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**Ferrous Powders - How Alloying Method Influences Sintering** by  
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## **INTRODUCTION**

The mechanical properties of P/M materials are directly related to their microstructure and the size, distribution, and morphology of the porosity they contain. Alloying additions are made to develop specific material performance characteristics. However, the manner in which the alloys are constituted has a significant effect on the porosity and microstructure of the final sintered product (1,2).

### **Alloying Methods**

Ferrous P/M materials may be classified according to the manner in which the alloy has been constituted:

**Admixed** - The alloying additions are made to the iron powder base in the form of elemental or ferroalloy powders. This is the least expensive and most commonly used alloying method. Since the iron powder base is unalloyed when the mix is pressed, admixed materials retain most of the compressibility of the iron base. The degree of alloying is limited by the diffusivity of the alloying elements in iron at the sintering temperature, and the resulting microstructures are chemically heterogeneous. This type of material is also subject to powder segregation and dusting during handling and pressing.

**Partially Alloyed** - The alloying additions are diffusion bonded to the base iron particles such that the compressibility of the base iron is essentially retained. The powders are highly compressible, and yield heterogeneous sintered microstructures consisting of lightly alloyed particle cores with a continuous network of more highly alloyed interparticle bonds.

**Prealloyed** - The alloying elements, except for carbon, are added to the melt before atomization. This results in homogeneous microstructures and uniform hardness even on a microhardness level. However, solution hardening of the powder particles by the alloy additions decreases the compressibility of the powders compared with admixed and partially alloyed materials.

The above alloying methods are shown schematically in Figure 1 and the advantages and disadvantages of each method are discussed in greater detail elsewhere ( 1 ).

A systems approach is required to optimize each stage involved in the production of a P/M part (3). There are three main steps involved:

- Mixing
- Compaction
- Sintering

P/M alloys are engineered materials and, in order to achieve the best combination of strength and toughness in P/M steels, it is necessary to optimize the properties of both the inter-particle and the intra-particle regions (4). The way the alloy is constituted will significantly influence the nature of the porosity and the microstructure of the finished part. Reduced levels of porosity have been shown to improve the dynamic properties of P/M materials (5).

High lubricant contents severely restrict the density to which a material can be compacted. The effect of lubricant content on the pore free density of compacted powder mixes is shown in Figure 2 where the relative influence of alloying additives and graphite is also illustrated (1). Lubricant content clearly has the most effect followed by that of graphite. Alloy additions such as copper or nickel have little or no effect. The pore free density is the maximum density to which the mixture can be compacted in the die cavity. However, the density of the resulting green compact will be lower than this value due to frictional losses during compaction and green expansion of the compact on ejection from the die cavity. Tool design and compaction sequencing also have a considerable influence on green part density and particular attention has to be paid to these items in any high performance part development program.

There are three key influences on part density during the sintering operation:

- Lubricant removal
- Alloying
- Dimensional change

Not only does the added lubricant affect density during the compaction phase, it also influences density during sintering when the lubricant is removed from the compact. This loss of material, without change in volume, reduces the density of the compact.

The way in which the alloy is constituted will determine the extent to which alloying during the sintering operation will alter the density and microstructure of the compact. The ability to optimize an alloy through the correct balance between prealloyed, partially alloyed, and admixed additions has been successfully applied to the production of steering column tilt levers (6,7).

Higher performance materials will result from reduced levels of porosity (2,5), and significant growth during sintering is counterproductive. Some shrinkage during sintering is therefore desired, with overall dimensional change values, with respect to die size, being close to zero.

A systems approach to the development of high performance P/M materials has been described in detail elsewhere (3). The purpose of the work summarized below is to determine the influence of alloying method on microstructural constituents, on pore size and pore shape, and to observe how these parameters relate to the mechanical properties of the materials studied.

## **EXPERIMENTAL PROCEDURE**

### **Test Specimen Preparation**

Admixed materials covered in the present study included nickel and copper steels (FN-0208 and FC-0208 based on Ancorsteel 1000B iron powder). Distaloy 4800A was the partially alloyed material and Ancorsteel 4600V the prealloyed material.

The mix compositions shown in Table I were blended with 0.5% lubricant. The lubricant was atomized ethylene bisstearamide wax (Lonza Acrawax C). In the case of the admixed materials, copper was added as Alcan 8081 and nickel as Inco 123. Asbury 3203 flake natural graphite was used in the amounts shown,

Most compacts were pressed at either 45 or 50 tsi. Selected materials were double pressed/double sintered in which case initial compaction was followed by sintering at 1550°F (presintering). The materials were then compacted for a second time prior to sintering at 2050°F or 2425°F. 'Dog-bone' tensile specimens were pressed in accordance with ASTM 158. Specimens for rotating bending fatigue (RBF) testing were

machined from 0.395" x 0.395" x 3" compacts to the dimensions shown in Figure 3.

Sintering was carried out in a pusher furnace. Samples were sintered at 2050°F for 3.0 minutes at temperature in a dissociated ammonia (D.A.) atmosphere unless other processing is indicated in the summary tables or text. The samples were placed on a bed of alumina powder in a ceramic tray, and covered with more alumina powder to reduce decarburization. Specimens sintered at 2425°F were held at temperature for 60 minutes in a D.A. plus methane atmosphere. After sintering, the trays were pushed directly into a water jacketed cooling zone.

Specimens for heat treatment were austenitized at 1550°F in a muffle furnace using a D.A. atmosphere, with methane added to maintain the correct carbon potential. The samples were quenched in circulating synthetic oil coolant, which was preheated to 150°F. Tempering was done in air. The admixed materials were tempered at 500°F for one hour while the prealloyed Ancorsteel 4600V based samples were tempered at 450°F.

### **Testing**

Tensile testing was carried out on an Instron tester at a crosshead speed of 0.02 in./min.

The RBF specimens were tested at 3300 RPM, with survival to 10 million cycles considered a 'runout'. Twenty specimens were used per test. Bending moments were chosen by the 'staircase' method (8) until appropriate stress levels were determined for a two-point estimate of the fatigue endurance limit.

### **Metallographic Examination**

Sections were cut from the shoulders of the fatigue test specimens and mounted for metallographic sample preparation. All metallographic preparation was done on a Struers Abrapoi automated grinder/polisher. Automated polishing is essential to provide the high quality scratch-free and stain-free surface required for quantitative metallography.

Pore size and shape measurements were carried out using a Leitz TAS plus automated image analyzer. Pores were sized according to their area using size intervals ranging from 0-100  $\mu\text{m}^2$  to 2400-2500  $\mu\text{m}^2$ . (A circular pore with a diameter of 50  $\mu\text{m}$  will have an area of 1963  $\mu\text{m}^2$ . To have an area of 100  $\mu\text{m}^2$  a circular pore would have a diameter of approximately 11  $\mu\text{m}$ ). Pore shape was assessed using the parameter  $4\pi A/P^2$ , where A is the area and P the perimeter. For a perfectly circular pore  $4\pi A/P^2 = 1$ .

