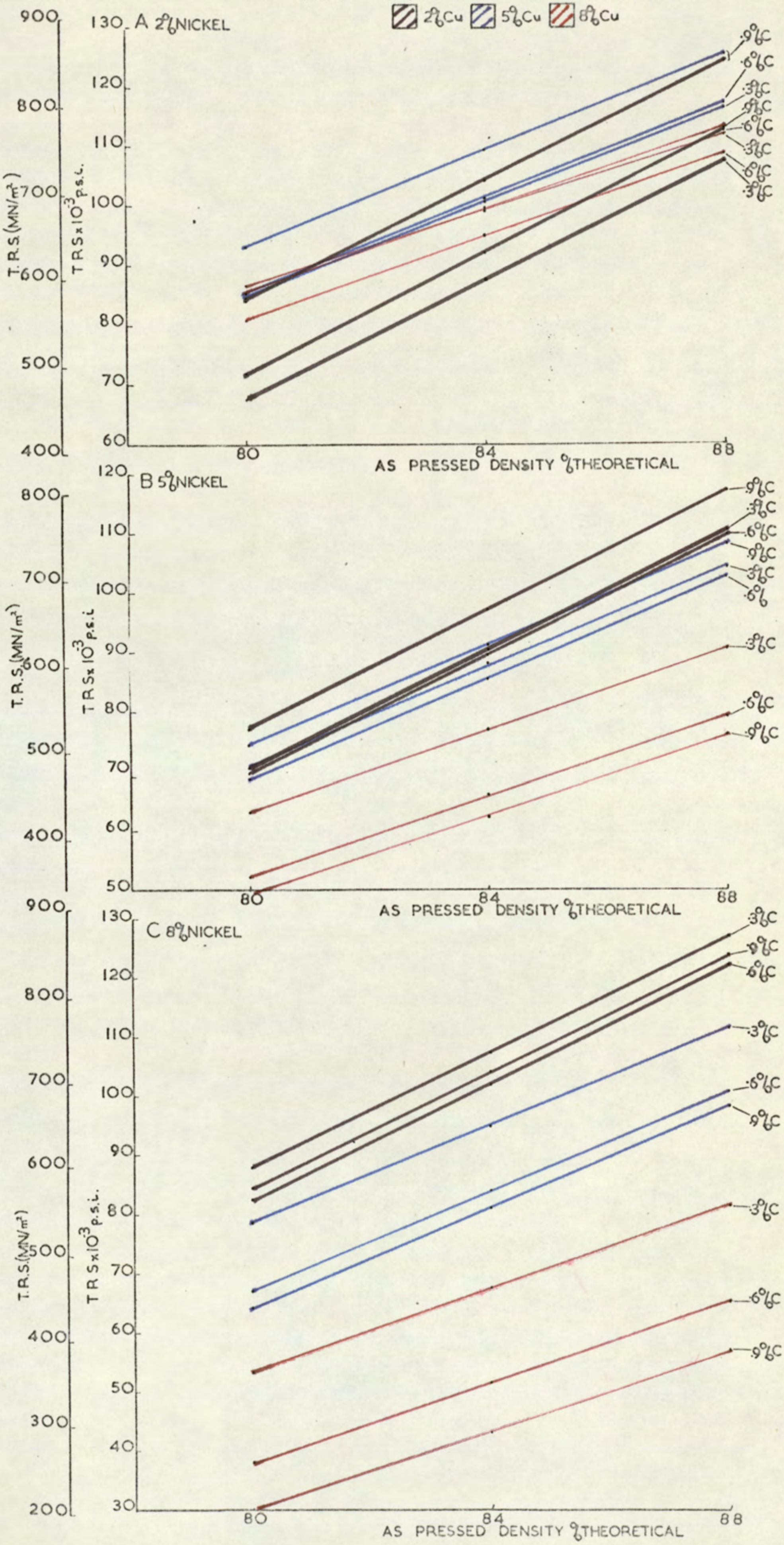


FIG 54 T.R.S. v %COPPER

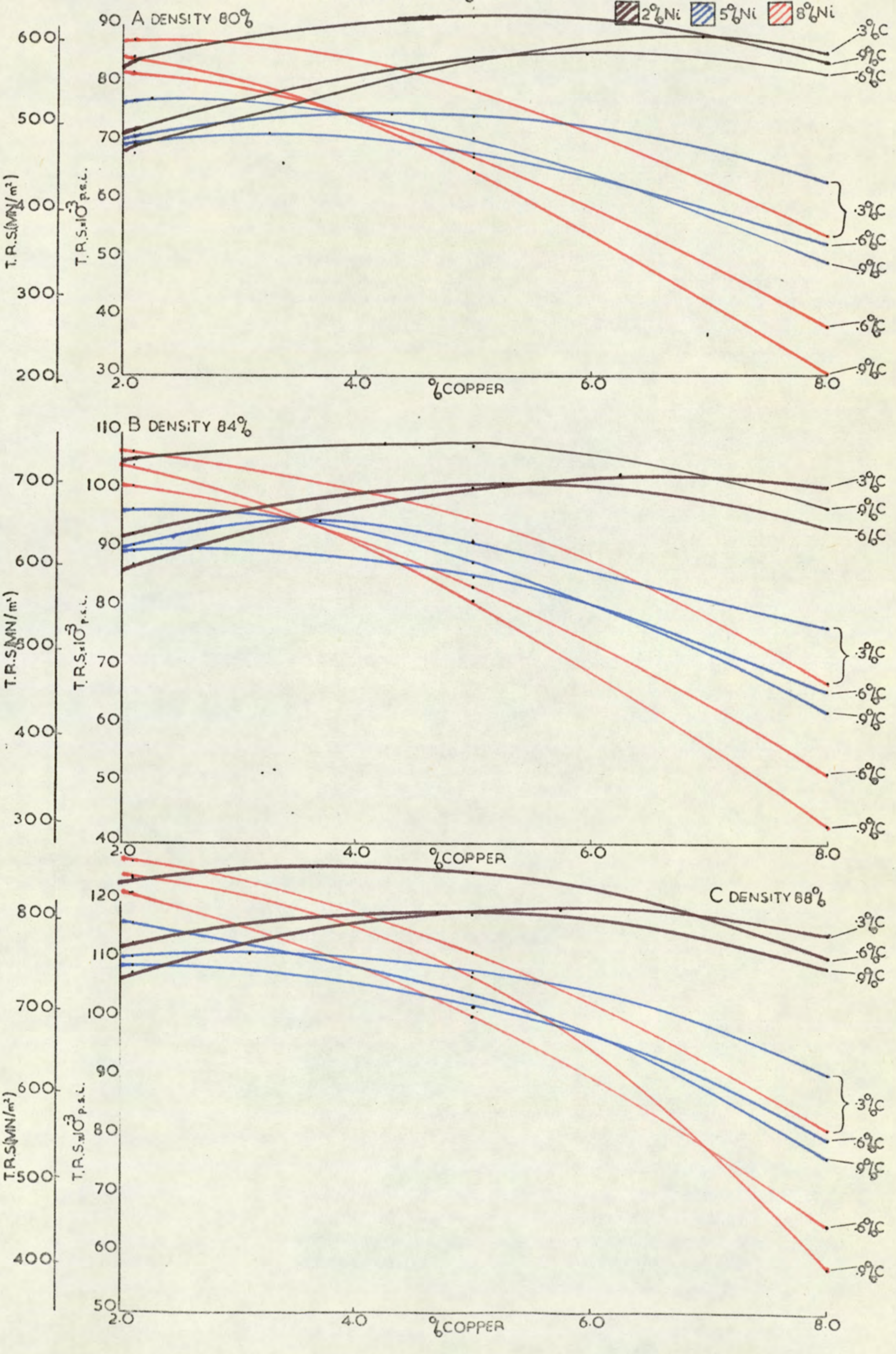


FIG55 T.R.S. v %CARBON

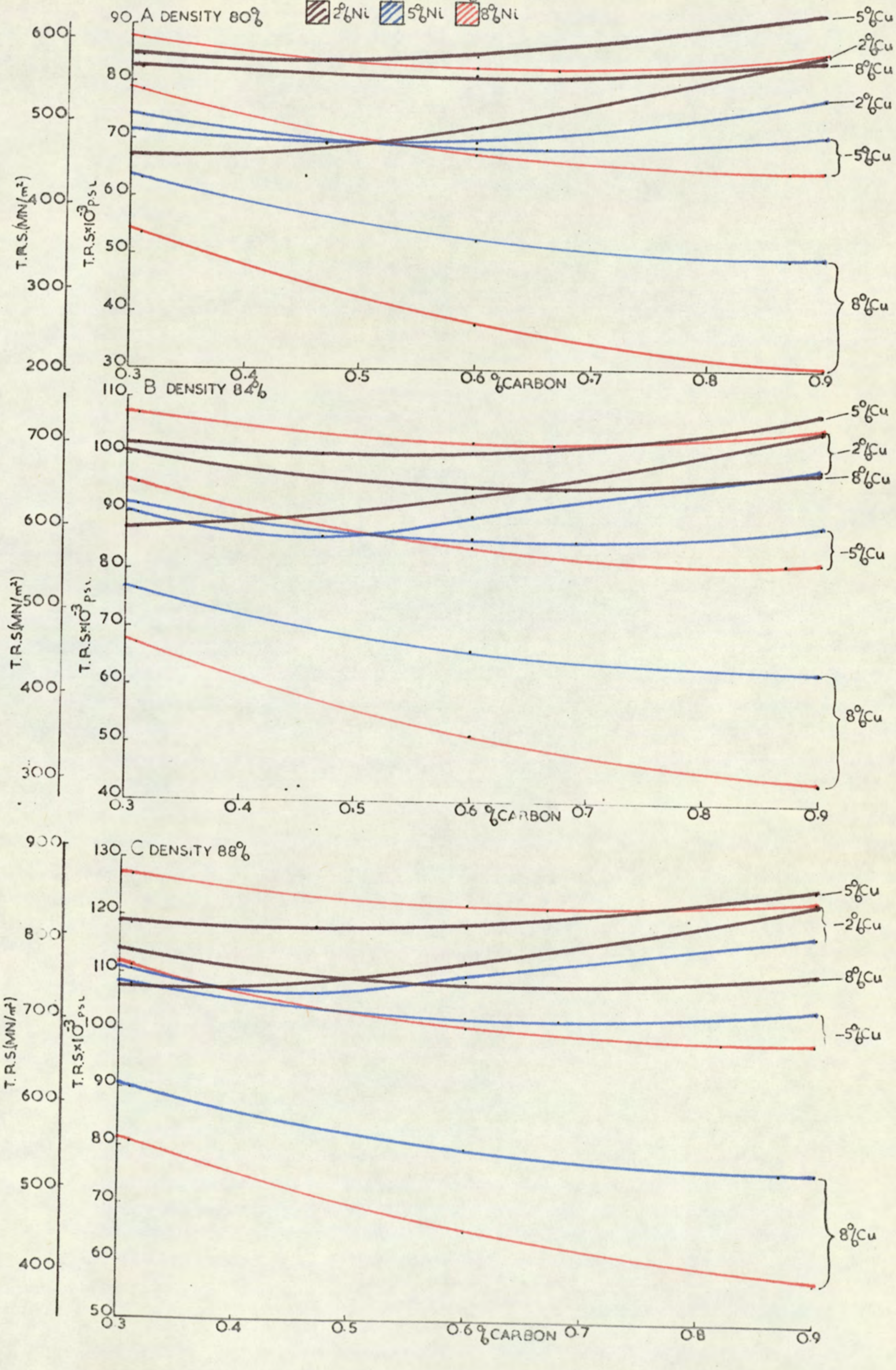


FIG56 T.R.S. V %NICKEL

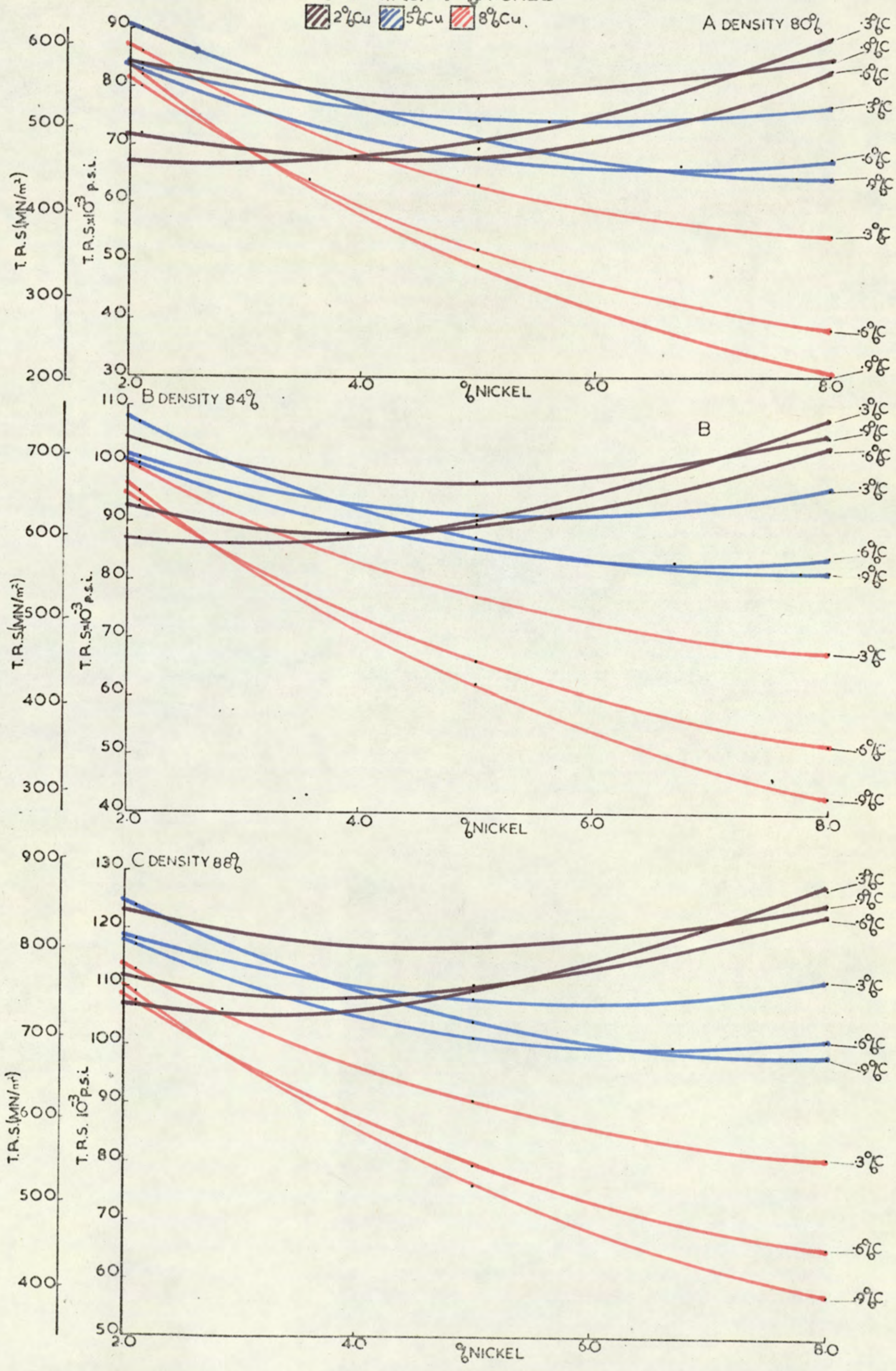


FIG 57 %ΔH v AS PRESSED DENSITY

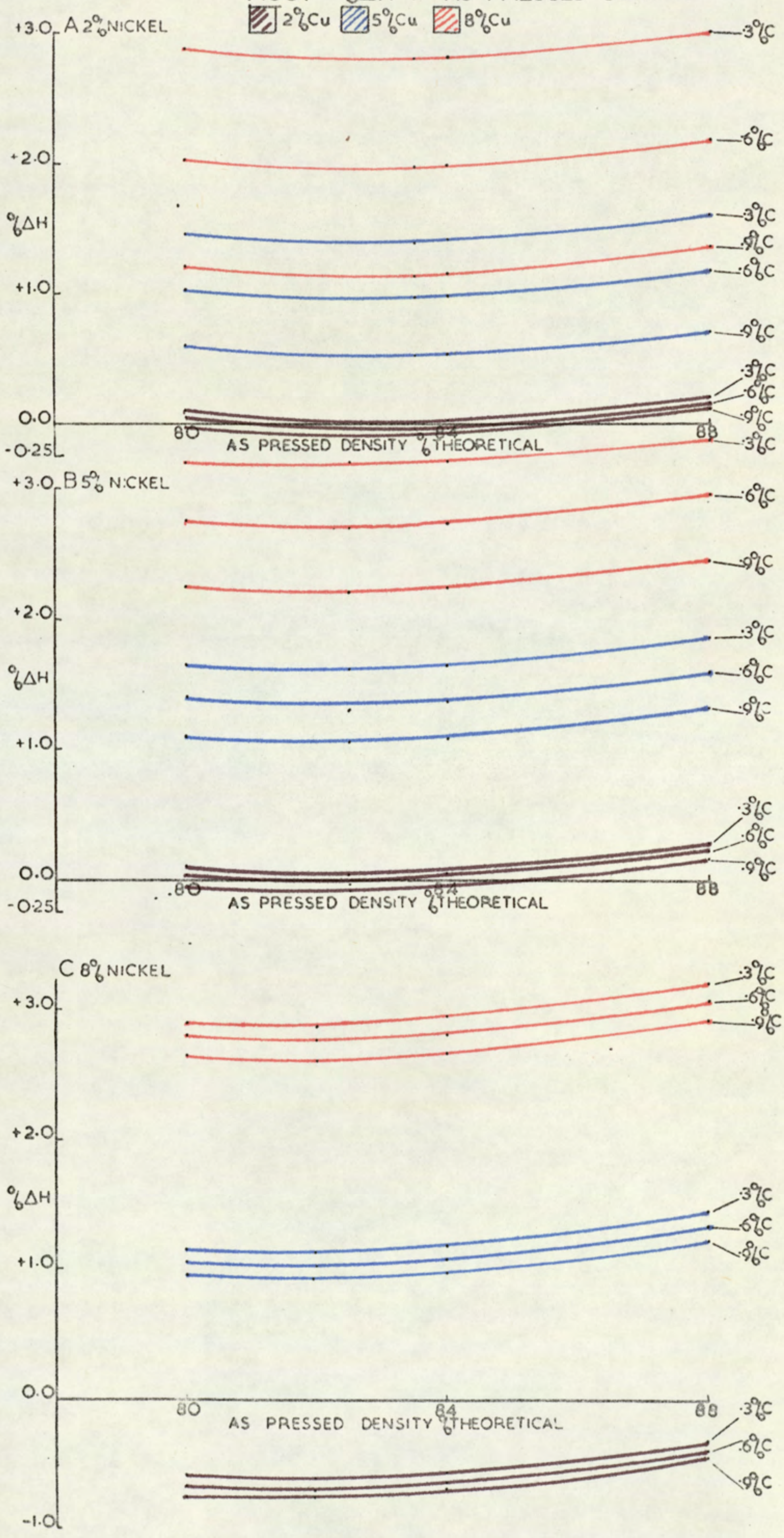


