HIGH PERFORMANCE FERROUS P/M MATERIALS:

THE EFFECT OF ALLOYING METHOD ON DYNAMIC PROPERTIES

W. Brian James and Robert C. OtBrien

Hoeganaes Corporation
Riverton, New Jersey 08077

Abstract

A comparison has been made between fully prealloyed, partially prealloyed, and elementally admixed alloys in the "as sintered" condition in order to assess the influence of microstructural and chemical homogeneity on the tensile, impact, and fatigue properties.

Elementally admixed and completely prealloyed powders were prepared with chemistry similar to that of the diffusion bonded Distaloy 4600A (nominally 1.8 wt. % Ni, 1.6 wt. % Cu, and 0.55 wt. % Ho). An addition of 0.6% graphite was made to each of these materials.

In one series of experiments, test pieces were prepared from each of the materi`1als pressed to a green density of 6.9 g/cm^3. Sintering was carried out at 2050°F for 30 minutes at temperature in a dissociated ammonia atmosphere. A second series of experiments was carried out in which a fixed compaction pressure of 45 tsi was applied to each of the materials. Sintering was carried out in a similar manner to the first series. An additional prealloyed material, Sumiron 4100S, was included in this second series of experiments. However, this material was sintered at 2300°F to reduce the tendency for oxidation of this chromium and manganese steel alloy.

For the samples pressed to a fixed green density of 6.9 g/cm^3, the tensile strength of the partially prealloyed material was significantly higher than the other materials. The impact energy of the partially prealloyed material was also higher.

A similar trend was found for the samples pressed at a fixed compaction pressure of 45 tsi. The partially prealloyed product, sintered at 2050OF, was even superior to the Sumiron 4100S; sintered at 2300°F. The differences in the tensile and impact properties were significant at the one percent level. The rotating bending fatigue performance of the partially prealloyed material was superior to that of the Sumiron 4100S.

Introduction

The experimental work reported in this paper assesses the influence of microstructural and chemical homogeneity on the tensile, impact, and fatigue properties of alloy steel in the "as sintered" condition. The study is part of a project aimed at gaining a better understanding of the factors which control the toughness and fatigue response of P/H materials in order to develop P/B alloys which can compete with wrought products.

The influence of chemical and microstructural homogeneity on the mechanical properties of sintered ferrous materials has been studied by a number of authors (I - 6). Some of the *investigations* studied the influence of increased homogeneity achieved via increased sintering time and temperature (3, 4, 5) while others compared alloys of similar chemistry produced by different methods (I, 2, 6). For a material of specific *chemistry*, produced by a given alloying method, there is some agreement that higher sintering temperatures and increased density result in better dynamic properties. Opinions differ, however, as to whether a homogeneous material per se is best.

Excellent reviews of the various alloying practices for *the* production of sintered steels have been written by Lindskog and skoglund (7), and by *Arbstedt* (8). The alloying methods, which are used, for the production of ferrous P/M parts can be divided into the following groups:

- admixtures of elemental or master alloy powders to a plain iron powder
- Diffusion bonded alloys - sometimes referred to as partially prealloyed powders.
- completely prealloyed powders

The above materials are illustrated schematically in Figure 1.

Elementally Admixed Materials

This first group covers most of the traditional P/M materials such as iron with carbon, copper, nickel, and molybdenum where the alloying elements are blended with the iron base. This is undoubtedly the easiest way of preparing an alloyed material. However, producing a uniform mixture involves a number of problems that make it necessary to select equipment, mixing procedure, raw materials, and control methods with the utmost care. Many powder users prefer to have their suppliers prepare press-ready mixes containing all the required ingredients because the powder supplier usually has more efficient equipment and greater experience in the handling of powders. The function of the alloying ingredients determines the particle size and shape to be used. Unfortunately, these seldom coincide with the optimum powder characteristics for mixing. Although this can make it difficult to achieve a uniform mix and prevent it from segregating, such mixes can be successfully produced and shipped over large distances without *segregation* by adopting a proper mixing procedure and packaging method.