A 10 x 10 meander was used to cover a total of 100 fields representing a total area of 4.7  $\text{mm}^2$  per sample. In order to ensure that only whole pores were used in the analysis, measurements were made in an area at the upper left corner of the screen (the guard frame) representing approximately one third of the measuring frame. Detected pores touching the outside edges of the guard or measuring frame were removed from the analysis. Those touching the inside edges were reconstructed and included in the assessment. This procedure prevents the analysis being biased towards the smaller pores, which would have occurred had all pores touching the edges been removed. For pore shape assessment, pores smaller than 4  $\mu\text{m}$  in length (maximum Feret diameter) were eliminated from the analysis. This was done by skeletonization and end point removal and not by erosion which could segment the pores or size them according to their minimum dimension.

### **RESULTS**

Tensile and fatigue data are summarized in Table 2 and Figures 4 to 7. Photomicrographs of the various

materials and the results of pore size and shape analysis are presented in Figures 8 to 21.

## **DISCUSSION**

### **Admixed Materials (FN-0208/FC-0208)**

These nickel and copper steels are admixed materials and have heterogeneous microstructures - Figures 8 - 12.

For the nickel steel, (FN-0208), increasing the sintering temperature from 2050°F to 2425°F increased the yield strength from 36,200 psi to 53,000 psi and the endurance limit from 22,200 psi to 31,200 psi - Figure 5. This is a consequence of increased nickel diffusion and pore rounding compare Figure 8 with Figure 12 and see Figure 16. High temperature sintering significantly reduced the number of pores but created a few larger than found in the sample sintered at 2050°F. Quenching and tempering the material sintered at 2050°F increased the yield strength to 109,000 psi and the endurance limit to 40,000 psi. The microstructure of the heat treated material is still heterogeneous with non-etching nickel rich areas - Figure 10.

The copper steel (FC-0208), had a yield strength of 73,100 psi and an endurance limit of 34,000 psi when sintered at 2050°F - Figure 5. Sintering at 2425°F increased the yield strength to 87,600 psi and the endurance limit to 39,000 psi, There were fewer pores in the copper steel compared with the nickel steel - compare Figures 6 and 17. High temperature sintering reduced the number of pores further and produced a very high percentage of rounded pores - see Figure 17, Large pores remained for all processing conditions. In contrast to the improvement seen on quenching and tempering the nickel steel, this treatment raised the yield strength of the copper steel to 123,000 psi but significantly decreased the endurance limit to 20,300 psi - Figure 5. The microstructures of the sintered (2050°F) and the heat treated copper steel are shown in Figure 9 and Figure 1 respectively. They are more homogeneous than the comparable nickel steel.

### **Partially Alloyed Materials (Distalloy 4800A)**

This partially alloyed material has a heterogeneous microstructure consisting of pearlitic particle cores surrounded by nickel rich areas of martensite with retained austenite and some bainite - Figure 13. The fatigue properties of this material benefit from this strong/tough intra-particle core with tough, high strength nickel rich martensite in the inter-particle regions, which arrests fatigue crack growth.

Increasing the density of the material containing 0.4% graphite from 7.20 g/cm<sup>3</sup> to 7.42 g/cm<sup>3</sup> through double pressing increased the yield strength from 63,500 psi to 71,000 psi and the endurance limit from 37,200 psi to 44,400 psi - an increase of 19% = Figure 6. In contrast, increasing the sintering temperature from 2050°F to 2300°F raised the yield strength to 77,900 psi but reduced the endurance limit to 34,500 psi. Increasing the sintering temperature to 2425°F, improved the yield strength to 98,700 psi while reducing the endurance limit to 31,300 psi for the material with a 0.6% graphite addition. The increased sintering temperature caused more diffusion of nickel from the inter-particle regions and reduced the crack arresting tendency of the material. The improved pore shape from high temperature sintering is apparently not able to offset this effect. Sintering at 2300°F reduced the number of pores slightly. Sintering at 2425°F significantly reduced the number of pores, particularly those smaller than 100 Nm<sup>2</sup>, while causing some slight pore coarsening - Figure 18. There were pores 2450 μm<sup>2</sup> in area in both single and double pressed samples sintered at 2050°F - Figures 18 and 19. Increasing the density of the 0.6% graphite material from 7.20 g/cm<sup>3</sup> to 7.43 g/cm<sup>3</sup> raised both the yield strength and the endurance limit of the material sintered at 2425°F to 105,700 psi and 47,600 psi respectively - Figures 6 and 14. The combination of double pressing and sintering at 2425°F eliminated the larger pores - Figure 19.

## **Prealloyed Materials (Ancorsteel 4600V)**

The highest endurance limit of all the materials tested was obtained with the prealloyed Ancorsteel 4600V. At a density of  $6.96 \text{ g/cm}^3$  this material, with a 0.6% graphite addition, had a tensile strength of 115,780 psi and an endurance limit of 41,800 psi in the quenched and tempered condition -Figure 7. This material has a homogeneous microstructure of tempered martensite - Figure 5. Increasing the density to  $7.25 \text{ g/cm}^3$  by double pressing resulted in a yield strength of 165,310 psi and an endurance limit of 57,500 psi (an increase of nearly 38%). Double pressing increased the number of smaller pores and slightly increased the total number of pores - Figures 18 and 19. However, double pressing eliminated the larger pores ( $> 1500 \mu\text{m}^2$ ) -Figures 18 and 19. Increasing the sintering temperature of the single pressed material from 2050°F to 2425°F had a similar effect to raising the density through double pressing. However, the yield strength and endurance limit did not quite reach the level of the higher density material - Figure 7. Combining high temperature sintering with double pressing gave the highest yield strength and endurance limit observed = 158,700 psi and 66,100 psi respectively (Figure 7). High temperature sintering significantly reduced the number of smaller ( $< 100 \mu\text{m}^2$ ) pores and resulted in a considerable amount of pore rounding = Figures 20 and 21.

## **CONCLUSIONS**

The results clearly indicate that the alloying method used to constitute ferrous P/M materials has a significant influence on the sintering response of the material and on the number, size, and shape of the pores the material contains. The data indicates that the fatigue strength of P/M steels is directly related to the amount of porosity they contain and their pore morphology. The single most effective method of improving fatigue properties is to increase the density. Sintering a prealloyed material (Ancorsteel 4600V) at high temperature followed by quenching and tempering increased both the tensile and fatigue properties. However, high temperature sintering of a partially alloyed material (Distaloy 4800A), increased the tensile yield strength but actually reduced the fatigue endurance limit due to a reduction in the proportion of nickel rich austenite and an increase in the amount of martensite. Increasing the density of the high temperature sintered, partially alloyed material, however, significantly increased the fatigue strength. These findings support Saito's hypothesis that in order to achieve the best combination of strength and toughness in P/M steels it is necessary to optimize the properties of both inter-particle and intra-particle regions (2).