FIG58 %ΔH v %COPPER

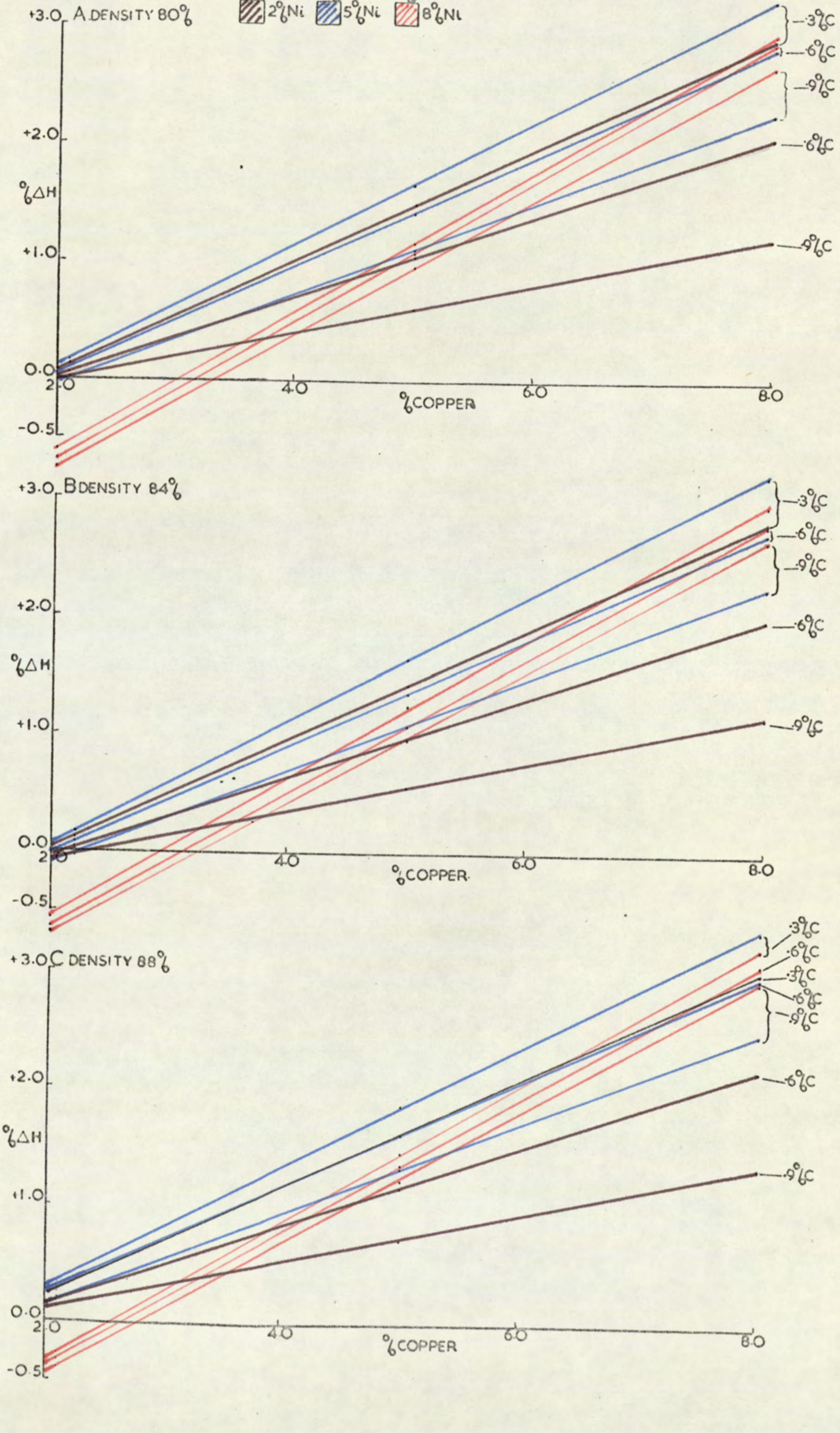


FIG59 %ΔH v %CARBON

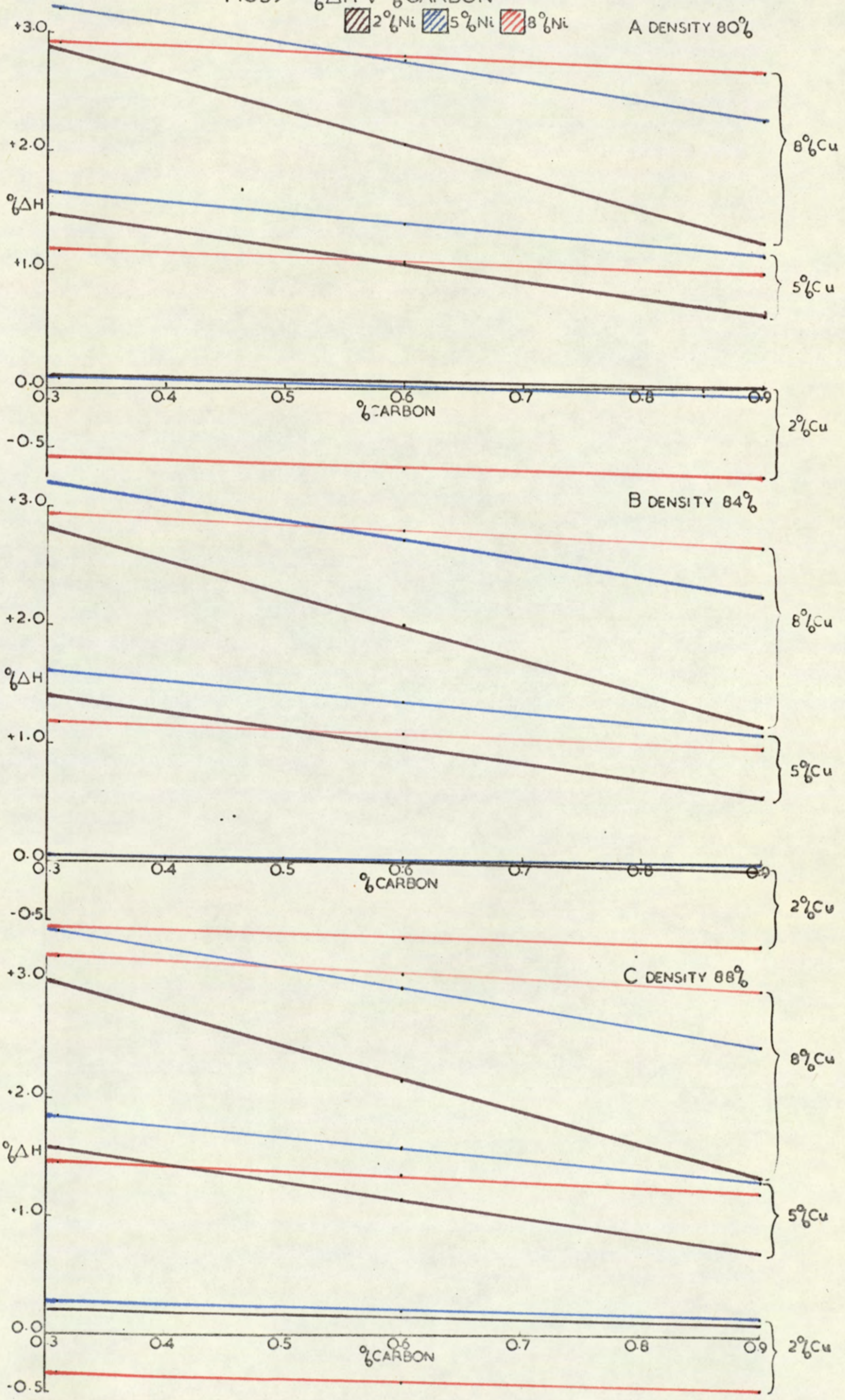


FIG 60 %ΔH v %NICKEL

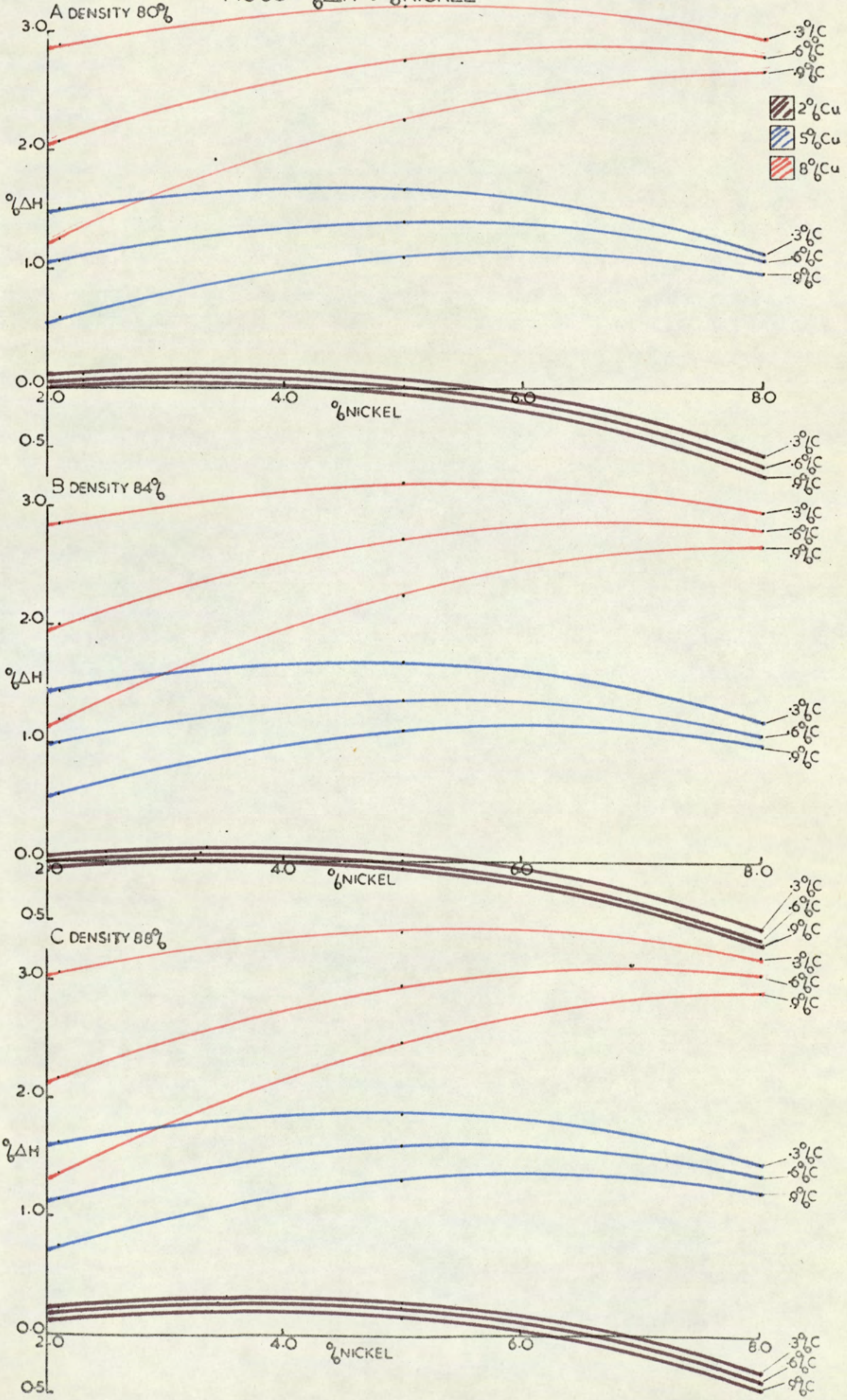


FIG 61 %ΔB v AS PRESSED DENSITY

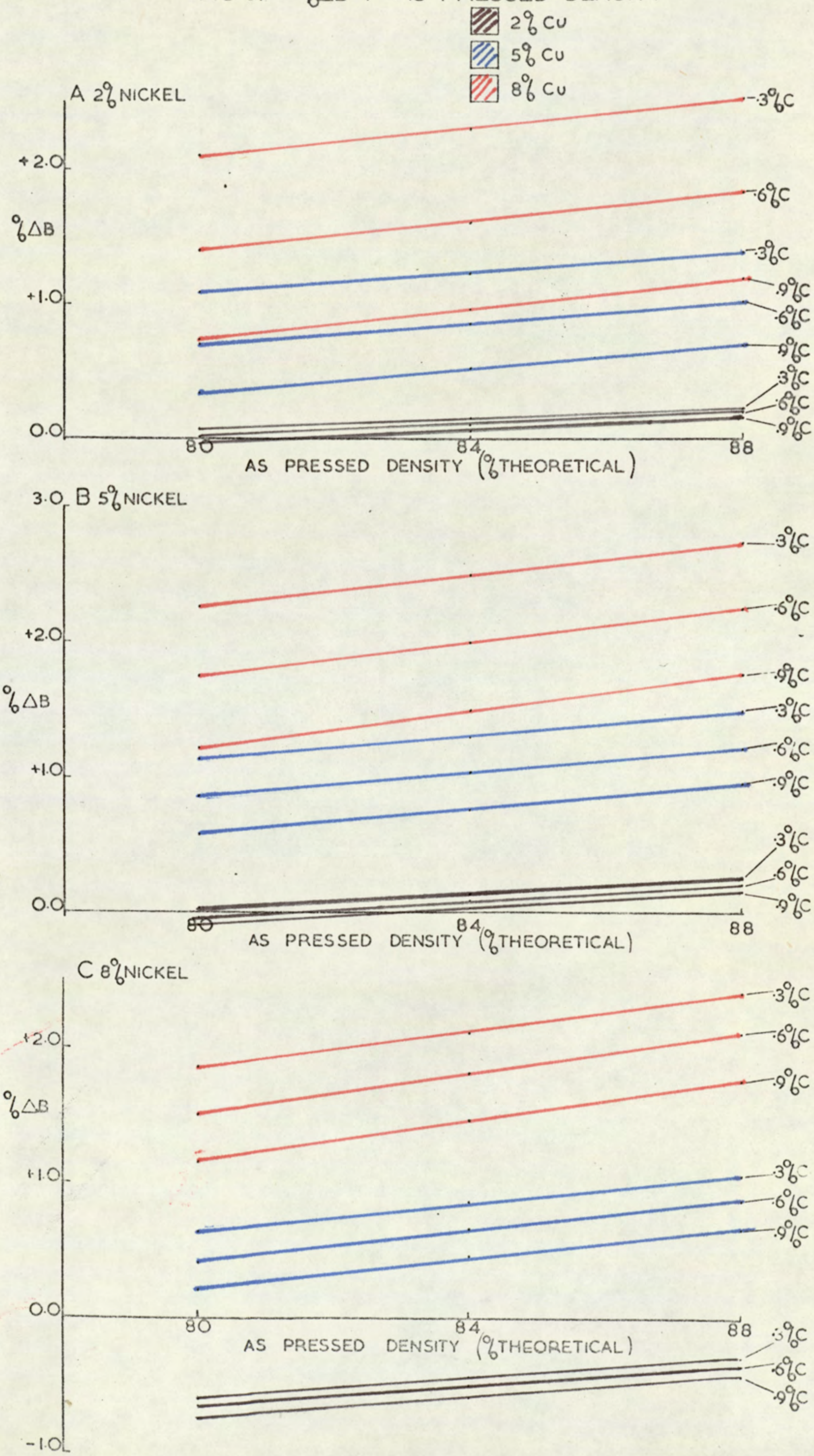
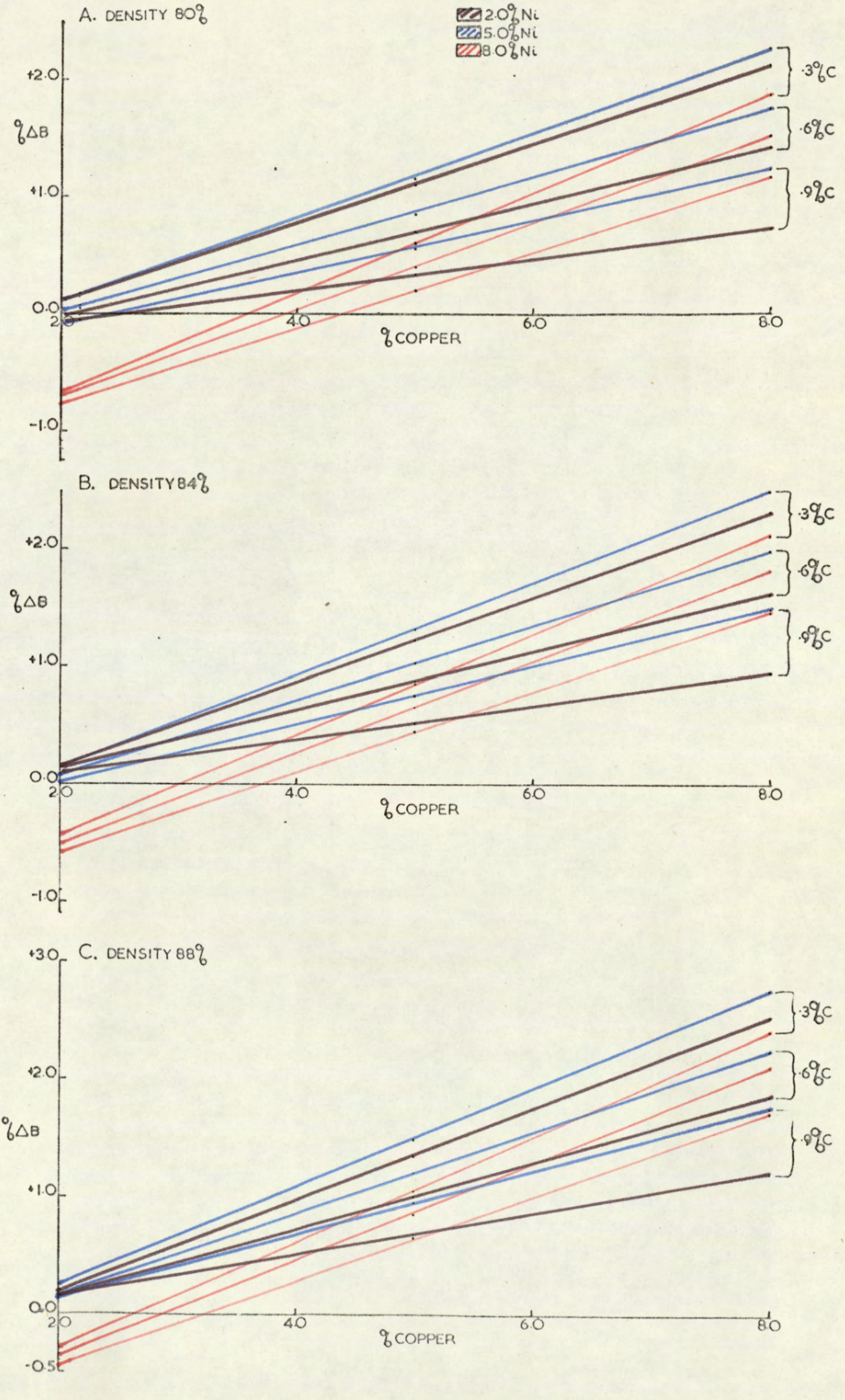