Partially Prealloyed Materials

Partially prealloyed or diffusion bonded powders have been used for quite some time for the production of high strength sintered components. The production of these powders involves the heat treatment of a mixture of iron powder and alloying elements in a reducing atmosphere. During this treatment, the alloying elements partially diffuse into the iron powder and a metallurgical bonding between the iron powder and the alloying element particles is achieved. There are a number of advantages associated with this approach to alloying. Because the alloying elements are only partially prealloyed, the high compressibility of the iron base is maintained. Very fine powders of the alloying elements can be added to the iron powder without the risk of segregation during transport and handling of the powder mix. Segregation may lead to compositional variations from part to part and may contribute to variation in dimensional change during sintering. Reduced segregation makes it easier for the parts producer to consistently meet close dimensional tolerance requirements. Diffusion bonding the fine particle size alloying elements to the iron base reduces their tendency to form lumps or agglomerates during subsequent blending with graphite and lubricant. The alloying additives will thus be more evenly distributed and can contribute more efficiently to improvements in the mechanical properties of the sintered parts than would be possible in regular mixes.

Completely Prealloyed Materials

Completely prealloyed powders consist of particles within which all the alloying elements are homogeneously distributed. Such powders are generally produced by water atomization of liquid alloy steel. The powders are characterized by dense particles with regular shape; i.e., similar shape characteristics to water atomized iron powder. With prealloyed powders, no further homogenization during sintering is necessary. However, prealloyed powders are less compressible than pure iron powder because of the solid solution strengthening effect of the alloying elements. The increase in particle hardness is roughly proportional to the concentration of alloying element and the magnitude of the effect varies considerably from one element to another. The transition elements have *little* influence, whereas elements with atomic radii deviating greatly from that of iron have a pronounced effect. Elements, which go into interstitial solid solution in the iron, reduce the compressibility of the powder to the greatest extent. For this reason, carbon is usually added separately as graphite.

Experimental procedure and Results

The experimental program was carried out in two phases. In one series of experiments, test pieces were prepared from each of three materials pressed to a green density of 6.9 g/cm· For a second series of experiments; a fixed compaction pressure of 45 tsi was applied to each of the materials. An additional prealloyed powder, Sumiron &I00S, was included in this series of experiments. Details of the various materials and their processing are given below.

Materials

Ancorsteel IO00B was selected as the high compressibility water atomized iron base for the elementally admixed alloy. Nickel (Inco 123), copper (Alcan 8081-3), and molybdenum (AMAX -325 mesh) powders were added to match the chemistry of the diffusion bonded alloy.

Distaloy 4600A was chosen for the diffusion bonded partially prealloyed material. In this material, the alloying additives are diffusion bonded to an Ancorsteel 1000B iron base by annealing in a reducing atmosphere.

The third material, a prealloyed water atomized powder of similar chemistry to the Distaloy 4600A, was produced in an R&D pilot plant facility.

An additional prealloyed powder, Sumiron 4100S, was included in the second phase of the program. This powder is o11 atomized to reduce oxidation of its primary alloying elements, chromium and manganese.

Chemical analyses of the materials are given in Table I.

Compaction and Sintering

Each material was blended with 0.6 wt. % Asbury 3203 graphite and 0.5 wt. I Acravax C (atomized grade) prior to compaction of impact and tensile test pieces In the first series of experiments, samples were compacted to a fixed green density of 6.9 g/tm3, whereas in the second series a fixed pressure of 45 tsi was applied. Compressibility curves for the materials are given in Figure 2.

The compressibility's of the elementally admixed and partially prealloyed materials were very similar and were significantly higher than the compressibility of the water atomized prealloyed powder. Consequently, a much higher compaction pressure was required to achieve a green density of 6.9 g/cm^3 in the prealloyed material. In the second series of experiments, where a fixed compaction pressure of 45 tsi was used, both prealloyed materials pressed to lower densities than the elementally admixed or partially prealloyed powders.

Unnotched Charpy impact test pieces were produced to the dimensions specified in ASTH E23-82. "Dog-bone" tensile specimens were prepared according to ASTH E8-85. In the second series of experiments, rotating bending fatigue (RBF) specimens were machined from sintered, rectangular compacts of partially pre-a11o~ed and Sumiron 4100S materials. The RBF specimens were machined to the dimensions shown in Figure 3.