## **ACKNOWLEDGEMENTS**

The tensile and RBF testing was supervised by R.C.O'Brien and was carried out by W.R. Bentcliff and R.J. Fitzpatrick. The 2425°F sintering was carried out by Remington Powder Metal Products. The author is grateful to G. Colin for preparing the metallographic samples and taking the photomicrographs. He is also grateful to T.F. Murphy for carrying out the image analysis and for discussions on pore size and shape characterization.

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Method of Alloy Addition	Name	Ni	Mo	Cu	Mn	Carbon Added as Graphite (Wt. %)	Typical Sintered Carbon (Wt. %)
Admixed	FN-0208	2.00			0.10	0.75	0.67
Admixed	FC-0208			2.00	0.10	0.90	0.81
Partially Alloyed	Distaloy 4800A	4.05	0.55	1.60	0.10	0.40	0.35
		4.05	0.55	1.60	0.10	0.60	0.55
Prealloyed	Ancorsteel 4600V	1.80	0.50		0.25	0.60	0.54

Table 1 : Material Compositions

MATERIAL	SINTERED DENSITY (g/cm <sup>3</sup> )	APPARENT HARDNESS	YIELD STRENGTH (psi)	UTS (psi)	% ELONG (in/ inch)	FAT'GE LIMIT (psi)
FN-0208 (2050°F)	7.12	77 HRB	36,200	64,500	3	22,200
FN-0208 (2425°F)	7.23	82 HRB	53,000	83,600	5	31,200
FN-0208 (2050°F) Q&T	7.10	33 HRC	109,000	126,000	< 1	40,000
FC-0208 (2050°F)	7.01		73,100	94,100	2	34,000
FC-0208 (2425°F)	7.11	92 HRB	87,600	112,700	3	39,000
FC-0208 (2050°F) Q&T	7.01	35 HRC	123,000	132,000	< 1	20,300
Distaloy 4800A						
0.4% gr.(2050°F)	7.20	92 HRB	63,500	103,200	4	37,200
0.4% gr.(2300°F)	7.23	98 HRB	77,900	119,100	4	34,500
0.4% gr.(2050°F) DP/DS	7.42	23 HRC	71,000	134,100	4	44,400
0.6% gr.(2425°F)	7.20	28 HRC	98,700	139,700	2	31,300
0.6% gr.(2425°F) DP/DS	7.43	32 HRC	105,700	171,200	2	47,600
A4600V + 0.6% gr.						
(2050°F)	6.96	26 HRC	No Yield	115,780	< 1	41,800
(2050°F) DP/DS	7.25	35 HRC	165,310	173,250	< 1	57,500
(2425°F)	6.98	32 HRC	149,900	166,510	< 1	56,800
(2425°F) DP/DS	7.20	38 HRC	158,700	190,500	1	66,100

Table 2 : Summary of Tensile and Fatigue Data





Effect of Second Phase Additions on the Pore Free Density of Ancorsteel 1000B

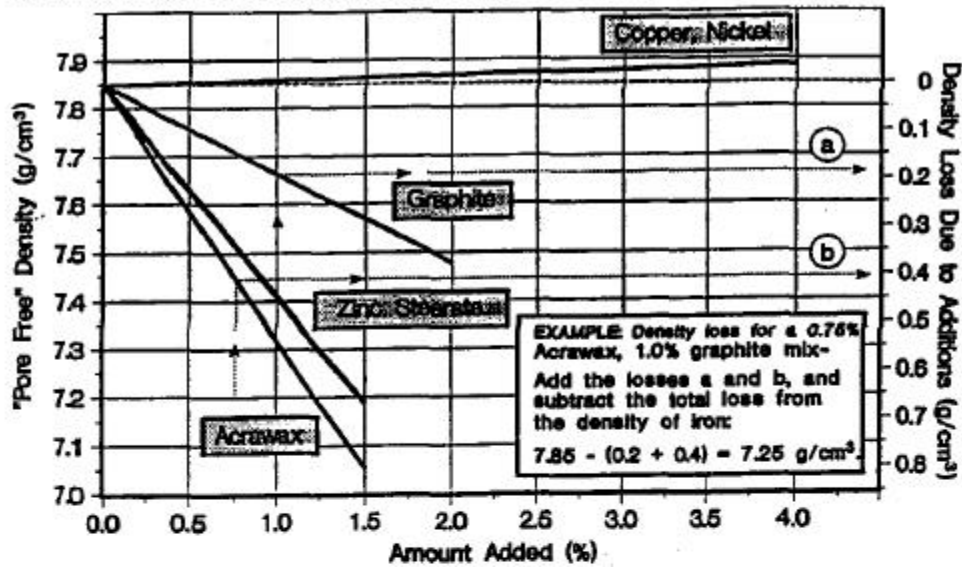


Figure 2 : Effect of premix additives on the pore free density of Ancorsteel 1000B.

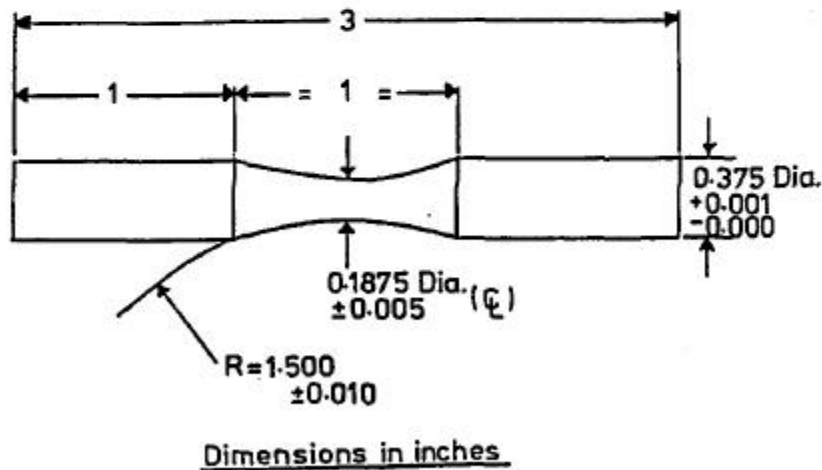


Figure 3 : Rotating bending fatigue (RBF) specimen.

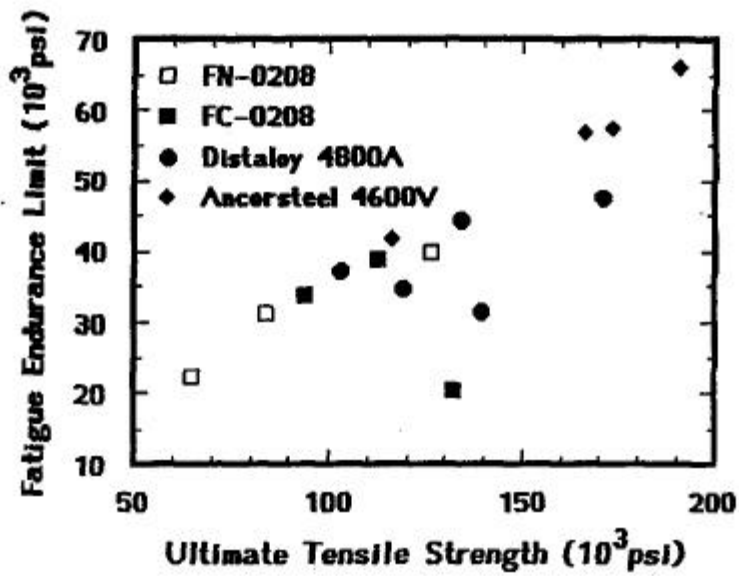


Figure 4 : Summary of tensile and fatigue data.