FIG 62 %ΔB v % COPPER



A. DENSITY 80%

B. DENSITY 84%

C. DENSITY 88%

FIG 63 %ΔB v %CARBON

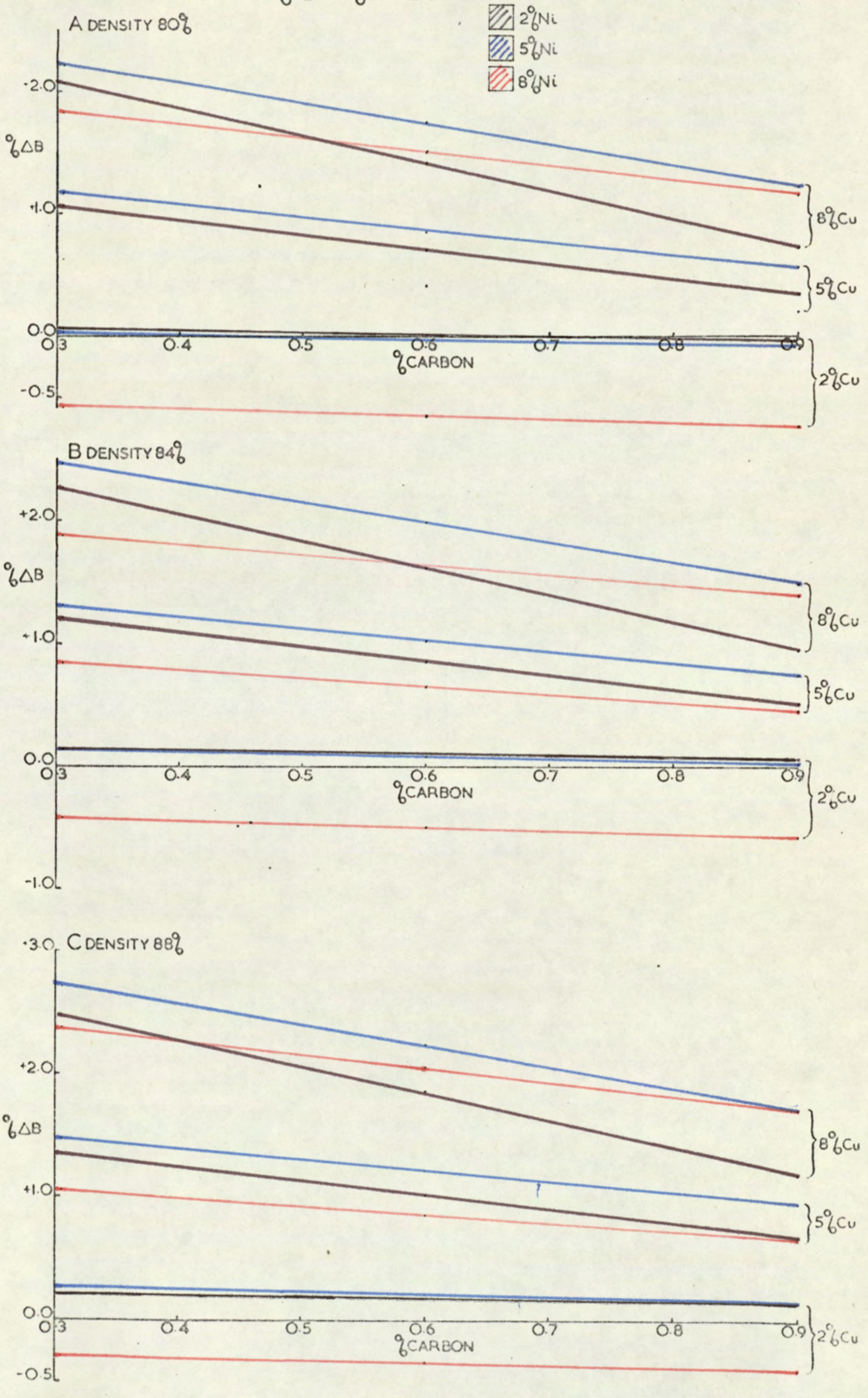


FIG 64 %ΔB v %NICKEL

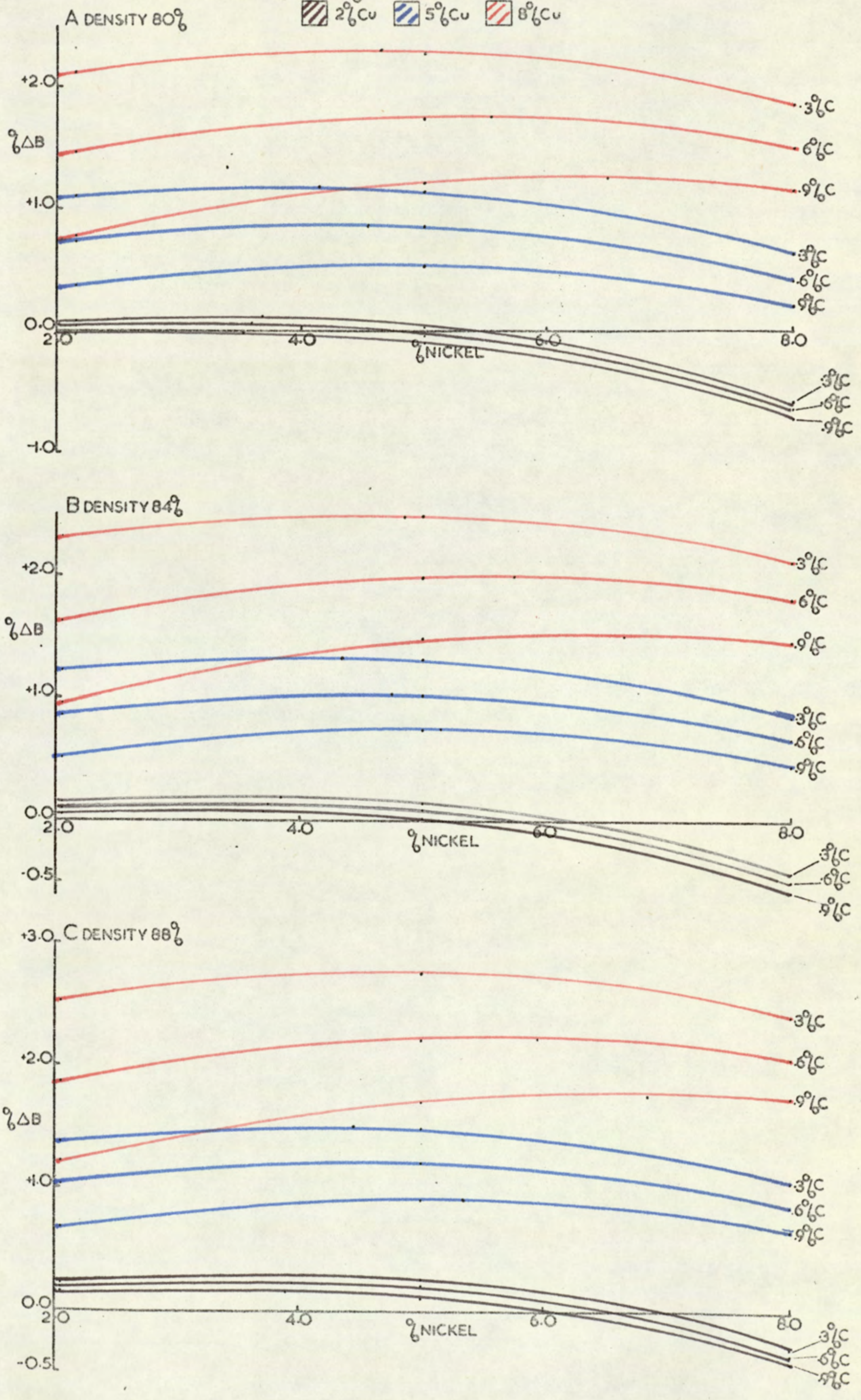


FIG 65 %ΔL v AS PRESSED DENSITY

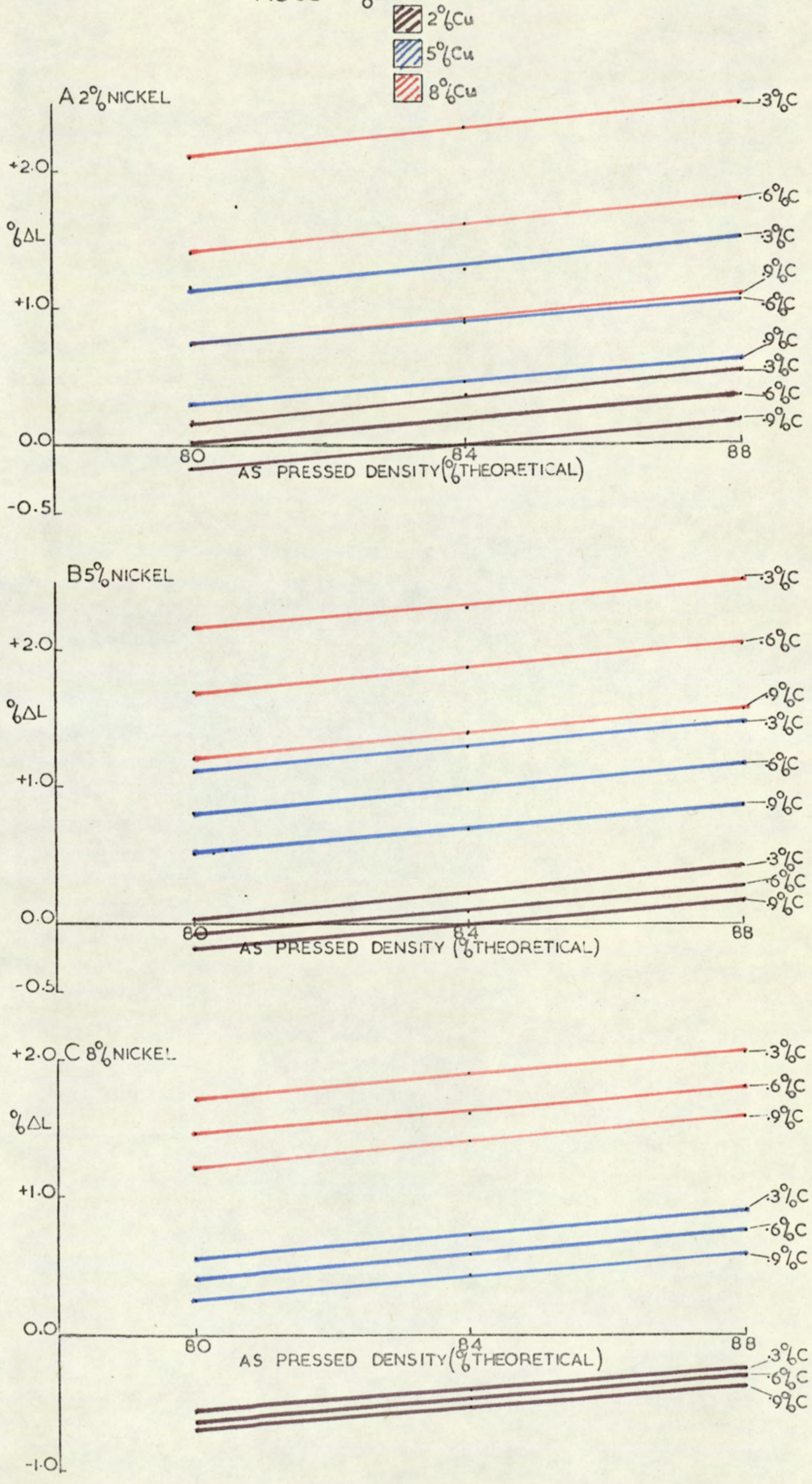


FIG 66 %ΔL v %COPPER

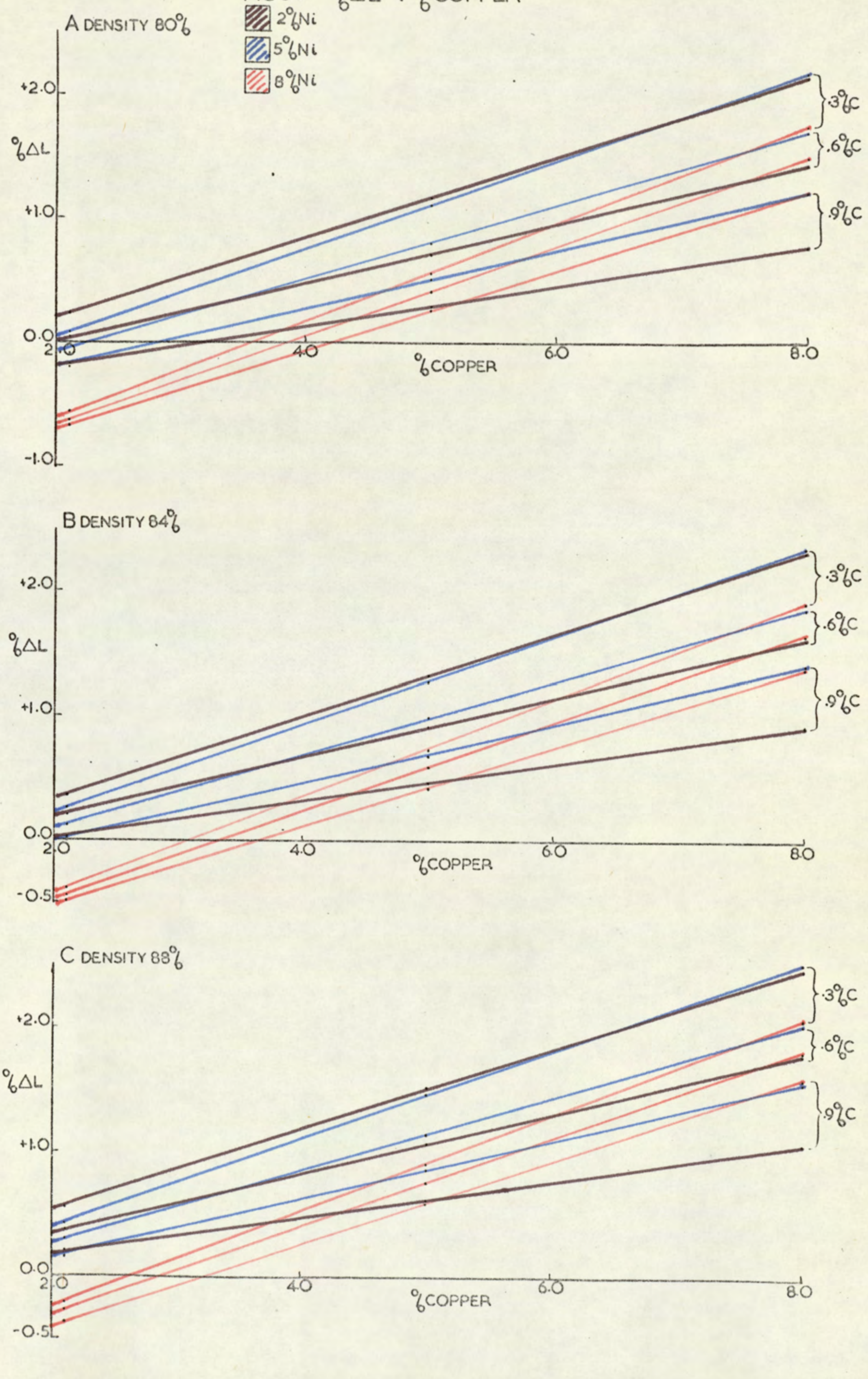


FIG 67 %ΔL v %CARBON

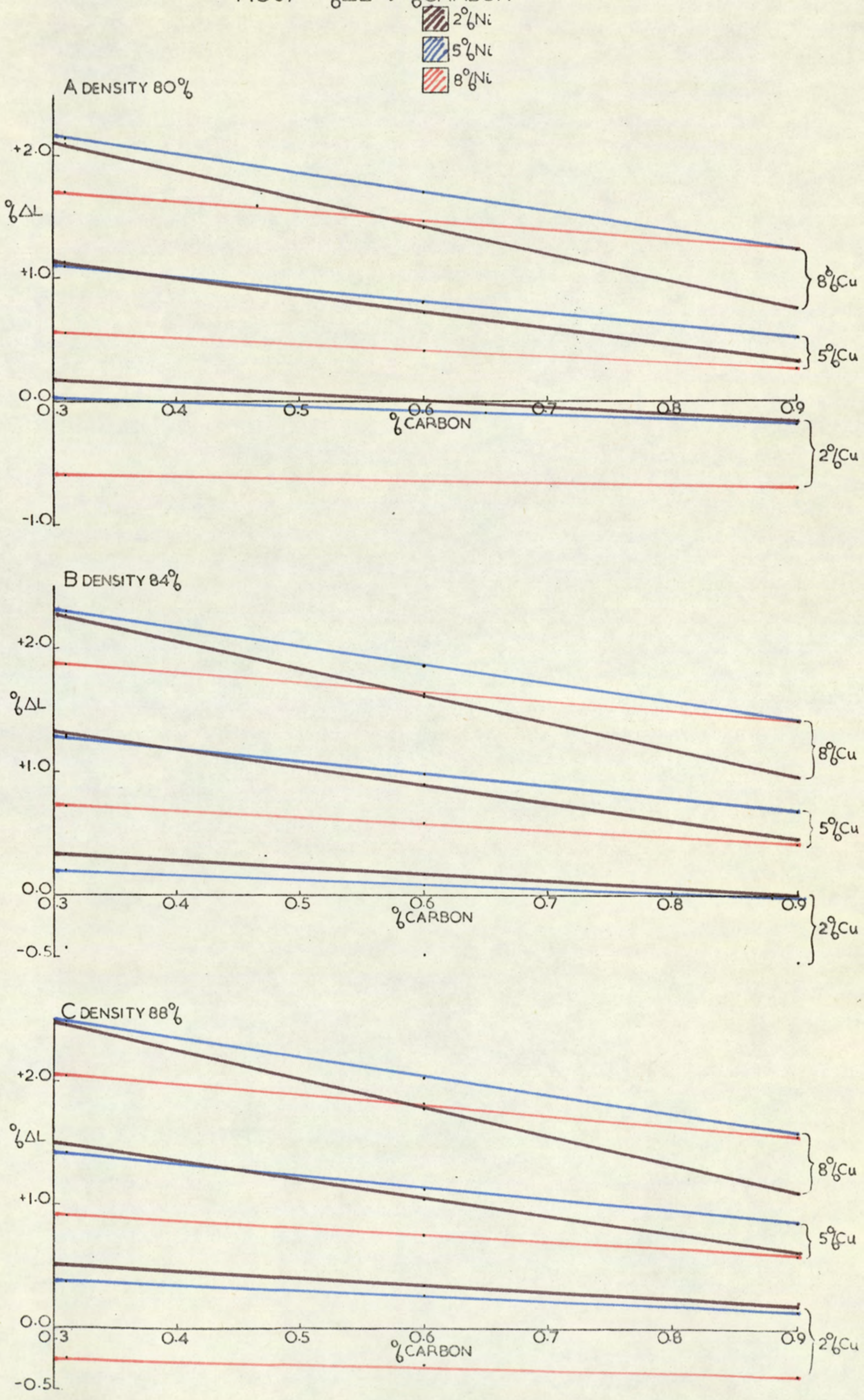


FIG 68 %ΔL V %NICKEL

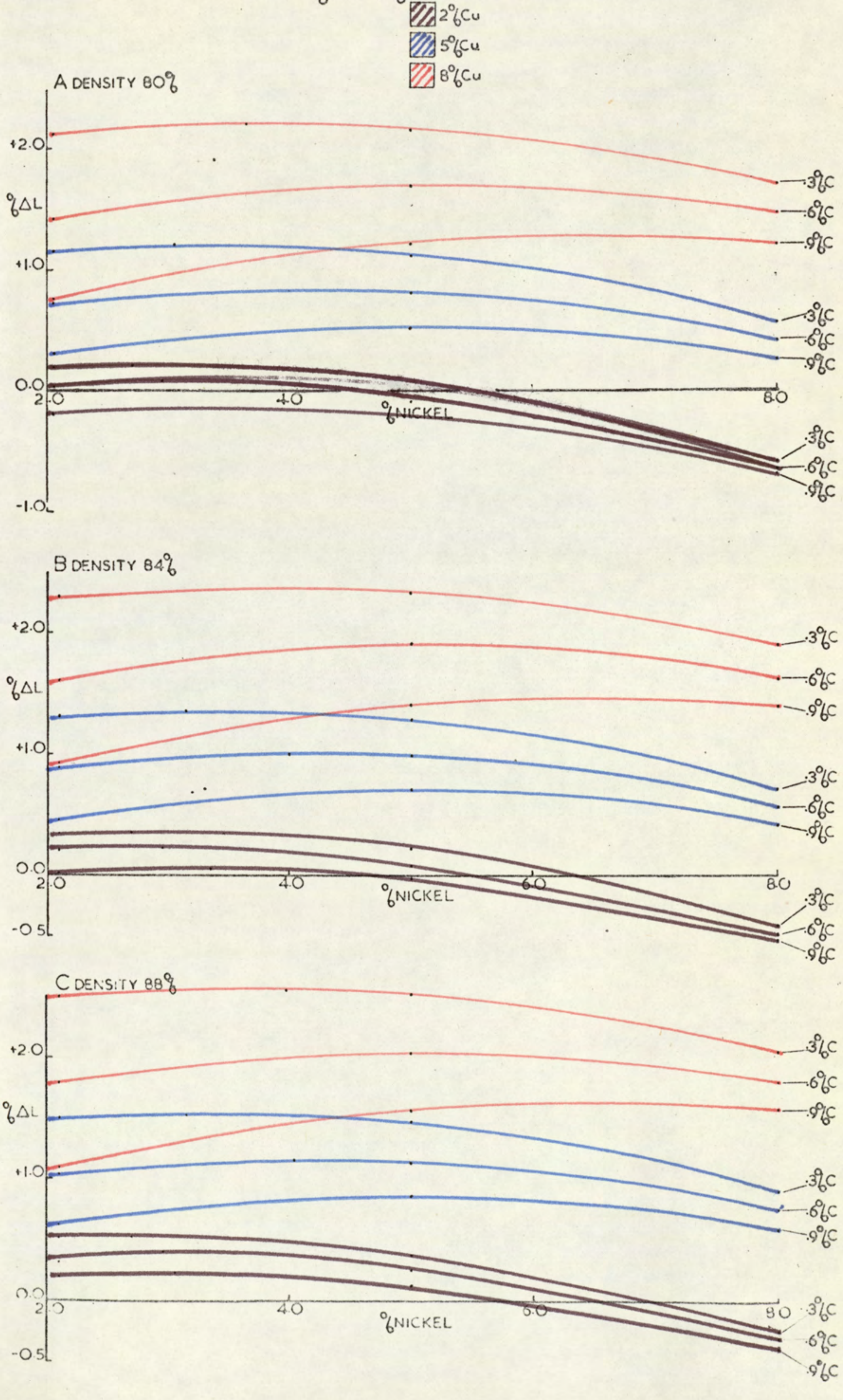


Table 21

Microstructures

Composition	Structure
.3%C 2%Cu 2%Ni .6%C 2%Cu 2%Ni	Ferrite + pearlite
.9%C 2%Cu 2%Ni	Pearlite + some retained austenite
.3%C 5%Cu 2%Ni	Ferrite + pearlite Very slight undissolved Cu
.6%C 5%Cu 2%Ni	Ferrite + pearlite
.9%C 5%Cu 2%Ni	Pearlite + retained austenite*
.3%C 8%Cu 2%Ni .6%C 8%Cu 2%Ni	Ferrite, pearlite + some undissolved Cu
.9%C 8%Cu 2%Ni	Pearlite, retained austenite, undissolved Cu
.3%C 2%Cu 5%Ni .6%C 2%Cu 5%Ni	Ferrite + pearlite
.9%C 2%Cu 5%Ni	Pearlite + retained austenite
.3%C 5%Cu 5%Ni .6%C 5%Cu 5%Ni	Ferrite + pearlite
.9%C 5%Cu 5%Ni	Pearlite, retained austenite
.3%C 8%Cu 5%Ni	Ferrite + pearlite - very slight undissolved Cu
.6%C 8%Cu 5%Ni	Ferrite + pearlite
.9%C 8%Cu 5%Ni	Pearlite, retained austenite, undissolved Cu*, large needles of either bainite or martensite