Sintering of all materials was carried out in a pusher type furnace in a dissociated ammonia atmosphere. The specimens were at the sintering temperature of 2050°F for 30 minutes except for the Sumiron 4100S samples, which were sintered at a temperature of 2300°F.

For the samples pressed to a fixed green density, the elementally admixed materla1 exhibited the greatest amount of dimensional change after sintering. The dimensional change of the partially prealloyed material (+0.11I) was forty percent less than that for the elementally admixed material (+0.39). The pre-alloyed material actually showed a 0.16I shrinkage compared with die size.

In the second series of experiments (45 tsi compaction pressure), the partially prealloyed material showed only half the growth of the elementally admixed samples (+0.241 compared with +0.481). Because of its lower compressibility, the prealloyed material had the lowest sintered density (6.90 g/cm3), even though it shrank 0.18I during sintering. The sintered densities of the elemental (7.06 g/tm3), the partially prealloyed Distaloy &600A (7.1l g/tm3), and the prealloyed Sumiron 4100S samples (7.07 g/tm3) were similar.

Mechanical Property Testing

Impact Testing

Room temperature, unnotched Charpy impact testing was carried out in accordance with ASTM E23-82 and the results are summarized in Table 2, Figure 4, and Figure 5. The results are the averages for five test specimens per material. Estimates of standard deviation are included. The specimens were oriented for testing so that the impacted face was perpendicular to the direction of pressing. The partially prealloyed material had the best impact energy values in both sets of experiments. For the samples pressed to a fixed green density of 6.9 g/cm^3, the difference in the impact energy values for the partially pre-alloyed and elementally admixed materials (10.5 ft. lb. compared with 8.5 ft. lb.) is significant at the 21 level. This means we can be 981 confidant that the difference is valid. For the samples pressed at 45 tsi, the difference between the impact energy of the partially prealloyed material (15.8 ft. lb.) and the other three materials is significant at the II level. We can therefore be 991 confident that the difference in impact energy between the partially pre-alloyed, Distaloy 46OOA sample sintered at 2050'F, and the sumiron 4100S sample sintered at 2300OF is valid.

Tensile Testing

Tensile testing was carried out in accordance with ASTM E8-85 on an Instron *test* machine using a crosshead displacement rate of 0.02 inches per minute. An extensometer was used to measure tensile elongation relative to the 1-inch gauge length. The results, summarized in Table 2, Figure 6, and Figure 7, are the averages for five *test* specimens per material.

In both sets of experiments, the partially prealloyed material had the highest ultimate tensile strength and the highest tensile elongation (85.5 and 98.9 tsi and 2.6 and 3.5I respectively). In each phase of the program, the differences in tensile strength are significant at the IZ level. The prealloyed materials had the highest yield strengths. The highest yield strength observed, 71.0 tsi, was that of the Sumiron 4100S compacted at 45 tsi and sintered at 2300°F.

Fatigue Testing

In the second series of experiments, a comparison was made between the rotating bending fatigue performance of the partially prealloyed Distaloy 4600A and the Sumiron 4100S material. Rotating bending fatigue (RBF) testing at 2,800 - 3,050 rpm was carried out using the two-point strategy procedure (9). The two-point strategy involves testing at regular stress intervals until a stress level that has produced all runouts produces a failure, and a higher stress level that has produced all failures produces a runout. After this point is reached, all further testing is concentrated at these two levels. At least six replicates should then be generated at these two levels.

Development of median fatigue strength *estimates* for the two-point method involves computing the percentage of failures at each of the two stress levels. These percentages are assumed to plot as a straight line on probability paper. In the present work, the data have been plotted on Weihull probability paper.

Application of the two-point strategy depends on a reasonable initial estimate of the median fatigue strength and the proper selection of the stress interval used. Preliminary testing can be valuable in this regard. If sufficient specimens are available, exploratory testing can be started at a stress level significantly above the estimated fatigue strength. Testing can then work down in stress level so that several points on the S/N curve can he identified in the process as shown in Figure 8.

The most advantageous spacing of the stress levels is in the range of 2/3 to 3/2 of the standard deviation of the underlying distribution (10 - 12). This spacing corresponds to approximately to 3% of the ultimate strength of most wrought materials.