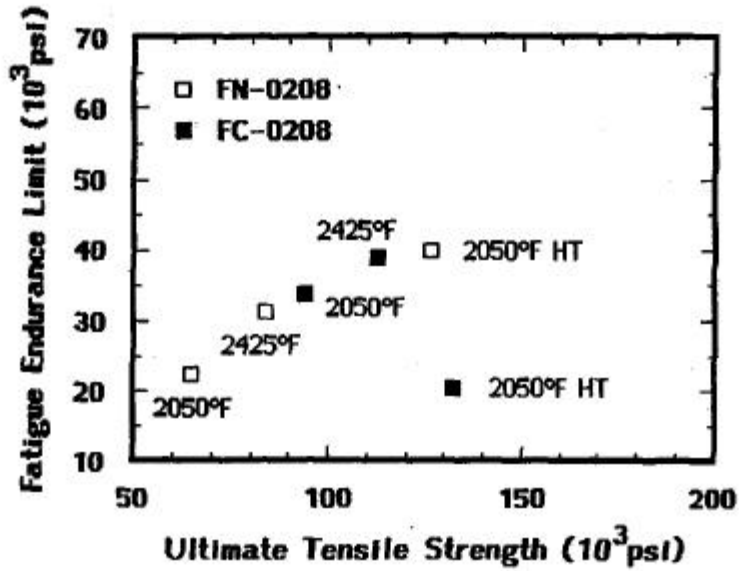


Figure 5 : Fatigue data - nickel and copper steels.

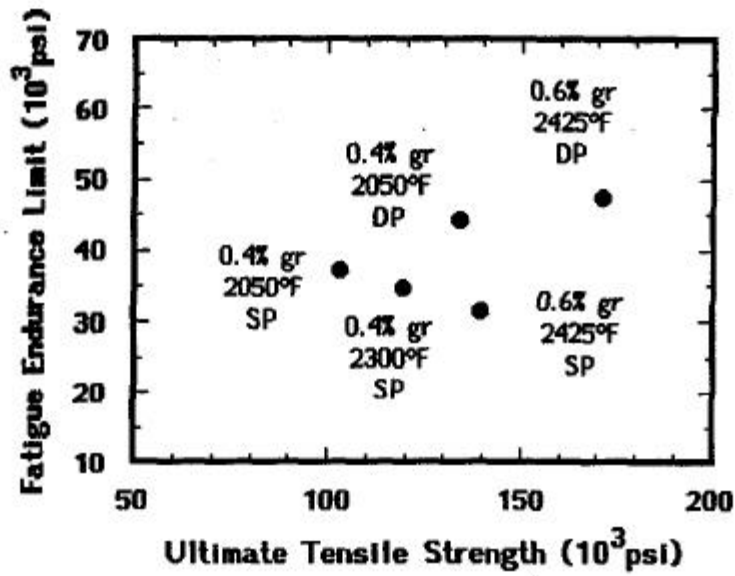


Figure 6 : Fatigue data - partially alloyed materials (Distaloy 4800A).  
(SP - single pressed ; DP - double pressed)

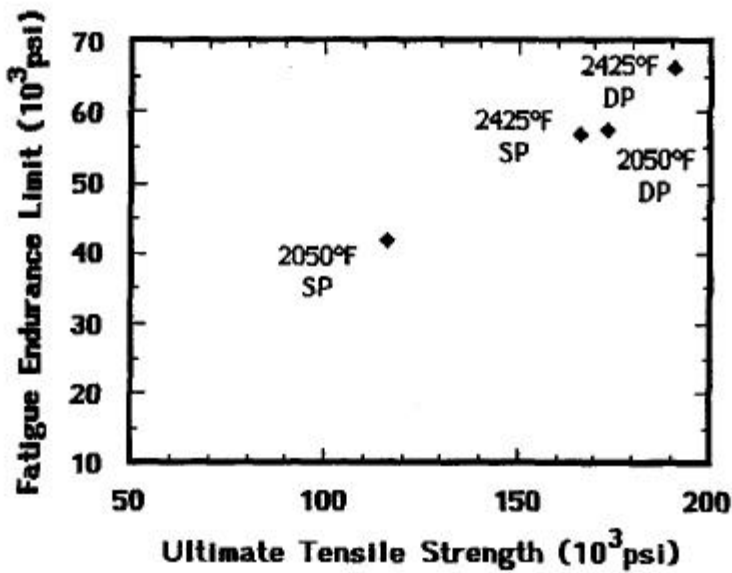


Figure 7 : Fatigue data - prealloyed materials (Ancorsteel 4600V).  
(SP - single pressed ; DP - double pressed)

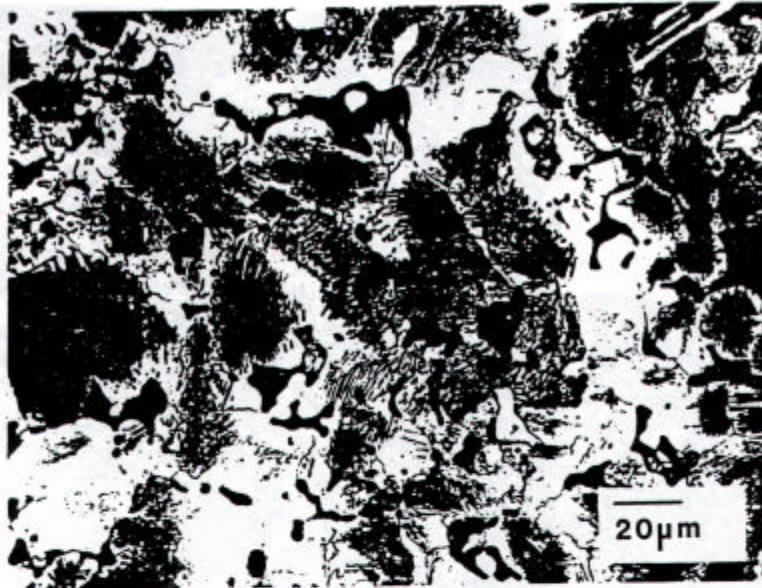


Figure 8 : Nickel steel (FN-0208) sintered at 2050°F. Etched in 2% nital/4% picral.