.3%C 2%Cu 8%Ni .6%C 2%Cu 8%Ni	Ferrite + pearlite
.9%C 2%Cu 8%Ni	Pearlite, retained austenite*, asicular bainite or martensite
.3%C 5%Cu 8%Ni .6%C 5%Cu 8%Ni	Ferrite + pearlite
.9%C 5%Cu 8%Ni	Pearlite, retained austenite*, asicular bainite or martensite
.3%C 8%Cu 8%Ni .6%C 8%Cu 8%Ni	Ferrite + pearlite
.9%C 8%Cu 8%Ni	Pearlite, retained austenite*, some undissolved Cu, asicular bainite, or martensite

* Samples examined on Stereoscan and Microprobe Analyser

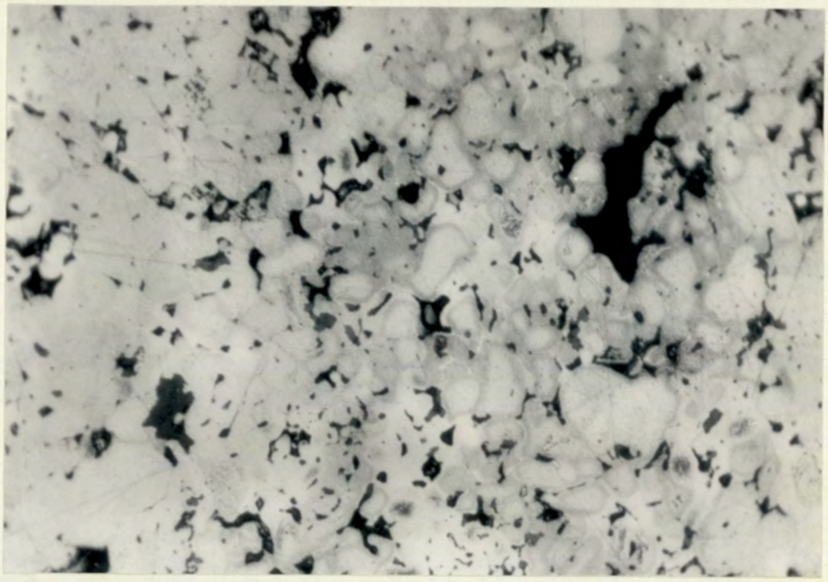


Figure 69

.3%C 2%Cu 2%Ni

(Mag 180 x 4)

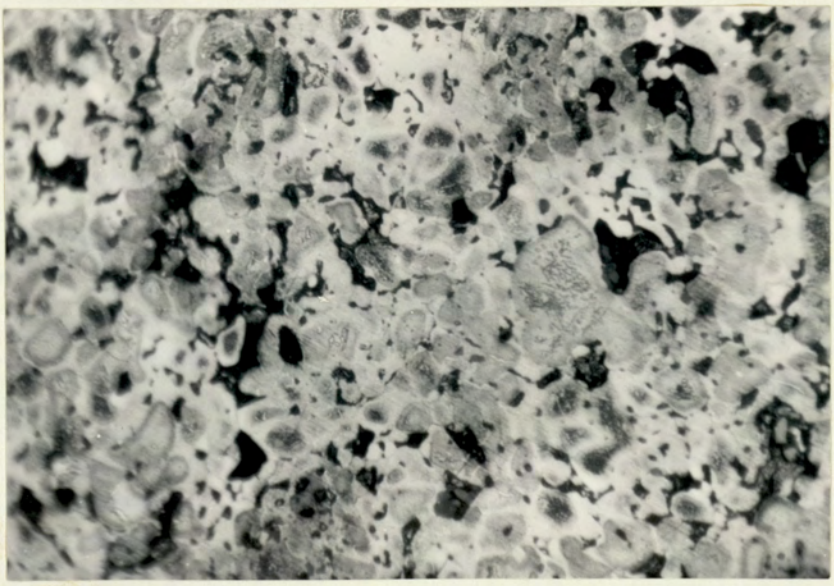


Figure 70

.3%C 5%Cu 2%Ni

(Mag 180 x 4)

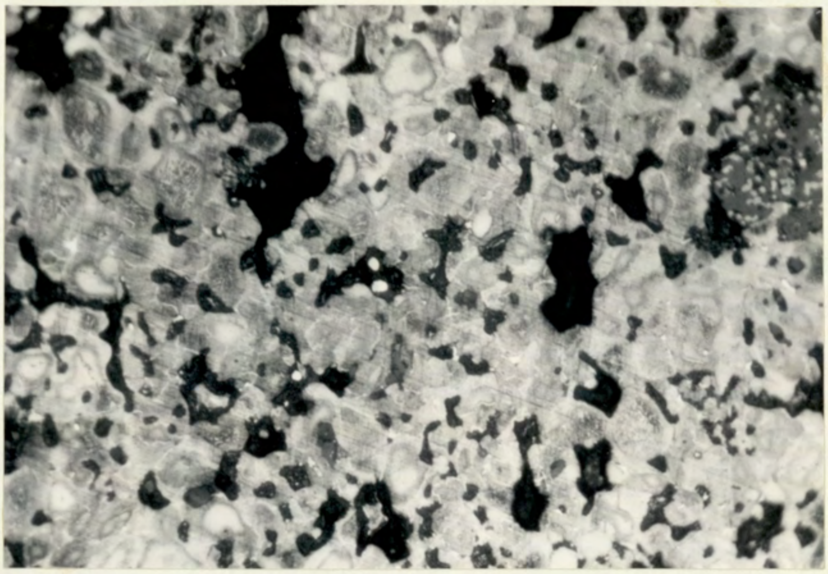


Figure 71
.3%C 8%Cu 2%Ni
(Mag 180 x 4)

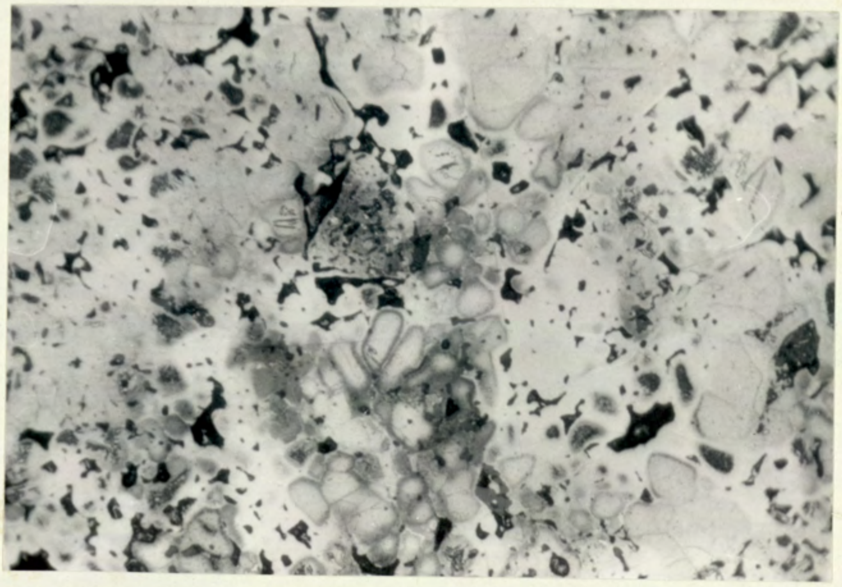


Figure 72
.3%C 2%Cu 5%Ni
(Mag 180 x 4)

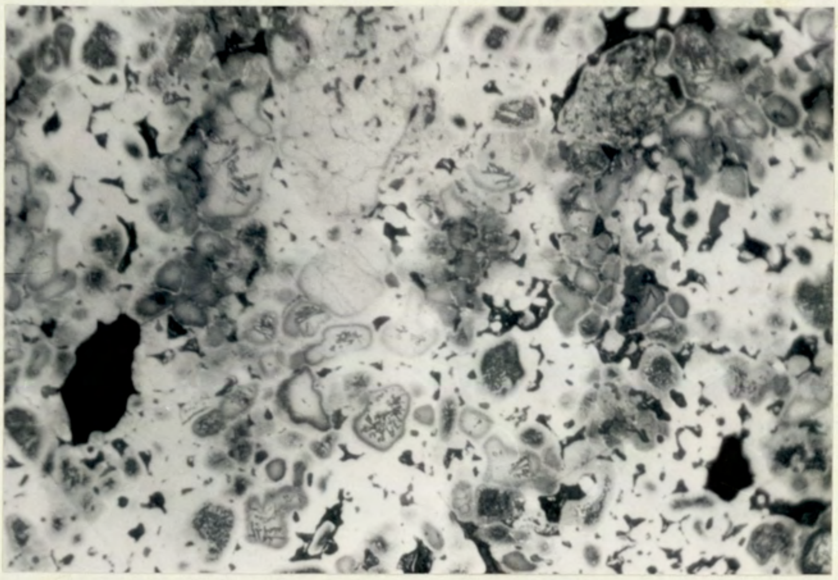


Figure 73

.3%C 5%Cu 5%Ni

(Mag 180 x 4)

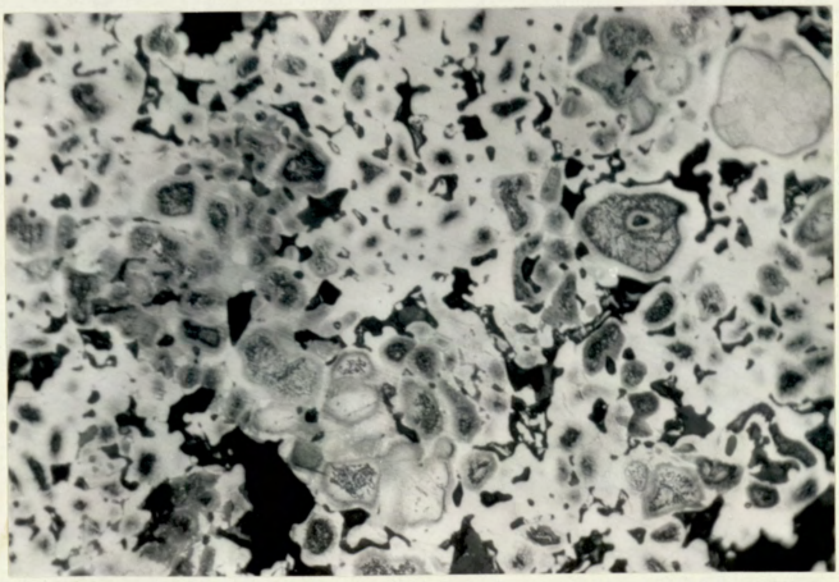


Figure 74

.3%C 8%Cu 5%Ni

(Mag 180 x 4)

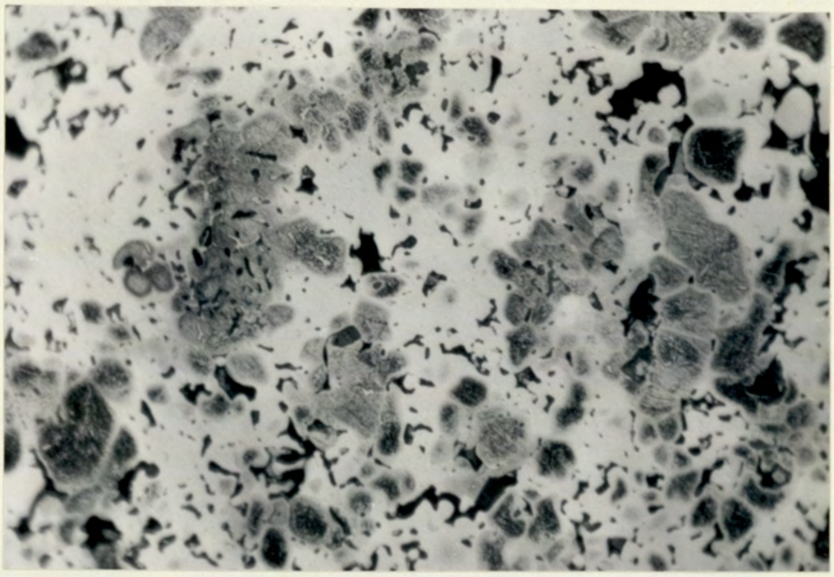


Figure 75
.3%C 2%Cu 8%Ni
(Mag 180 x 4)

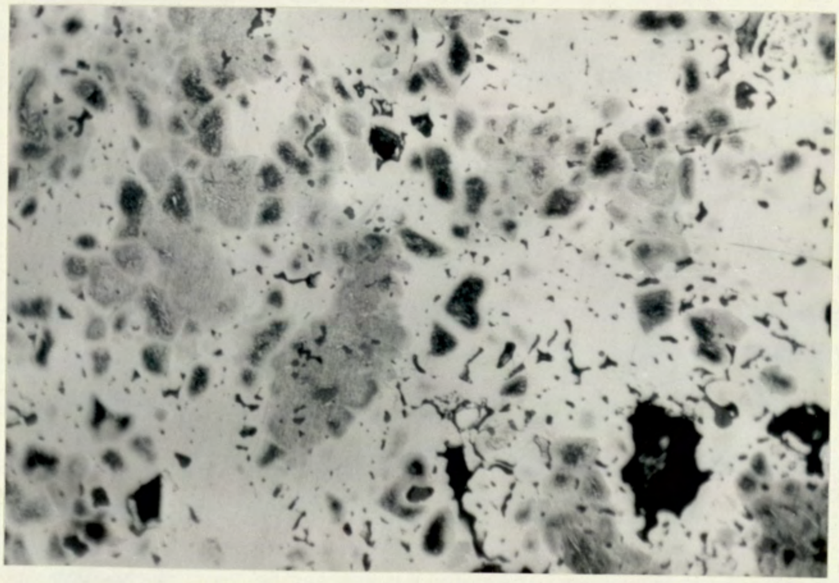


Figure 76
.3%C 5%Cu 8%Ni
(Mag 180 x 4)

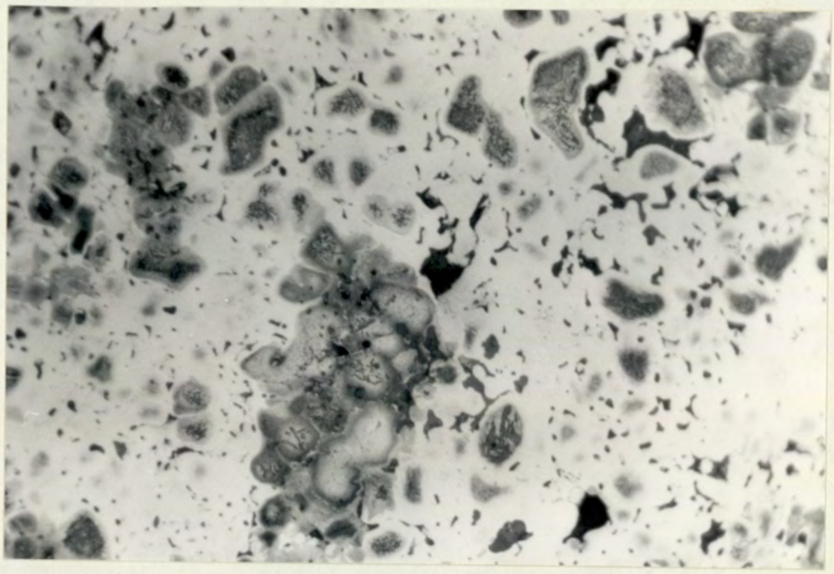


Figure 77

.3%C 8%Cu 8%Ni

(Mag 180 x 4)