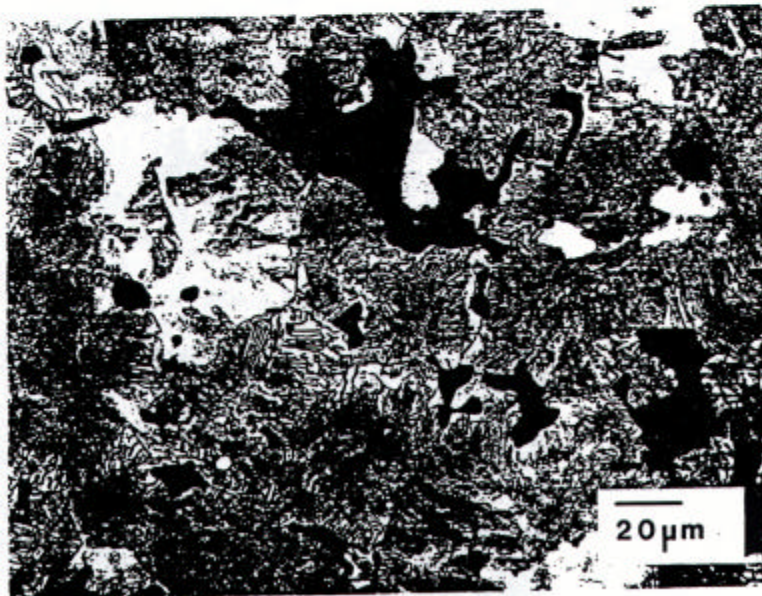


Figure 9 : Copper steel (FC-0208) sintered at 2050°F. Etched in 2% nital/4% picral.

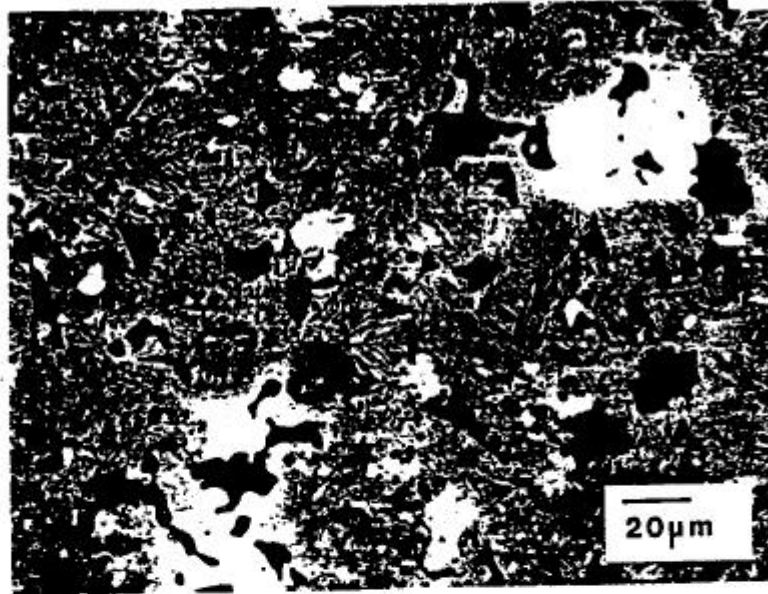


Figure 10 : Nickel steel (FN-0208) sintered at 2050°F. Quenched and tempered. Etched in 2% nital/4% picral.

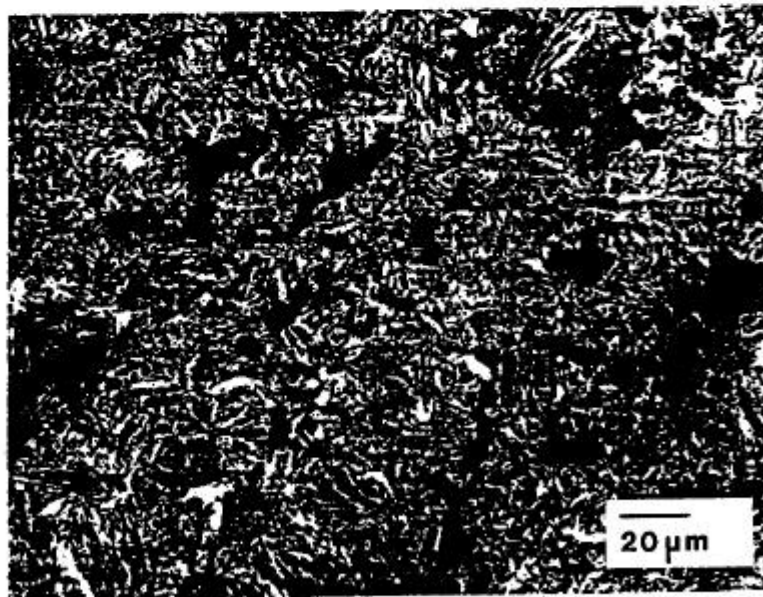


Figure 11 : Copper steel (FC-0208) sintered at 2050°F. Quenched and tempered. Etched in 2% nital/4% picral.



Figure 12 : Nickel steel (FN-0208) sintered at 2425°F. Etched in 2% nital/4% picral.



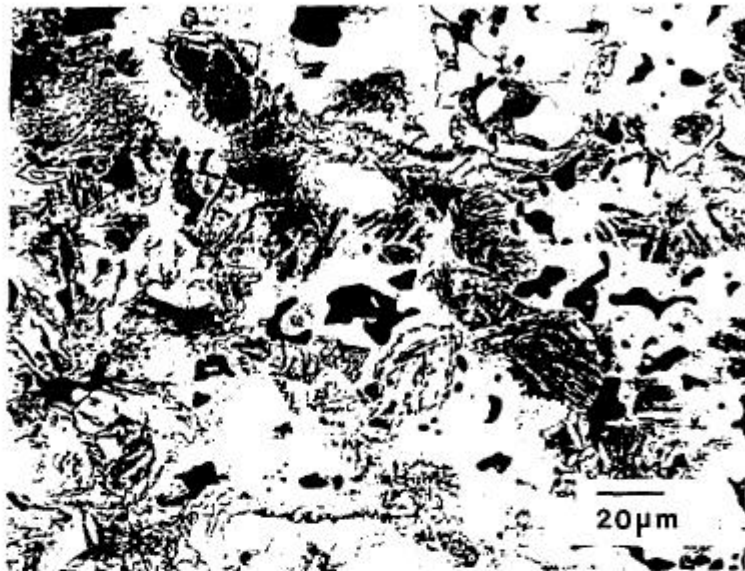


Figure 13 : Partially alloyed material (Distaloy 4800A) + 0.4% graphite, single pressed, sintered at 2050°F. Etched in 2% nital/4% picral.

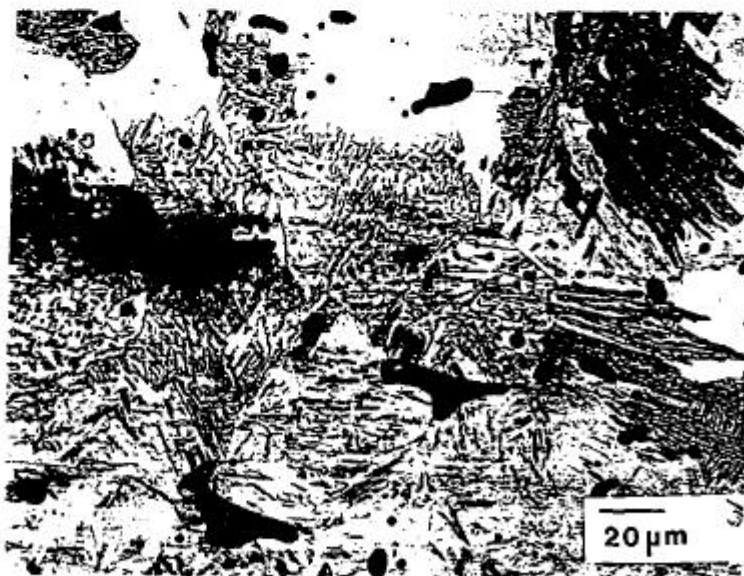


Figure 14 : Partially alloyed material (Distaloy 4800A) + 0.6% graphite, double pressed, sintered at 2425°F. Etched in 2% nital/4% picral.



Figure 13 : Partially alloyed material (Distaloy 4800A) + 0.4% graphite, single pressed, sintered at 2050°F. Etched in 2% nital/4% picral.

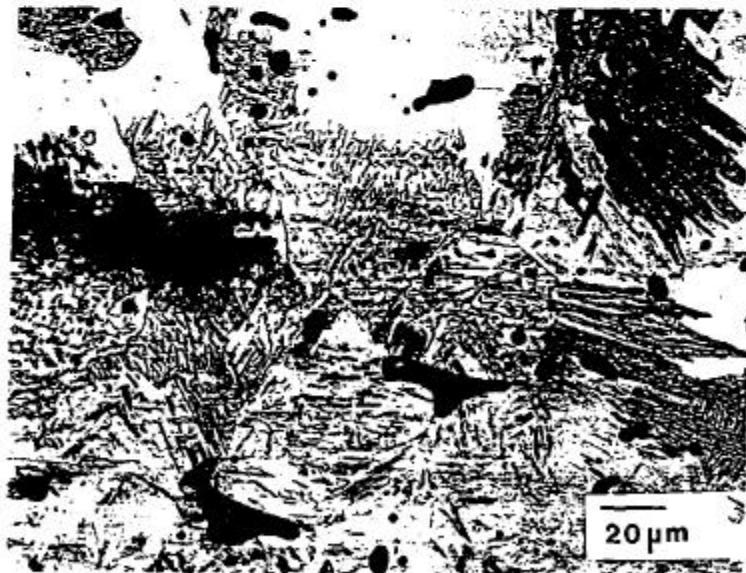


Figure 14 : Partially alloyed material (Distaloy 4800A) + 0.6% graphite, double pressed, sintered at 2425°F. Etched in 2% nital/4% picral.



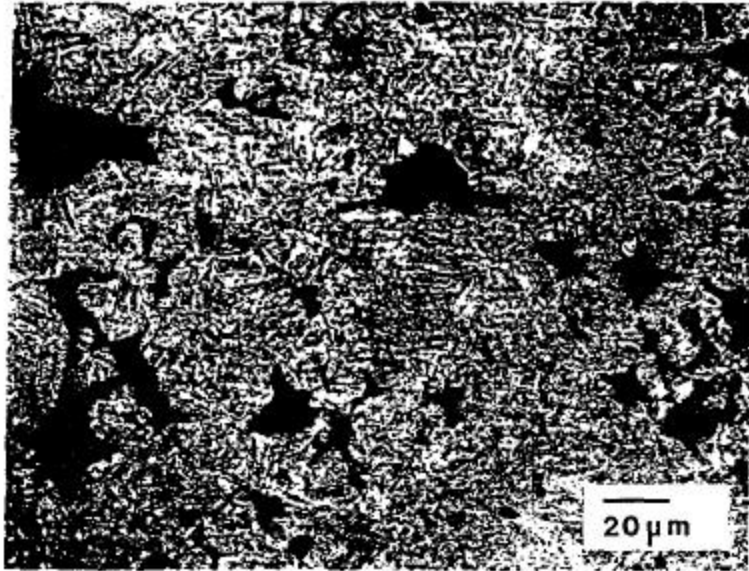


Figure 15 : Prealloyed material (Ancorsteel 4600V), sintered at 2050°F, quenched and tempered. Etched in 2% nital/4% picral.

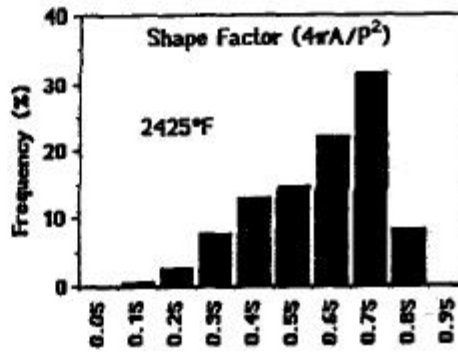
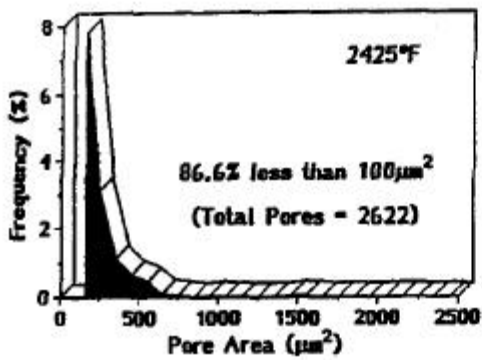
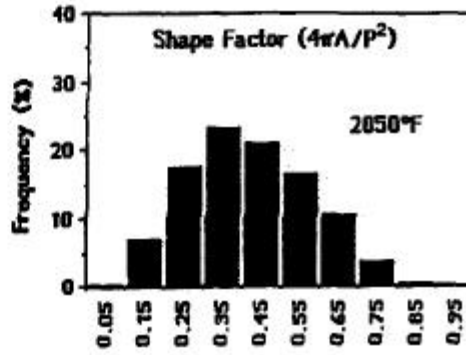
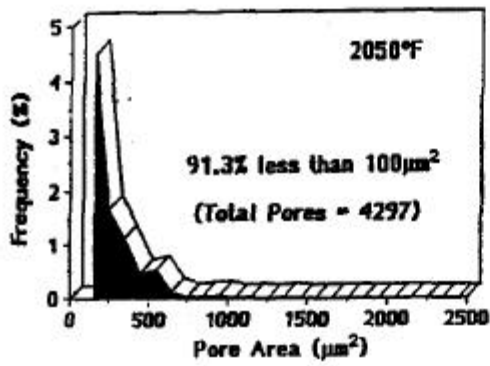


Figure 16 : Pore size and pore shape distribution nickel steel (FN-0208). Samples compacted at 45 tsi.