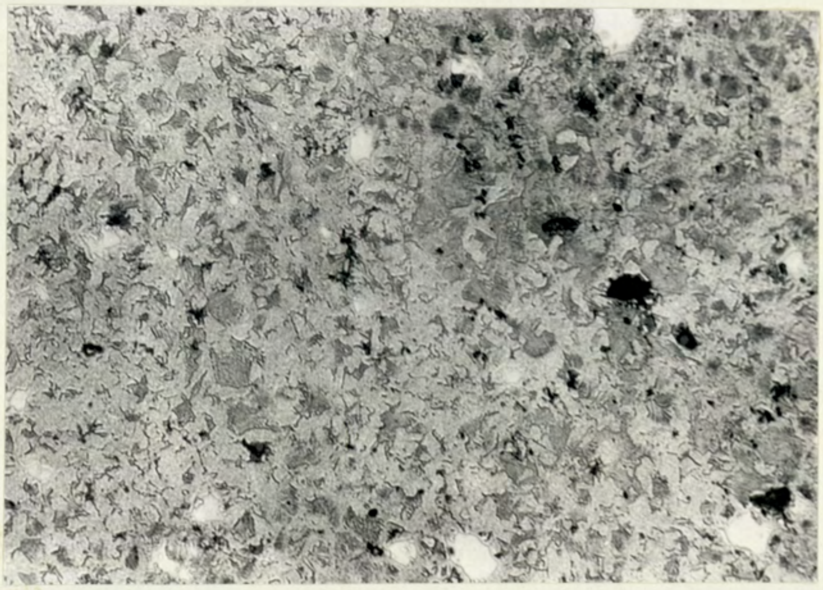


Figure 78

.6%C 2%Cu 2%Ni

(Mag 180 x 4)

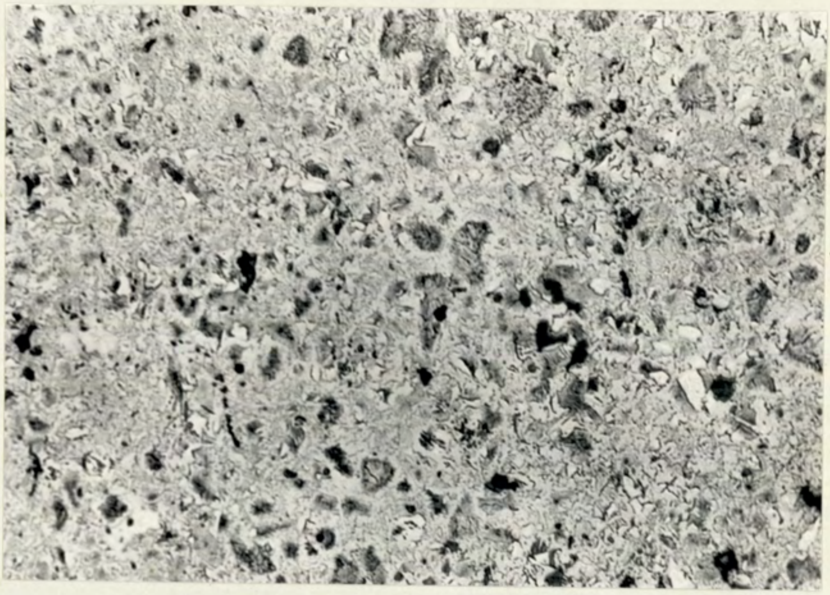


Figure 79
.6%C 5%Cu 2%Ni
(Mag 180 x 4)

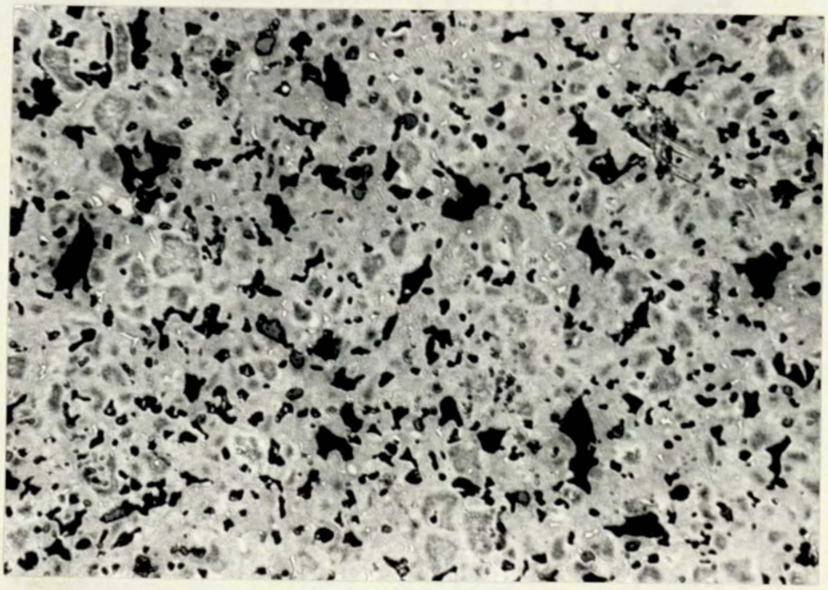


Figure 80
.6%C 8%Cu 2%Ni
(Mag 180 x 4)

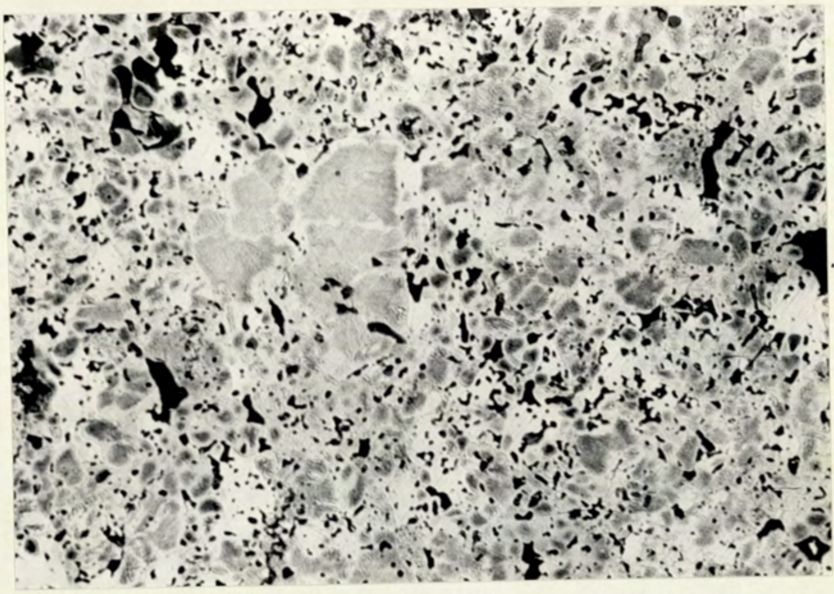


Figure 81
.6%C 2%Cu 5%Ni
(Mag 180 x 4)

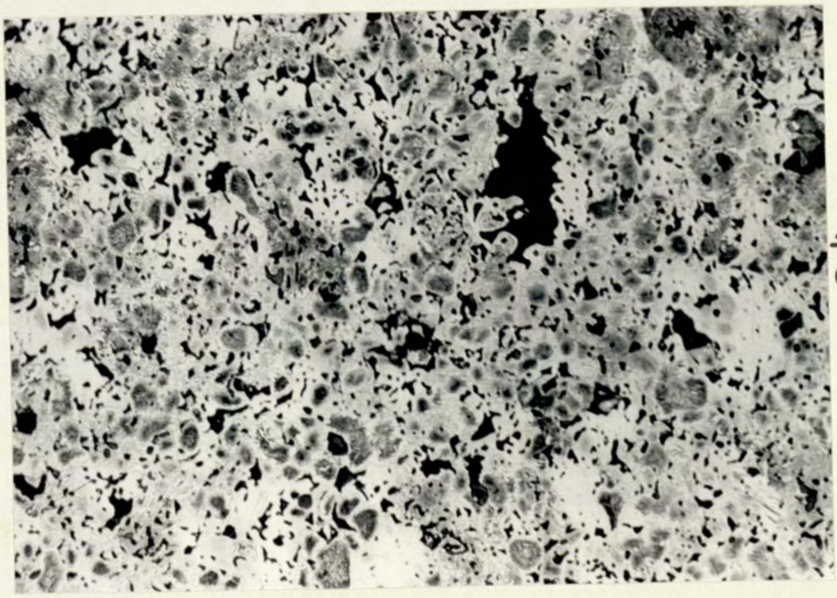


Figure 82
.6%C 5%Cu 5%Ni
(Mag 180 x 4)

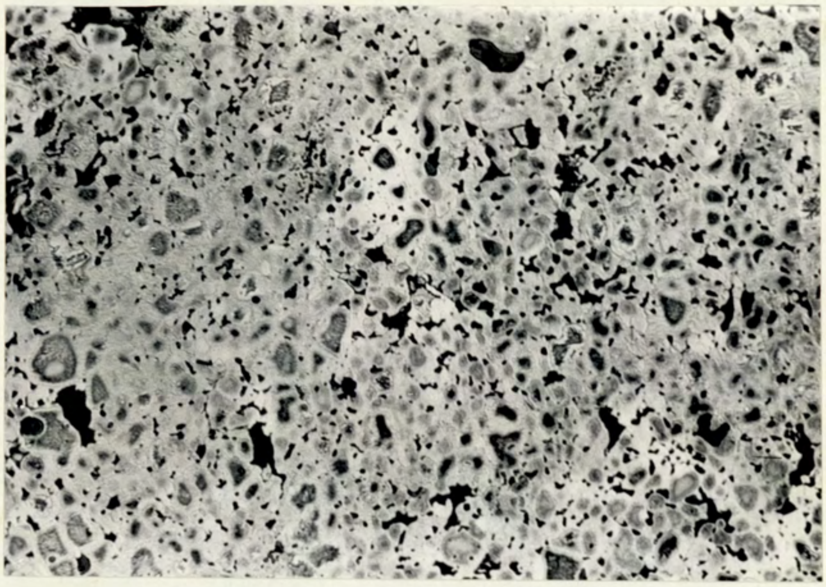


Figure 83
.6%C 8%Cu 5%Ni
(Mag 180 x 4)

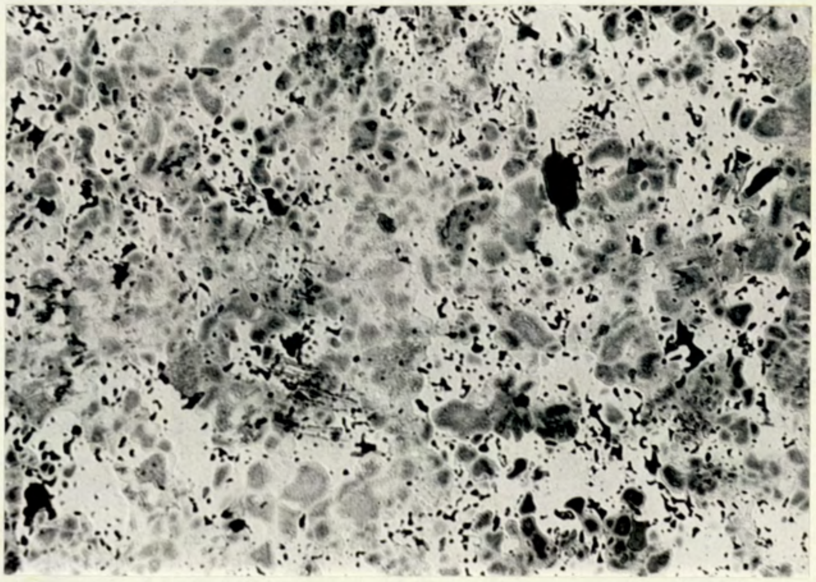


Figure 84
.6%C 2%Cu 8%Ni
(Mag 180 x 4)

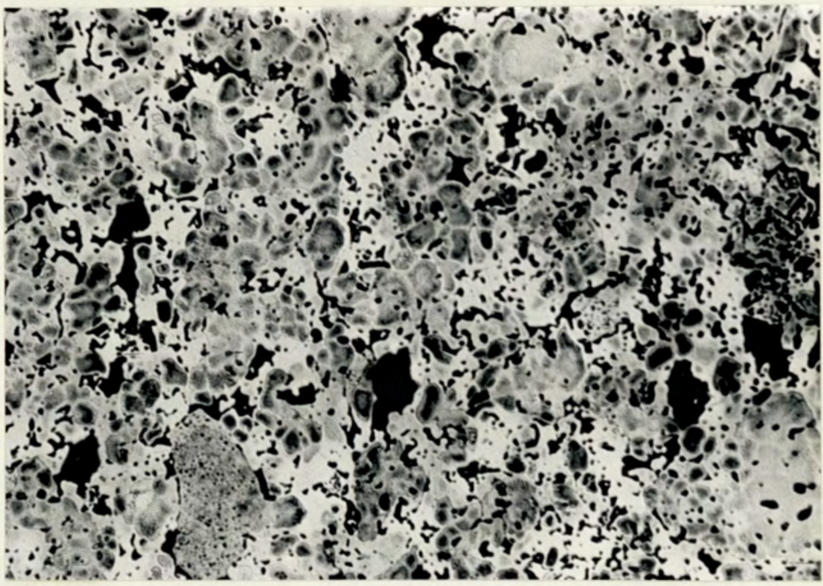


Figure 85
.6%C 5%Cu 8%Ni
(Mag 180 x 4)

Figure 86
.6%C 8%Cu 8%Ni
(Mag 180 x 4)

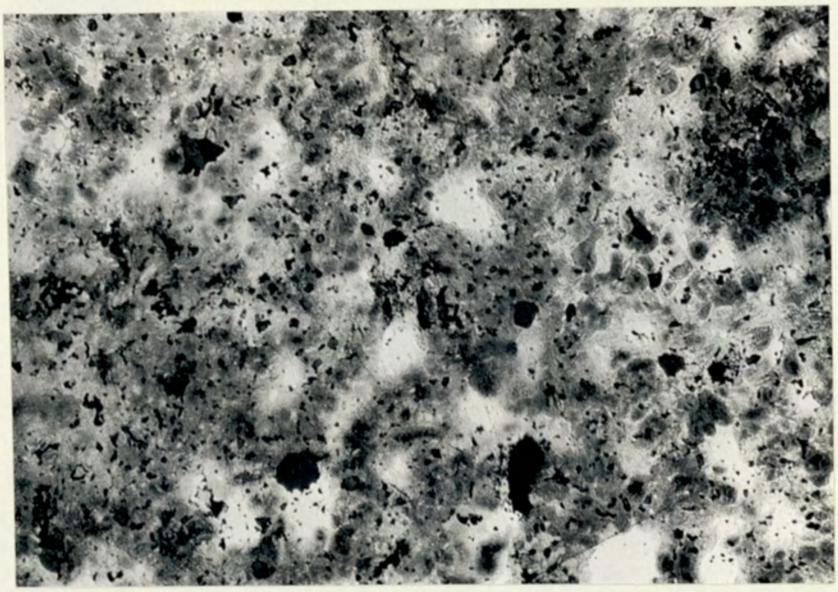


Figure 87
.9%C 2%Cu 2%Ni
(Mag 180 x 4)

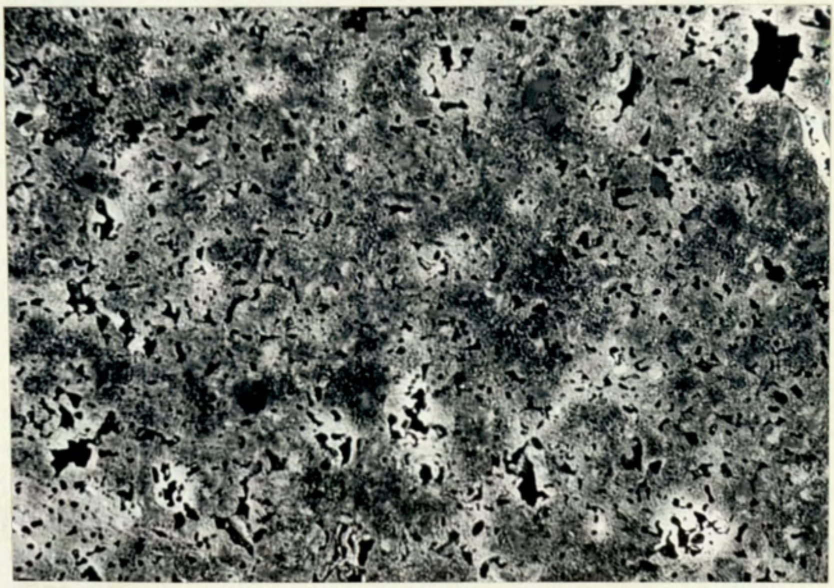


Figure 88
.9%C 5%Cu 2%Ni
(Mag 180 x 4)

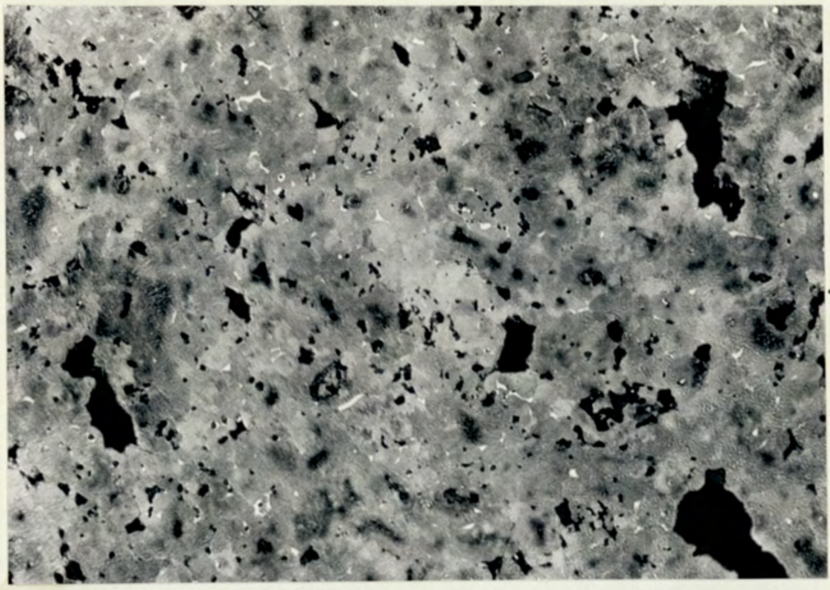


Figure 89
.9%C 8%Cu 2%Ni
(Mag 180 x 4)