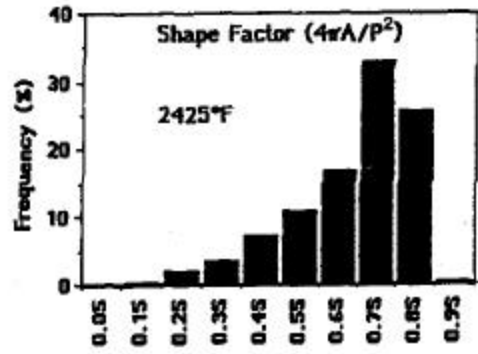
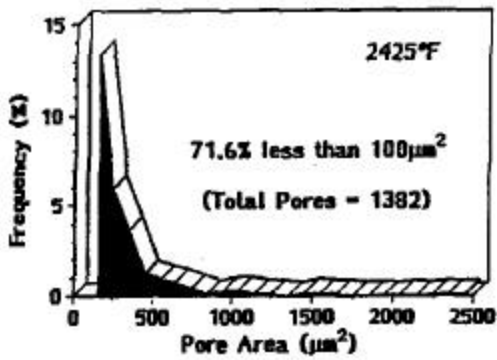
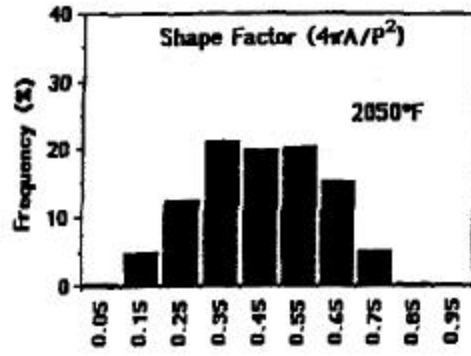
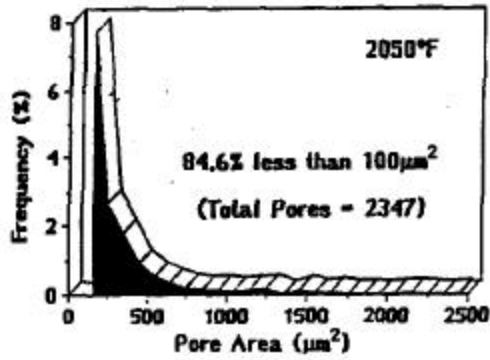


Figure 17 : Pore size and pore shape distribution copper steel (FC-0208). Samples compacted at 45 tsi.

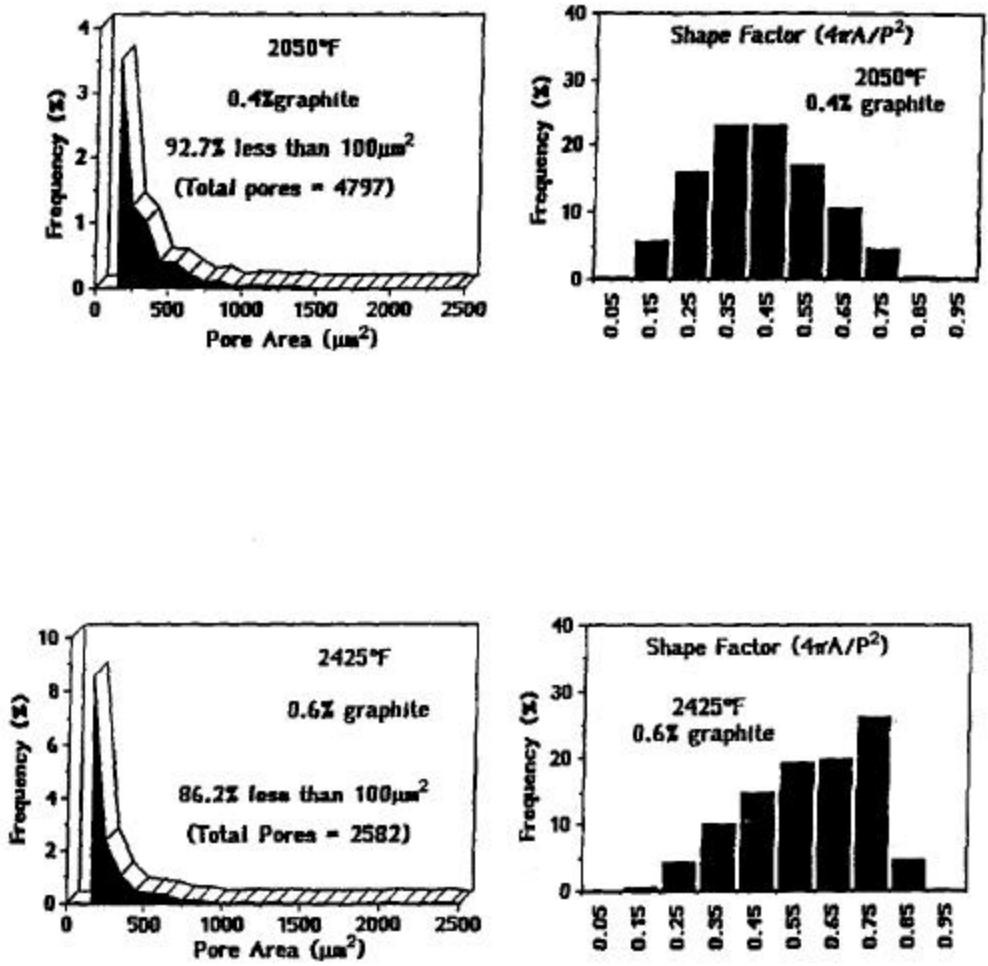


Figure 18: Pore size and pore shape distribution Distaloy 4800A - single pressed. Samples with 0.4% graphite were compacted at 50 tsi while those with 0.6% graphite were compacted at 45 tsi.

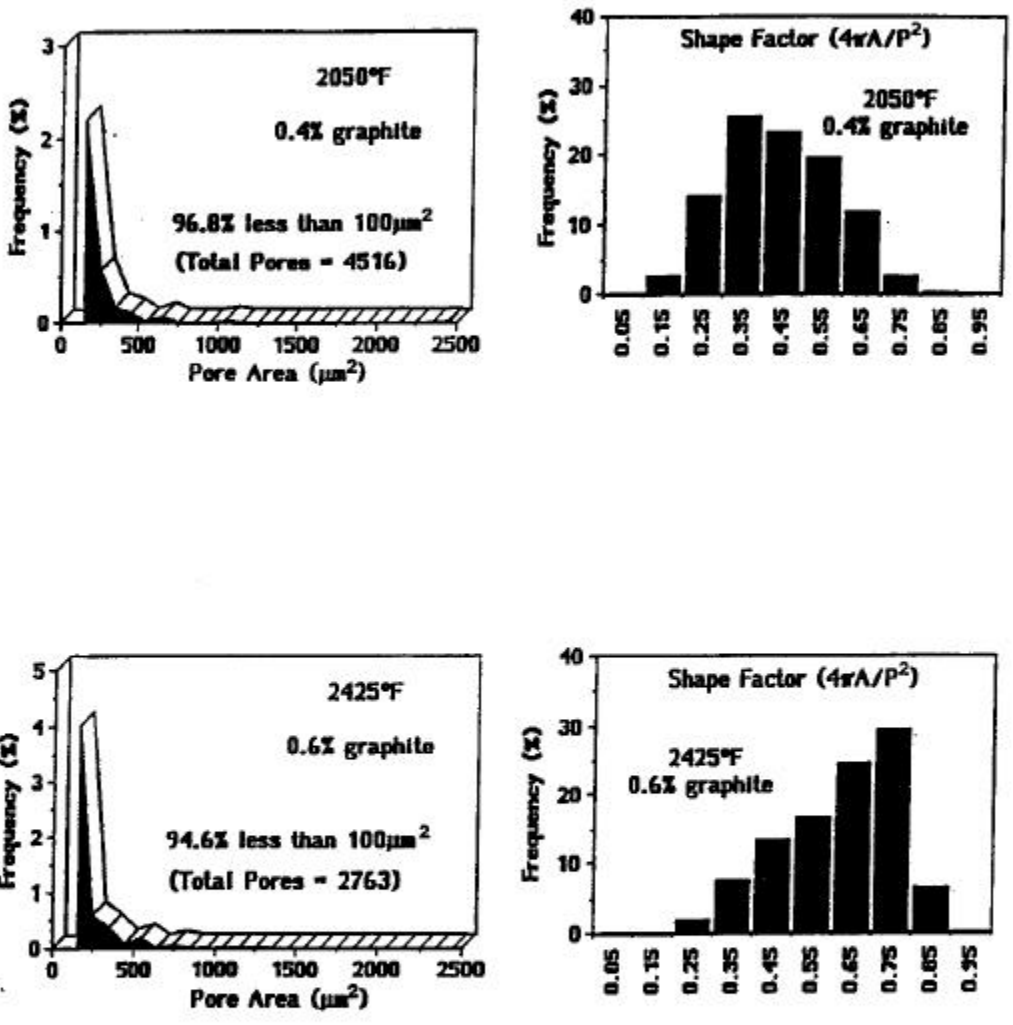


Figure 19 : Pore size and pore shape distribution Distaloy 4800A - double pressed. Samples were compacted at 50 tsi, presintered at 1550°F followed by compaction at 50 tsi.

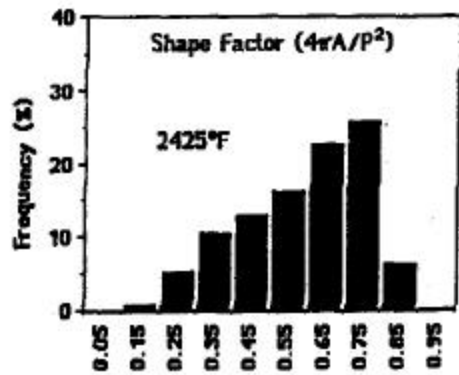
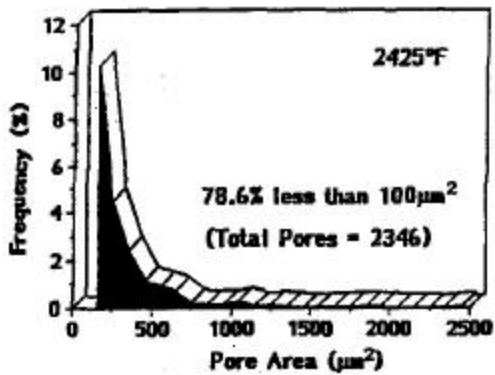
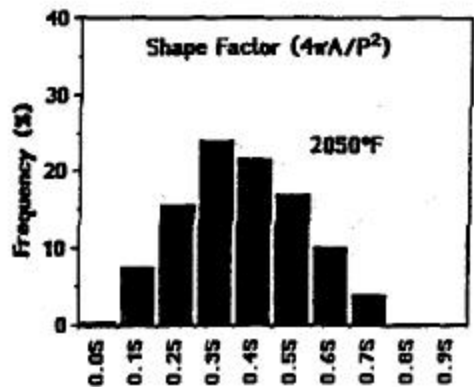
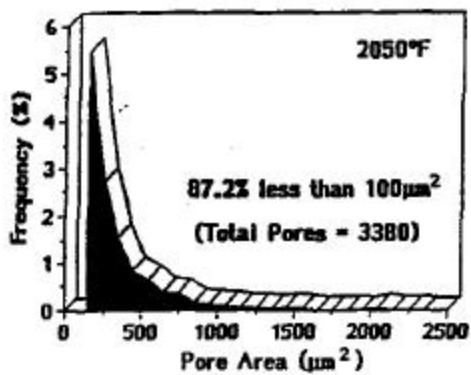


Figure 20 : Pore size and pore shape distribution Ancorsteel 4600V - single pressed. Samples compacted at 45 tsi.

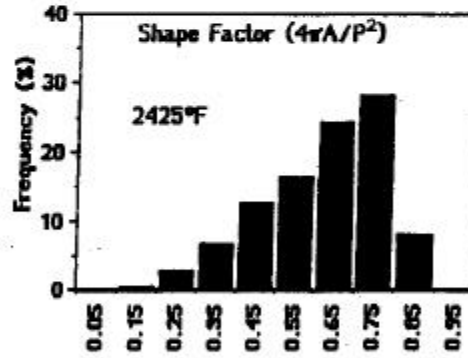
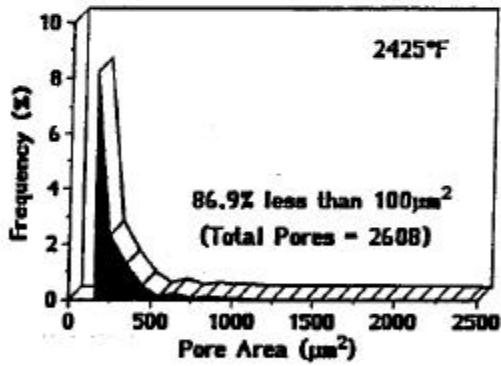
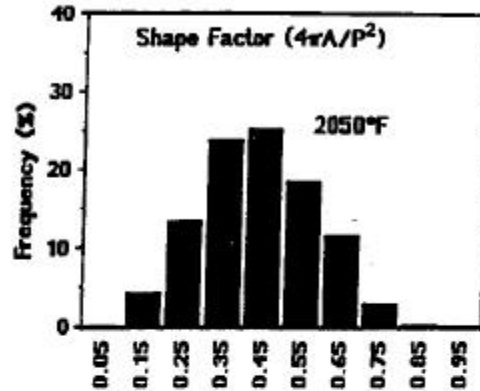
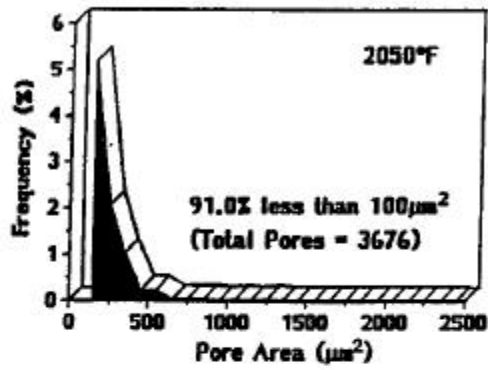


Figure 21 : Pore size and pore shape distribution Ancorsteel 4600V - double pressed. Samples were compacted at 45 tsi, presintered at 1550°F followed by compaction at 45 tsi.