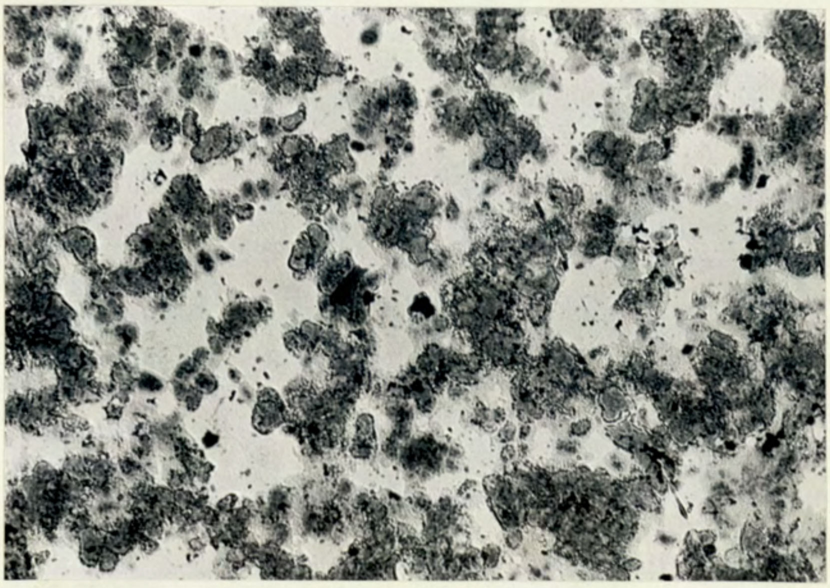


Figure 90
.9%C 2%Cu 5%Ni
(Mag 180 x 4)

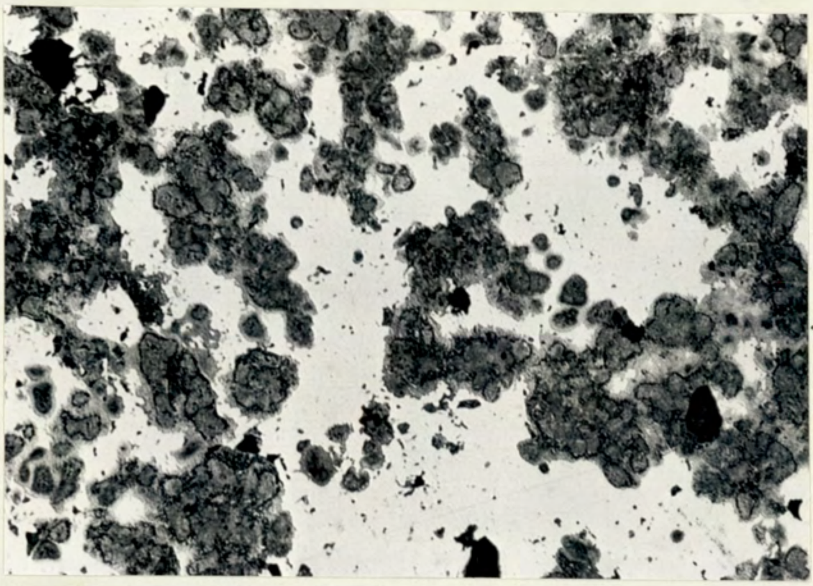


Figure 91

.9%C 5%Cu 5%Ni

(Mag 180 x 4)

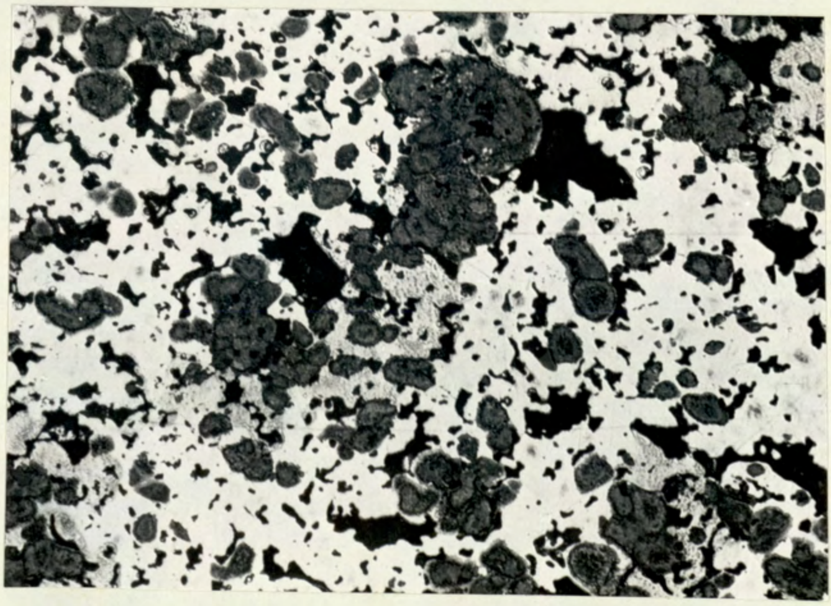


Figure 92

.9%C 8%Cu 5%Ni

(Mag 180 x 4)

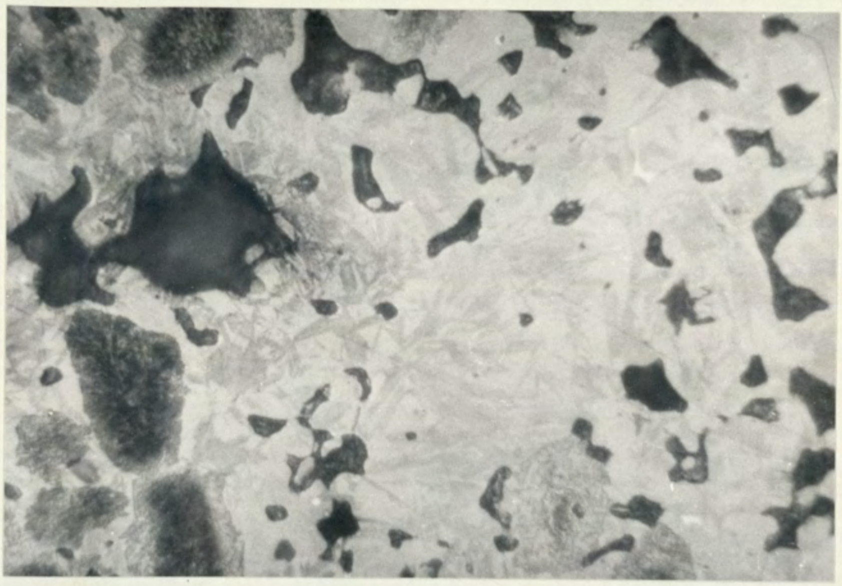


Figure 93
.9%C 8%Cu 5%Ni
(Mag 420 x 4)

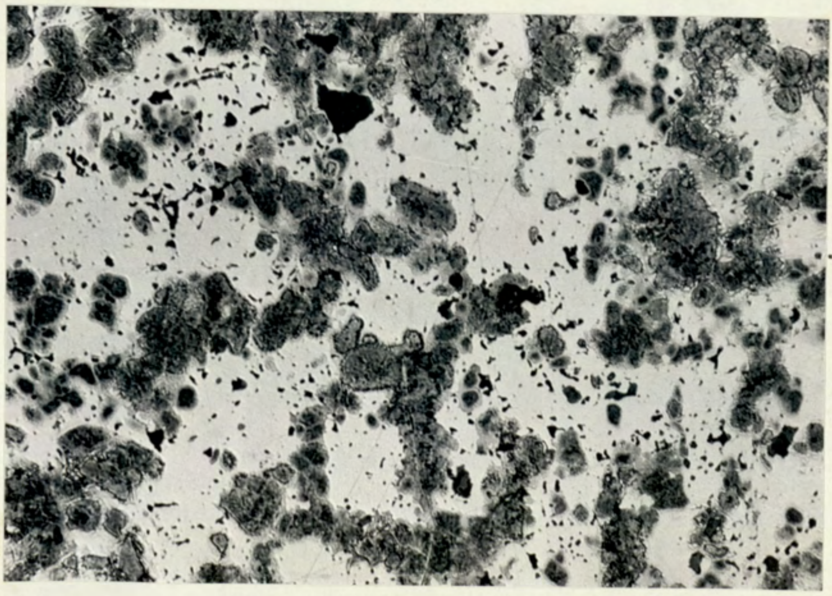


Figure 94
.9%C 2%Cu 8%Ni
(Mag 180 x 4)

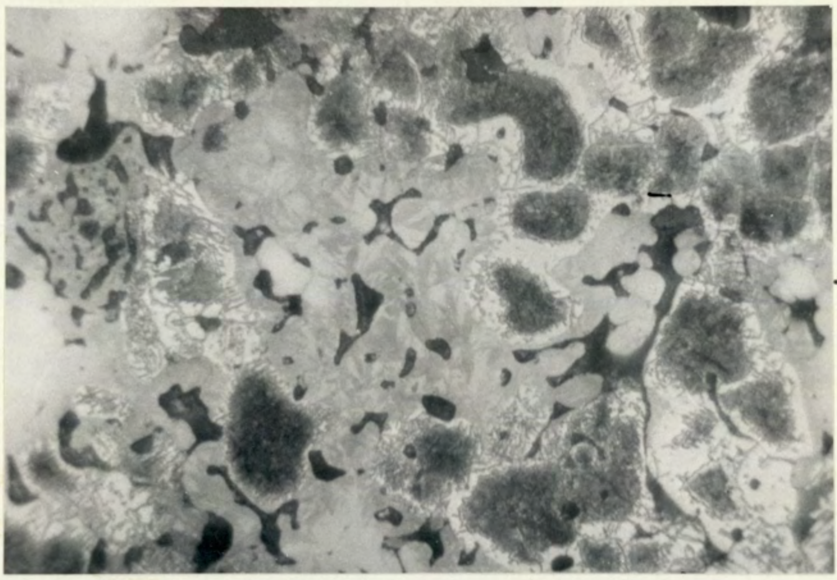


Figure 95

.9%C 2%Cu 8%Ni

(Mag 420 x 4)

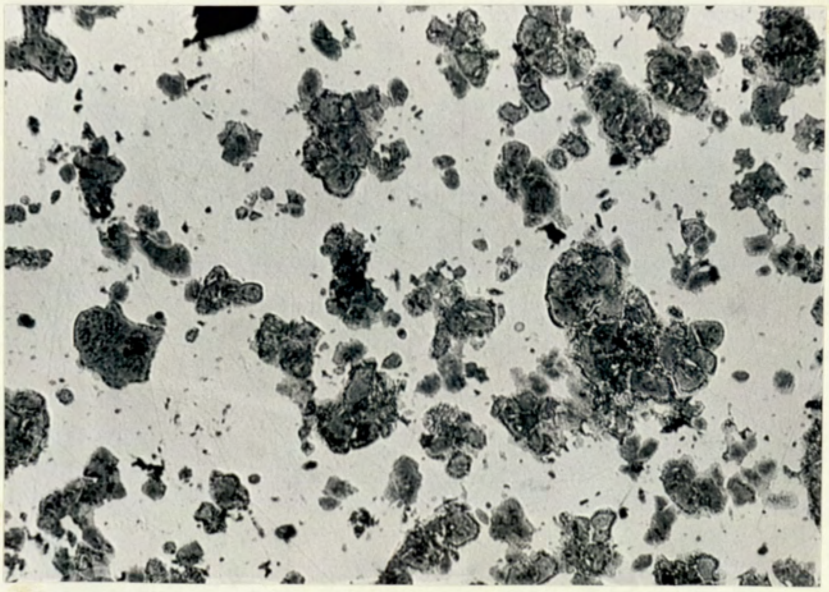


Figure 96

.9%C 5%Cu 8%Ni

(Mag 180 x 4)

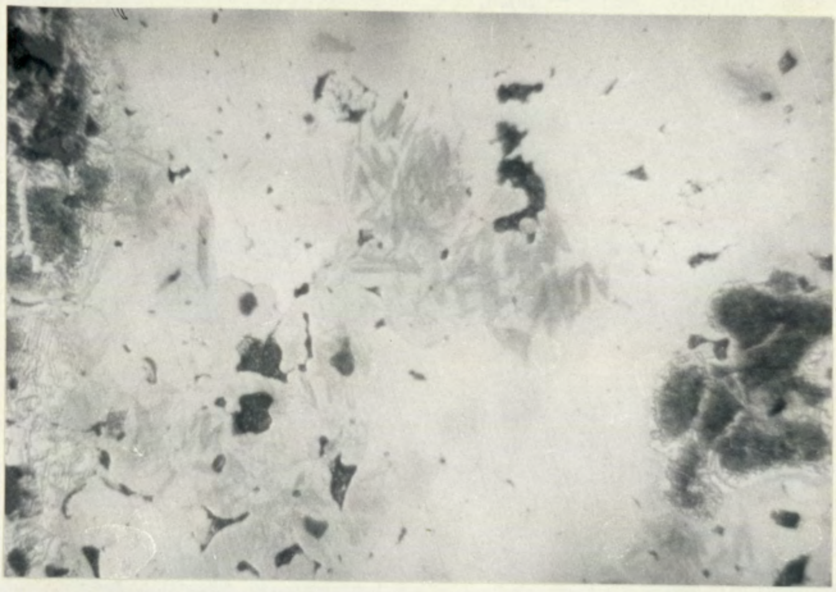


Figure 97
.9%C 5%Cu 8%Ni
(Mag 420 x 4)

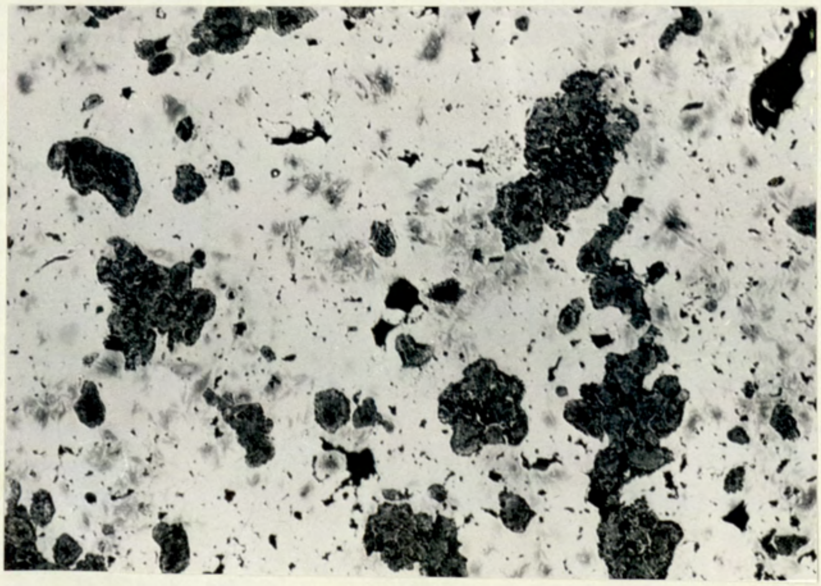


Figure 98
.9%C 8%Cu 8%Ni
(Mag 180 x 4)

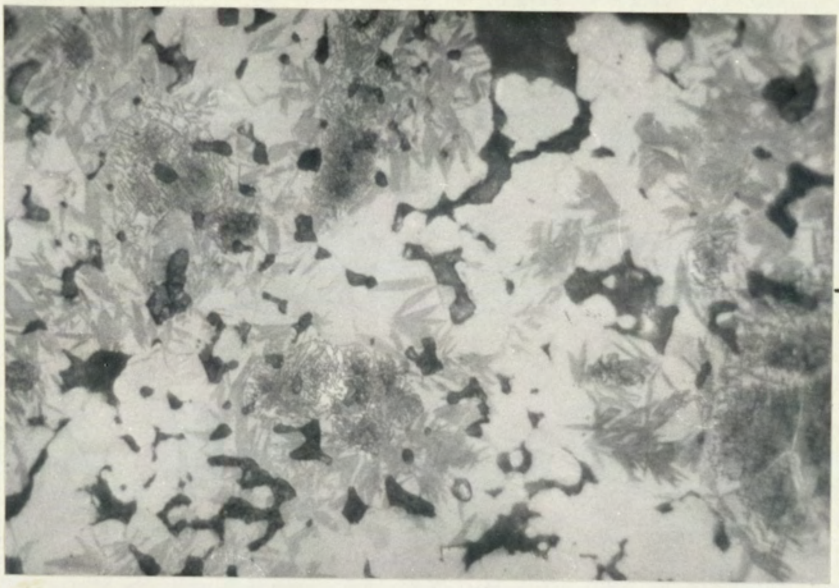


Figure 99

.9%C 8%Cu 8%Ni

(Mag 420 x 4)

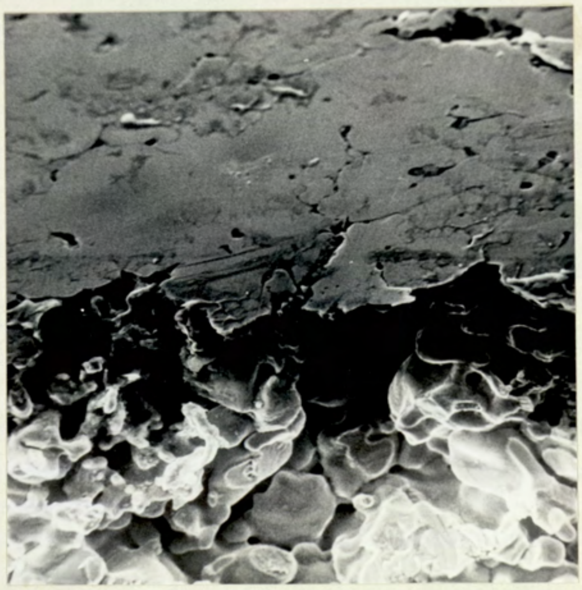


Figure 100

.9%C 8%Cu 8%Ni

(Stereoscan x 810)

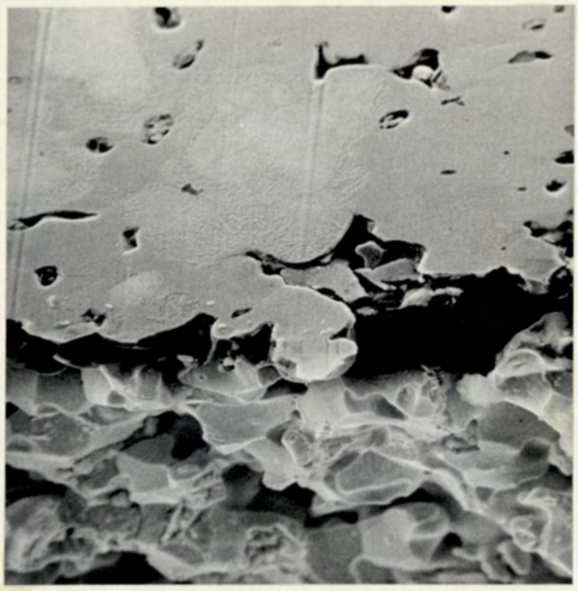


Figure 101

.9%C 8%Cu 5%Ni

(Stereoscan x 870)

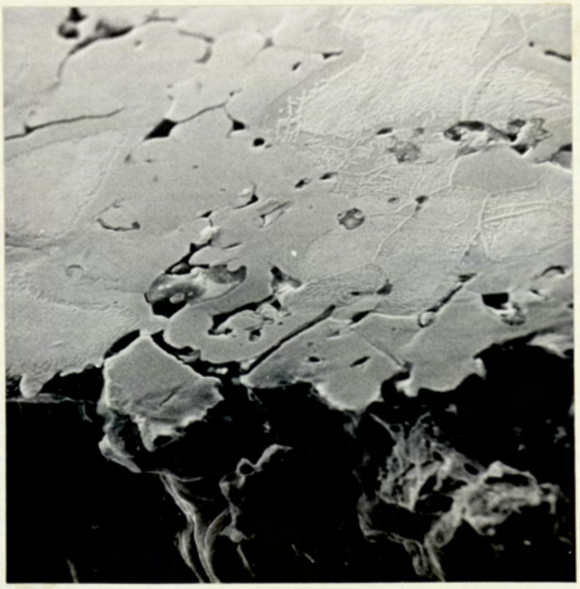


Figure 102

.9%C 5%Cu 8%Ni

(Stereoscan x 930)

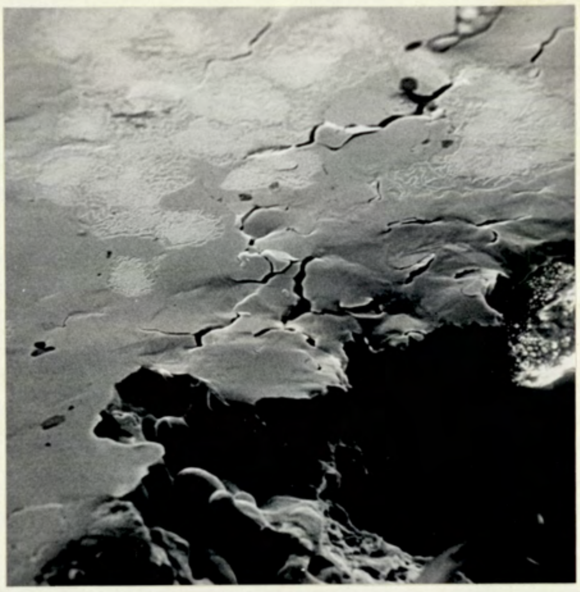


Figure 103

.9%C 2%Cu 8%Ni

(Stereoscan x 930)

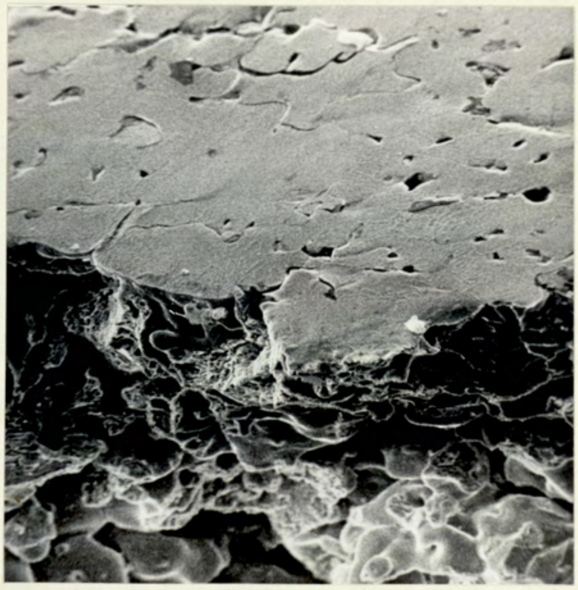


Figure 104

.9%C 5%Cu 2%Ni

(Stereoscan x 700)

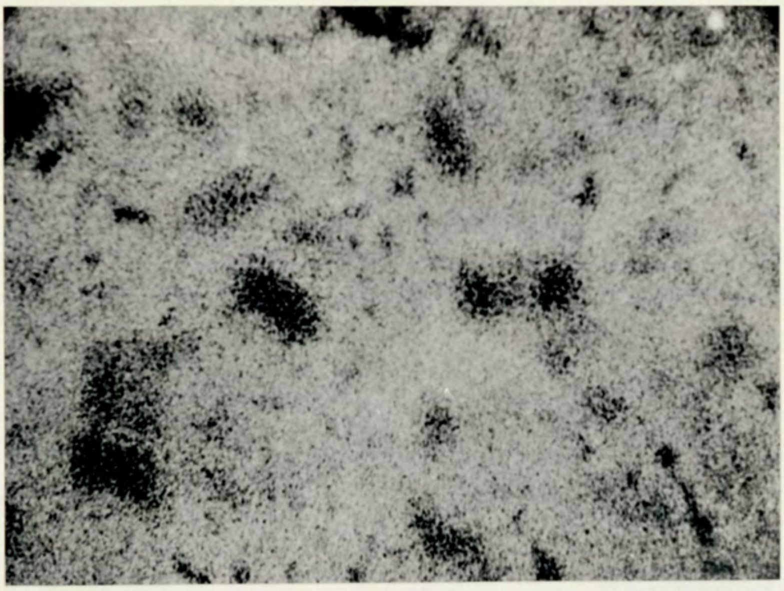


Figure 105

.9%C 5%Cu 2%Ni

(Cu X-ray x 380)

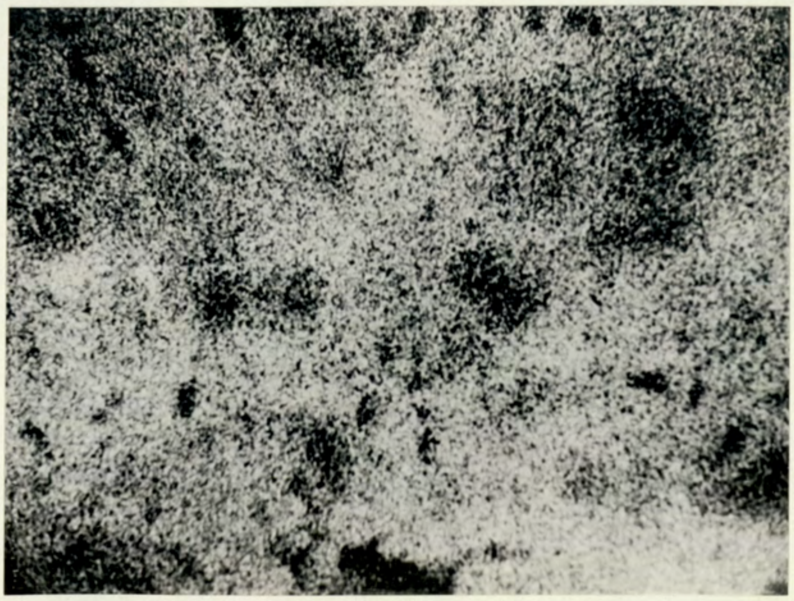


Figure 106

.9%C 5%Cu 2%Ni

(Ni X-ray x 380)

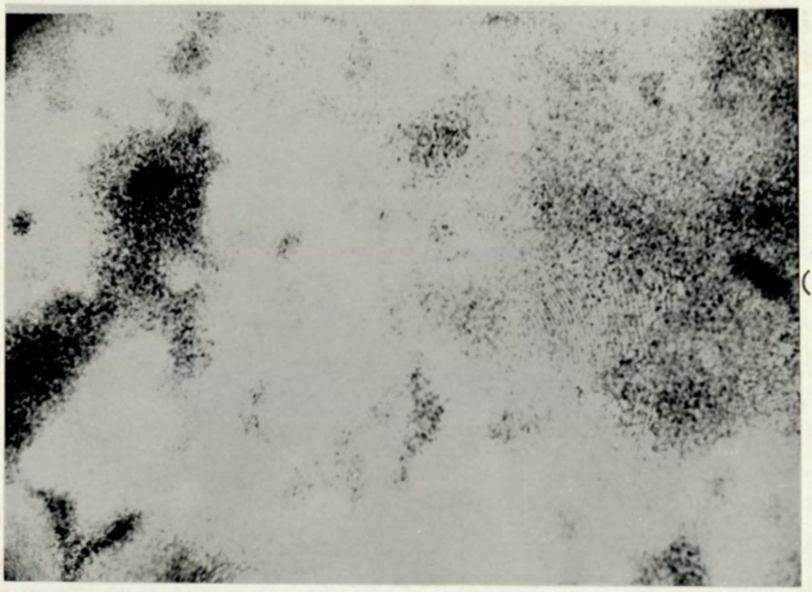


Figure 107
.9%C 8%Cu 8%Ni
(Cu X-ray x 614)

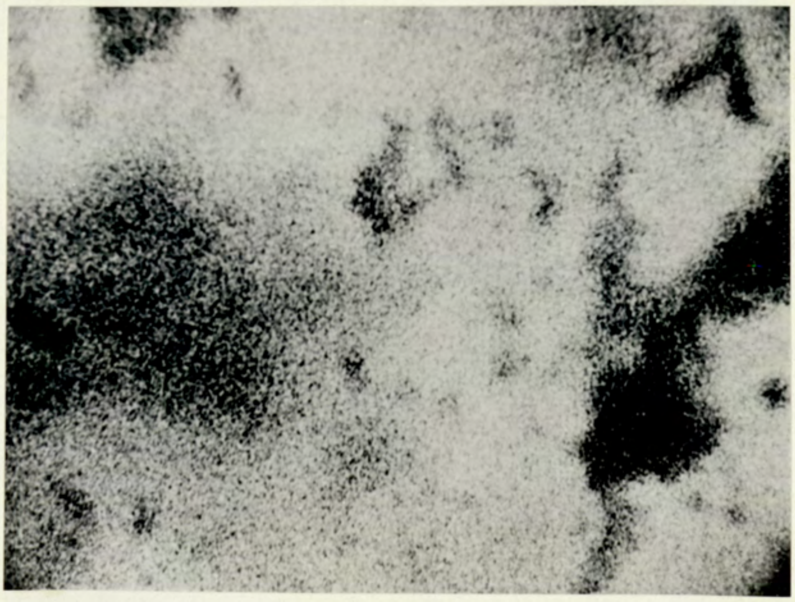


Figure 108
.9%C 8%Cu 8%Ni
(Ni X-ray x 614)

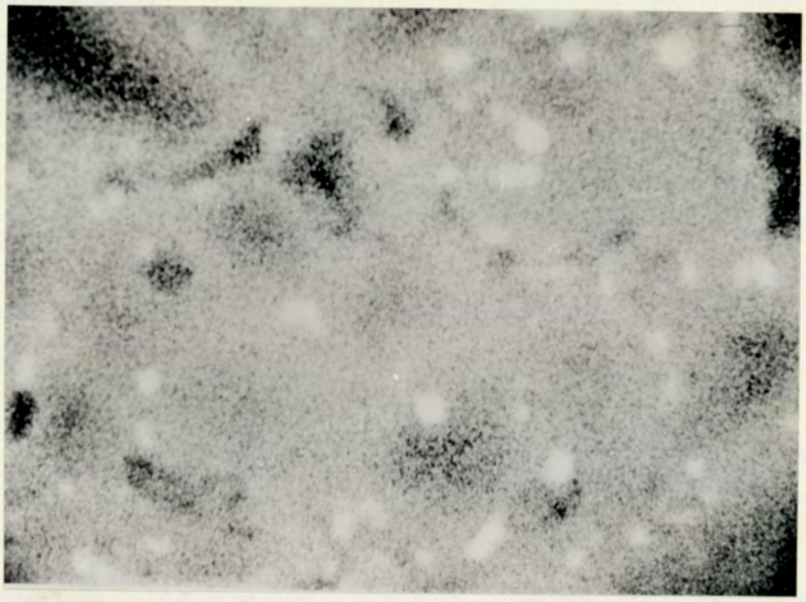


Figure 109

.9%C 8%Cu 5%Ni

(Cu X-ray x 614)

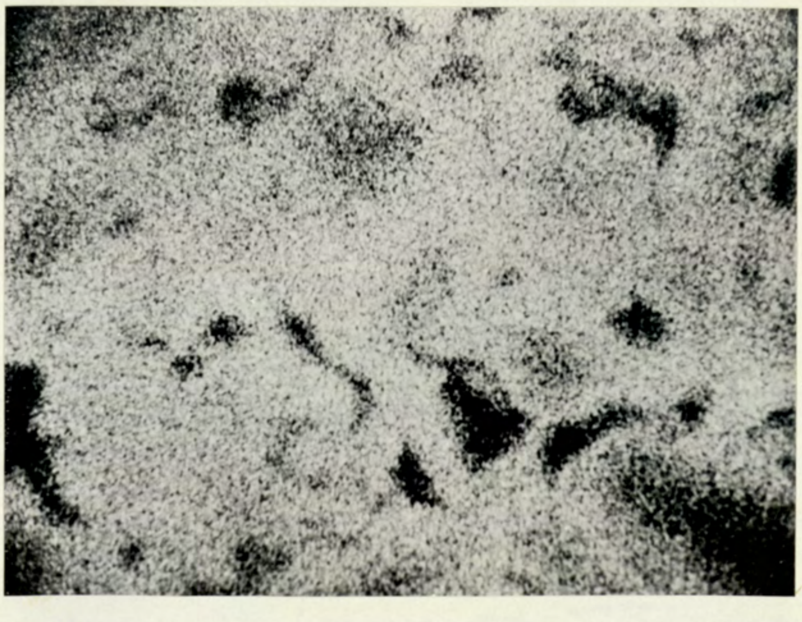


Figure 110

.9%C 8%Cu 5%Ni

(Ni X-ray x 614)

Figure 111

.9%C 5%Cu 8%Ni

(Cu X-ray x 1053)

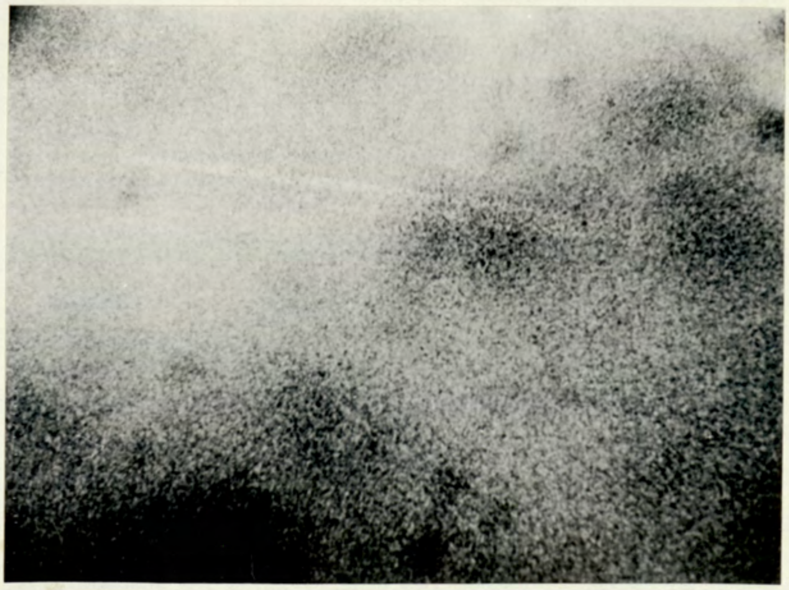


Figure 112

.9%C 5%Cu 8%Ni

(Ni X-ray x 1053)

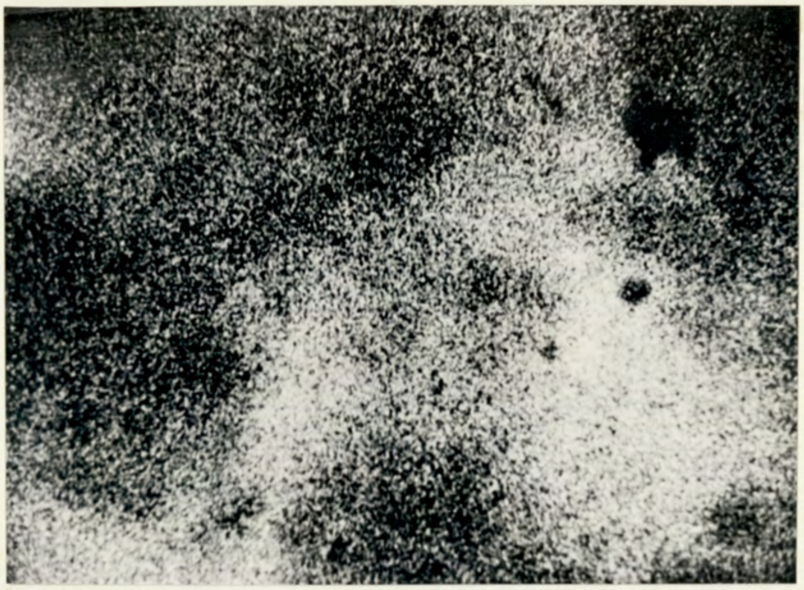


Figure 113

.9%C 2%Cu 8%Ni

(Cu X-ray x 380)

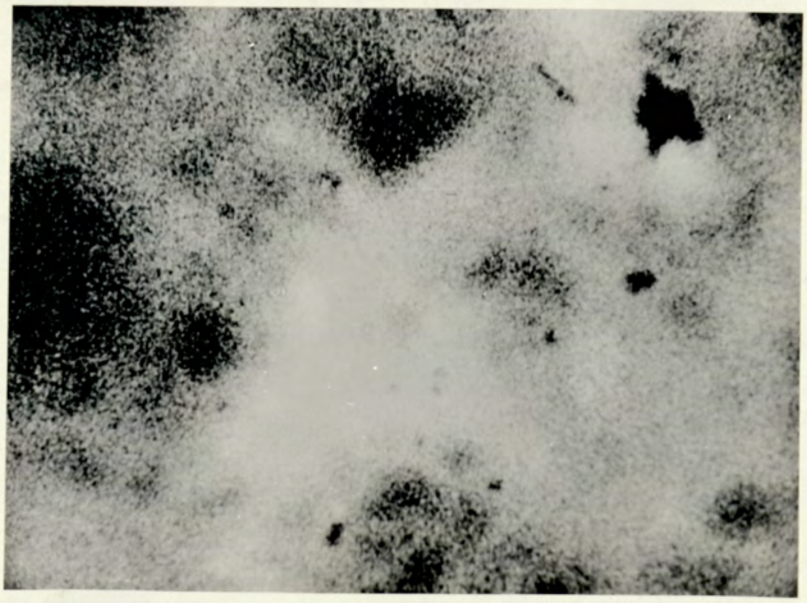


Figure 114

.9%C 2%Cu 8%Ni

(Ni X-ray x 380)

iii Microprobe Analysis

X-ray photographs showing the distribution of copper and nickel in a randomly selected area from each of the five systems above are shown in Figures 105 - 114.

5.6 Discussion

a Combination of Factorial and Multiple Regression Techniques

In the final regression equations, it can be seen that some of the effects shown to be significant by the analysis of variance are not included, and there are a number of possible explanations for this:

i There is built into the regression programme a significance test which eliminates effects which do not significantly enhance the explanation of variation predicted by the presence of terms already in the equation. In the analysis of variance, each main effect and interaction is examined in isolation and not in combination with the other effects.

ii It was considered necessary to include additional squared effects in the regression analysis since published literature indicates some non-linear effects. The inclusion of some of these squared terms in the final equations may have resulted in the elimination of other effects. These squared terms were not included in the factorial analysis since they would have significantly increased the complexity of the design and the analysis, particularly when dealing with interactions.

b Analysis of Transverse Rupture Strength

i Factors Affecting Strength

The strength of any sintered material depends on two major factors:

- the number and area of interparticle bonds which are in turn dependent upon particle characteristics (size and shape), density and the chemistry of the system
- the strength of the individual particles themselves which is largely governed by the presence of alloying elements

ii Effect of Density

The sintered strength regression equation predicts a linear increase in TRS with increasing "as pressed" density, and strengthening appears to be a result of increased bond area since microexamination of the sintered structure indicates an overall reduction in porosity at increasing levels of "as pressed" density. This increased bond area at higher density levels is primarily produced during compaction as a result of the increased extent of cold welding at point contacts between powder particles.

The graphical representation shown in Figures 53 - 56 indicate less pronounced strengthening with increasing density levels at high alloy contents. This is a result of the increased effect of the various alloy interactions as the alloy content increases, which could be detrimental.

iii Effect of Alloying Elements

Copper

Limited published information concerning the sintering of iron-copper alloys[8] suggests that strengthening is a result of two mechanisms:

- the production of a solution strengthened structure due to copper diffusion into the iron particles
- enhancement of sintering by the presence of liquid copper/iron alloy

The limited copper X-ray photographs obtained using the microprobe analyser indicate some solution of copper in the systems examined and micro-examination of the simplest system, viz Fe 2%Ni .3%C at 2%Cu and 5%Cu levels indicates the absence of free undissolved copper. Although solution strengthening is reported as a possible strengthening mechanism, its extent depends on the rate of cooling, since under equilibrium conditions only ~.1% copper is soluble in iron at room temperature, dissolved copper being precipitated as the temperature falls. It is claimed that this precipitated copper stains brown when nital is used as an etchant but since all the samples examined contained carbon and hence pearlite, it is impossible to detect this precipitated copper metallographically.

Sintering was carried out at 1120°C, ie above the melting point of copper, and hence liquid copper, or liquid copper with dissolved iron, must obviously be present at some stage during sintering and the presence of smooth rounded interfaces in the Stereoscan photographs, particularly at high copper levels, Figure 101, is a clear indication of the action of a liquid phase. It is difficult to quantify separately the enhancement of strength due to copper diffusion and the presence of a liquid phase since diffusion is greatly accelerated when liquid copper is present.

Nickel

The mechanisms involved when sintering iron-nickel alloys have been examined in detail by Fedorchenko and Ivanova[32] who propose an activated sintering mechanism resulting in increased strength. This proposed mechanism was developed by considering the predominant direction of diffusion flow, ie Kirkendal effect, when sintering iron-nickel alloys. The partial diffusion coefficient of nickel into iron is greater than that of iron into nickel and hence on the surface of the nickel a thin layer of diffusion porosity is created. This facilitates creep during sintering, produces rapid densification, ie shrinkage, with a resultant increase in strength. Although a simple iron-nickel system was not analysed, metallographic examination of the Fe 2%Cu .3%C system at 2%Ni, 5%Ni and 8%Ni seems to indicate a reduction in the number and size of pores, particularly at 8%Ni and examination of dimensional change data indicates a correspondingly large shrinkage at 8% nickel. The relevant curve in Figure 56 also shows a corresponding increase in transverse rupture strength.

A solution hardening mechanism is not considered in published literature but in view of the limited nickel diffusion indicated by the X ray photographs it would seem a probable mechanism.

Carbon

During the sintering of iron-carbon alloys, carbon diffuses rapidly into the iron lattice, resulting, on cooling, in the production of pearlite in the particles and inter-particles bonds and an increase in strength. Maximum strengthening occurs at ~ .9%C, ie eutetoid composition, and beyond this level the strength decreases due to the production of an intergranular carbide network. Although a simple iron carbon system was not analysed, micro-examination of the Fe 2%Cu 2%Ni system indicates increased pearlite as the carbon content increases from .3% - .9% and the relevant curve in Figure 55 shows a corresponding increase in TRS.

iv Effect of Interactions

The inclusion of a number of negative and hence weakening effects in the regression equation, viz $(\text{copper})^2$ nickel x copper, nickel x carbon, copper x carbon, density x copper, indicates a highly complex situation when considering the variation of all four variables simultaneously over the range examined. The graphical representation of the equation shows clearly the dependence of the effect of each variable, particularly the effect of alloying elements, on the levels of the other variables. Although it is difficult to discuss each of these terms in isolation, some limited discussion is possible.

$(\text{Copper})^2$ and Copper x Density

The weakening effect of copper at high concentrations and hence the inclusion of a negative $(\text{copper})^2$ effect has been related to the production of excess undissolved copper which forms weak areas throughout the structure. Although a simple iron copper system was not examined metallographic examination of the Fe 2%Ni .3%C system at 2%Cu, 5%Cu and 8%Cu, showed the presence of undissolved copper at the 8% level only and the relevant curve in Figure 54 indicates a maximum strength at ~6.5% copper. The production of weak areas of undissolved copper can also be related to the inclusion of a negative density x copper interaction since at high density diffusion is accelerated due to the closer physical proximity of the iron and copper particles and is less reliant on concentration gradients for diffusion. Hence, a similar negative effect arising from high copper can be produced using a lower copper level combined with a high density.

$(\text{Copper})^2$ is only a mathmatical method of indicating non-linearity in linear multiple regression analysis and such a term is difficult to discuss in physical terms. Clearly, the situation when using copper additions is extremely complex with a number of different effects occurring at various stages during sintering and where the overall effect is summarised mathmatically by the copper, $(\text{copper})^2$ and copper density interaction terms.

Copper x Carbon

The inclusion of a negative copper x carbon interaction can be related to published work which suggests that carbon reduces the rate of copper diffusion,[31] resulting in less solution hardening and more undissolved copper. Microexamination of Fe 2%Ni 8%Cu at .3%C, .6%C and .9%C, viz Figures 71, 80, 89, indicate an increase in undissolved copper as carbon increases and the production of a maximum strength in the .9%C alloy at 4.2% copper shows reasonable agreement with published work which indicates a maximum strength at ~5% Cu in an iron 1% C alloy.

Nickel x Carbon

Both nickel and carbon are austenite stabilisers and the presence of a negative nickel x carbon interaction is probably due to the production of areas of weak retained austenite. Nickel is also slow to diffuse into iron at the sintering temperature used, viz 1120°C, and hence local nickel rich areas may be produced, resulting not only in the retention of austenite but also the production of brittle transformation products on air cooling. The photomicrograph of the iron 2% Cu 8%Ni .9%C alloy indicates the presence of both retained austenite and asicular transformation products.

Copper x Nickel

The photomicrograph of the iron 8%Cu 8%Ni .3%C alloy, Figure 77, indicatesthe presence of both retained austenite and undissolved copper, which weaken the structure and probably account for the negative copper x nickel interaction.

Although the above discussion accounts to a certain extent for the inclusion of the negative effects in the regression equation, it is clear that the magnitude of these effects and hence the resultant strength, is highly dependent on the specific levels of the alloying elements used. A most significant effect shown clearly in all the graphs is the detrimental effect of high copper, high carbon, high nickel combinations. A number of mechanisms appear to be involved and micro examination indicates the presence of undissolved copper, retained austenite and asicular transformation products. However, more detailed examination probably on the microprobe analyser is required before specific mechanisms can be identified.

c Analysis of Dimensional Change

The dimensional change of iron base alloys during sintering under specified conditions, ie time, temperature and atmosphere, is governed to a large extent by three major factors:

i the effect of alloying elements diffusing into the iron lattice

ii the effect of interactions between alloying elements

iii the reduction of porosity

i Effect of Density

It is well established in published literature that, for a simple iron system, the effect of increasing density on dimensional change during sintering is to reduce shrinkage, since there is a reduction in free pore surface area as the "as pressed" density increases. The situation is more complex in the systems examined since they contain alloying elements, some of which have a significant interaction with density. However, to a certain extent, the predicted results shown graphically in Figures 57, 61, 65 agree with the established mechanisms in that in systems which show shrinkage during sintering, viz low copper/high nickel, there is a reduction in shrinkage as density increases. These graphs also indicate that in systems showing expansion, ie 5%Cu and 8%Cu, there is an increase in expansion as density increases. Although a minimum point is shown in Figure 65, it is not very pronounced and the overall effect of density is as described above. It is difficult to account for this minimum other than by relating its occurrence to the presence of a variety of alloying elements.

ii Effect of Alloying Elements

Copper

The general effect of increasing the copper content of sintered iron base materials is to promote expansion as a result of copper diffusing into the iron lattice producing a substitutional solid solution of increased lattice parameter. This expansion is counteracted to a limited extent by the solution of iron in liquid copper and its subsequent reprecipitation.

Although a simple iron-copper system was not analysed, the graphical representation of all three dimensional change equations indicates increased expansion with increasing copper content and the limited microprobe analysis and optical metallography indicates copper solution.

Carbon

During the sintering of a plain iron-carbon system carbon diffuses into an interstial position in the iron lattice, producing expansion which is a maximum at .8%C, ie eutectoid content.

The predicted effect of increased carbon on all three dimensions measured is to reduce expansion in systems showing expansion and to increase shrinkage in systems showing shrinkage. This apparent disagreement with published literature is due to the presence of copper and nickel and the inclusion of a negative copper x carbon and copper x nickel interactions, the magnitude of which are clearly greatest at high alloy contents. The possible mechanisms involved are discussed in iii below.

Nickel

It is widely reported in published literature that the addition of nickel to iron base sintered materials results in shrinkage during sintering. Although a simple iron-nickel system was not examined, the increase in shrinkage with increasing nickel content shown by the iron-nickel 2%Cu .3%C system appears to confirm this shrinkage effect.

Detailed work carried out by Fedorchenko and Ivanova indicates that the mechanism involved is one of activated sintering (see 5.6,b,iii, Nickel). Although some of the optical metallography indicates reduced porosity in the iron 8%Ni 2%Cu .3%C alloy compared with the iron 2%Ni 2%Cu .3%C system, there is no direct evidence to identify the detailed mechanisms involved. A positive nickel effect is included in equation 4 and its presence may be related to the limited bulk diffusion of nickel resulting in the production of an iron-nickel solid solution and hence expansion of the iron lattice. This, to a limited extent, counteracts some of the shrinkage produced by the proposed activated sintering mechanism, but since bulk diffusion of nickel at the sintering temperature used, viz 1120°C is very slow, the magnitude of this proposed mechanism is likely to be small.

iii Effect of Interactions

The three dimensional change equations indicate a complex situation in which interactions between the four variables examined have a predominant effect. It is difficult to discuss each interaction from every equation in isolation but as with the analysis of the strength equation a limited discussion is possible and some comparison with published work can be made.

Copper x Carbon

The graphical representation of all three dimensional change equations indicates a marked reduction in the expansion produced by copper additions as the carbon content increases, thus accounting for the negative coefficient allocated to the copper x carbon interaction. Two mechanisms are proposed in published literature to explain this effect:

- reduced copper diffusion due to carbon segregation to the grain boundaries[28]
- the production of an iron-copper-carbon ternary eutectic which increases the amount of liquid phase present resulting in yielding of the sintered iron skeleton under residual surface tension, and hence producing a sudden shrinkage[8,9,10]

The limited metallography carried out in this investigation confirms reduced copper diffusion as the carbon content increases, see photomicrographs, Figures 71, 80, 89. However, there is no significant evidence of carbon segregation at grain boundaries and there is no evidence of an iron-copper-carbon ternary eutectic in any of the alloys examined.

Nickel x Carbon

Since it has been established that carbon reduces the extent of diffusion of copper, it appears likely that the bulk diffusion of nickel may also be reduced by the presence of carbon, thus inhibiting the formation of an iron-nickel solid solution. This would in turn reduce any expansion and hence account for the negative coefficient allocated to the nickel x carbon interaction.

Density x Carbon, Density x Nickel, Density x Copper

The mechanisms by which each of the individual alloying elements, viz carbon, copper and nickel, can produce expansion during sintering have already been reviewed. Increasing density increases the physical proximity of the iron particles and the alloying elements, thus increasing the size of the iron/alloying element bond. This accelerates the rate of alloy diffusion which becomes less reliant on concentration gradients and accounts for the positive coefficient allocated to each of the interactions.

Nickel x Copper x Carbon

It is difficult to predict a possible mechanism to account for the positive coefficient allocated to this complex three factor interaction in all three dimensional change equations other than by relating it to a combined solid solution effect due to the diffusion of all three elements. However, the coefficient is small and hence the effect of this interaction is relatively small and is outweighed by the majority of the other factors in the equations.

iv Effect of Nickel on Copper Expansion

It has been suggested in published literature that the expansion resulting from the presence of copper can be reduced by the addition of nickel[8] and from Figures 60, 64, 68, it can be seen that this is the case in a number of the alloys examined. However, in some alloys, viz iron-nickel 5%Cu .9%C, iron-nickel 8%Cu .6%C, iron-nickel 8%Cu .9%C, there is a marked increase in expansion with increasing nickel until a critical nickel level is reached. The limited X-ray photographs indicate a slight increase in the diffusion of nickel at high copper levels, thus producing more extensive solution and hence expansion, until some critical nickel content is reached, at which point shrinkage due to the activated sintering mechanism proposed for nickel additions is predominant.

v Fractography

The stereoscan examination indicates that the predominant fracture path is across the pores, thus supporting the major strengthening effect predicted for increased density. There is also some indication that the fracture passes around the pearlite areas. In the high copper alloys examined the presence of some rounded edges associated with the fracture indicates the presence of a liquid phase at some stage during sintering. However, the systems are clearly complex and more detailed examination of each system is required in order to positively identify the fracture processes involved.

d Optimum Combination of Strength and Dimensional Change

Although the experiment was not designed to predict a system producing the optimum combination of strength and dimensional change, it is possible to select such a system from those examined by simple inspection of the predicted results.

The strength equation 1 predicts a similar maximum strength in a number of alloys and these are listed below, together with the relevant predicted dimensional change.

Alloy System	Predicted TRS $\times 10^3$psi (MN/m^2)	Predicted Dimensional Change		
		h	B	l
1 Fe .9%C 2%Cu 2%Ni 88% density	126.6 (872)	+.11	+.15	+.17
2 Fe .9%c 5%Cu 2%Ni 88% density	127.3 (876)	+.72	+.67	+.62
3 Fe .3%C 2%Cu 8%Ni 88% density	127.3 (876)	-.31	-.29	-.24
4 Fe .6%C 2%Cu 8%Ni 88% density	123.3 (850)	-.38	-.35	-.30
5 Fe .9%C 2%Cu 8%Ni 88% density	126.8 (872)	-.45	-.41	-.37

Of the systems examined, the best combination of high strength and minimum dimensional change is the iron .9%C 2%Cu 2%Ni system at 88% theoretical density. The predicted dimensional changes are equivalent to .0011 in/in, .0015 in/in and .0017 in/in for ΔH, ΔB and Δl respectively and hence do not present any serious problems in controlling close dimensional tolerances.

This system has a further advantage of a lower cost than the other four since the expensive alloying elements, ie nickel and copper, are at their low levels.

From the results obtained in this investigation, it is clear that a large number of alloys would not be suitable for practical use because of either their low strength and/or excessive dimensional change during sintering. The results also indicate similar levels of strength in systems containing 8%Ni 2%Cu and those containing 2%Ni and up to 5%Cu. Nickel is approximately twice as expensive as copper and hence from an economic viewpoint nickel content should be kept to a minimum.

2½-3

Taking all this into account, it appears that the most likely system to be used in practice is the Fe 2%Ni Cu C system and the following equations were obtained using the first 36 observations.

$$\text{TRS} = -317.45 + 4.94\,(\text{density}) + 4.64\,(\%\text{Ni} \times \%\text{C}) + 1.41\,(\%\text{Cu} \times \%\text{C}) - 3.10\,(\%\text{Ni})$$

Residual Error = 8.52

Multiple Correlation Coefficient = .906

$$\%\Delta h = -.602 + .571\,(\%\text{C}) + .550\,(\%\text{Cu}) - .409\,(\%\text{Cu} \times \%\text{C}) - .099\,(\%\text{Ni})$$

Residual Error = .273

Multiple Correlation Coefficient = .961

$\% \Delta b$ = -.228 + .401 (%Cu) + .041 (Density x % C) - 3.107 (%C) - .314 (%Cu x %C) - .097 (%Ni)

Residual Error = .158

Multiple Correlation Coefficient = .977

$\% \Delta l$ = - 2.327 + .030 (density) + .353 (%Cu) - .273 (%C x %Cu) - .121 (%Ni)

Residual Error = .227

Multiple Correlation Coefficient = .951

The equations are much simpler than those obtained using all the data but they clearly only relate to the Fe 2%Ni, Cu, C system at 80%, 84% and 88% theoretical density. The number of terms is reduced since complex squared terms and three factor interactions were omitted, the rest of the terms being rejected by the built-in significance testing at the 5% significance level.

5.7 Conclusions

a Density has the largest effect on strength of the four variables examined. The mechanism involved is one of increased area of bonding and an increase in the number of interparticle bonds as the density increases.

b Reported strengthening mechanisms for the addition of each of the alloying elements are confirmed, ie

  i copper enhances strength by a solution hardening mechanism and increased sintering rate due to the presence of a liquid phase

X

ii carbon enhances strength by the production of pearlite in slow cooled structures

iii nickel enhances strength by inter-diffusion during sintering, resulting in rapid elimination of porosity and hence growth of inter-particle bonds

c A number of interactions between the four variables have a significant effect on strength, particularly interactions between the alloying elements used, viz copper, nickel and carbon, and hence the magnitude of the effect of each alloying element is highly dependent on the levels of the other elements.

d Although increase in alloying elements initially improves strength, the combination of high levels of copper, nickel and carbon results in a dramatic decrease in strength.

e Dimensional change is highly dependent upon interactions and hence the magnitude of the effect of each of the variables examined is dependent upon the levels of the other variables. However, a number of general conclusions can be made:

i increased density reduces shrinkage in alloys showing shrinkage and increases expansion in alloys showing expansion

why.

ii increased carbon promotes shrinkage in the alloys examined iii increased copper promotes expansion

iv increased nickel at low levels of copper promotes shrinkage which is counteracted at medium and high levels of copper

f Of the twenty-seven alloys examined, the optimum combination of high strength and minimum dimensional change during sintering is produced in the iron 2%Cu 2%Ni .9%C system at 88% theoretical density. This system also has a low cost which is only slightly above the minimum cost alloy, viz Fe 2%Cu 2%Ni .3%C.

g The limited fractography indicates the main fracture path is across the pores.

h The use of a statistical approach to the experimental work has offered much clearer interpretation of results than would be possible by a classical approach. This is particularly so in regard to the effect of factor interactions.

5.8 Recommendations for Future Work

a The preliminary analysis outlined in this experiment provides a qualitative assessment of optimum conditions, ie maximum strength and minimum dimensional change during sintering. However, quantitative statistical techniques are available for determining optimum conditions, eg hill climbing, and this appears to be a logical eontinuation of the analysis of the experimental results.

b The experimental work indicates that although nickel is slow to diffuse at 1120°C, it has a strengthening effect almost equivalent to that of copper. Systems containing nickel are usually sintered at ~1250°C in order to improve nickel diffusion and hence a better assessment of the effect of nickel on strength and dimensional stability could probably be obtained by repeating the experiment using a sintering temperature of ~1250°C.

c The mechanisms involved in the sintering of iron-copper alloys are reasonably well established, but those involved when sintering iron-nickel alloys are not. Hence, a detailed examination of these mechanisms is required, preferably using industrial systems.

d Properties such as fatigue strength and fracture toughness of sintered materials are not widely reported in published literature and since sintered components are being used increasingly in more highly stressed situations, a detailed analysis of factors affecting these properties would be beneficial.

e A number of iron powders are available which, manufacturers claim, have the property of reducing expansion when used in the production of sintered iron base alloys containing copper. Since copper is widely used in the production of sintered iron base materials, a detailed assessment of these powders would be useful and an assessment of the mechanisms of growth inhibition could greatly improve the control of dimensions during sintering.

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