The fatigue data is summarized in Figure 9 in which stress is plotted against the percentage probability of failure. The fatigue performance of the partially prealloyed material is better than that of the prealloyed Sumiron 41005 even though the latter was sintered at 2300°F. The stress corresponding to 5% probability of failure (95I probability of survival) is 28 tsi for the sumiron 4l00S and 32.5 tsi for the partially prealloyed material.

Discussion

The higher yield strengths of the prealloyed materials are probably due to their more homogeneous chemistries and microstructures. Photomicrographs of the various sintered microstructures, for samples compacted at 45 tsi, are presented in Figures 10 and 11.

The lower sintered density of the prealloyed material of Distaloy ~600A chemistry is apparent in the unetched photomicrograph of Figure 10c. Despite a good degree of sinter, there is still some evidence of prior particles in this sample. While the other three materials have similar sintered densities, their pore morphologies and pore distributions are different. The background porosity levels are similar for the partially prealloyed and elementally admixed materials but the latter material contains larger individual pores (Figure 10a and b). The porosity in the Sumiron 4100S sample (Figure l0d) is on a similar scale to that found in the partially prealloyed material but is less continuous.

The more homogeneous microstructure of the prealloyed materials is evident from Figure 11c and d. The microstructures consist predominantly of divorced peerlite with some free ferrite. The microstructures of the elementally admixed and partially prealloyed materials are quite complex and contain a number of different phases (Figure 11a and b). Pro-eutectoid ferrite, colonies of very fine pearlite, areas of divorced pearlite, martensite, and light etching nickel rich regions are found in the partially prealloyed material (Figure lib). A similar microstructure, differing only in the proportions of the various constituents, is present in the elementally admixed alloy (Figure 11a).

The present data show that monotonic tensile properties are not necessarily the best predictor of a materials performance under conditions of cyclic loading. For example, in the second series of experiments, the Sumiron &100S material has a higher monotonic, 0.2Z offset yield strength than the partially prealloyed materla1. This higher yield strength does not, however, correlate with a better fatigue strength - the partially prealloyed material has the best fatigue properties. A partial explanation for this behavior may be that these materials respond differently under conditions of cyclic loading. The cyclic yield strength of the partially prealloyed materla1 is higher than its monotonic yield strength whereas for the prealloyed Sumiron 4IOOS, the reverse is true (13). Cyclic hardening and cyclic softening are illustrated schematically in Figure 12.

The good dynamic properties of the partially prealloyed material may be the result of its heterogeneous microstructure with high interparticle bond strength, tough nickel rich areas, and fine pearlite colonies.

The relationship between properties and microstructure is apparently quite complex. Further work is required to quantify the pore morphology and distribution in the various materials, to better understand the interaction between pore morphology end microstructure and how this influences properties under conditions of cyclic loading. The results indicate that cyclic, as well as monotonic, tensile data need to be determined in order to better characterize a materials performance.

Conclusions

The partially prealloyed material has the best impact, tensile, and fatigue performance of the materials examined.

For samples compacted to a fixed green density of 6.9 g/cm^{3}, the impact properties of the partially prealloyed materla1 (10.5 ft. lbs.), were significantly different from those of the elemental and prealloyed materials. In this series of experiments, the tensile strength of the partially prealloyed samples (85.5 tsi) was higher than that of the prealloyed or elemental materials. The prealloyed material had the highest 0.2% offset yield strength (64.4 tsi).

When the materials were compared at a fixed compaction pressure of 45 tsi, the partially prealloyed material again had the best impact (15.8 ft. lbs.) and tensile (98.9 tsi) properties.

The results indicate that monotonic tensile data is not necessarily a good predictor of properties under conditions of cyclic loading. Porous materials with heterogeneous microstructures which cyclically harden may be better from the viewpoint of fatigue strength than materials produced from homogeneous pre-alloyed powders. The rotating bending fatigue performance of the partially pre-alloyed material of Distaloy 4600A chemistry was in fact better than that of the prealloyed Sumiron 4100S.

References

"Methods of Extending the Applicability of Sintered Steels," G. Wastenson, Powder Metallurgy, 18, (35) 1975, 124.

"Prealloyed, Premixed, and Hybrid Sintered Steels," D. J. Burr, The International Journal Of Powder Metallurgy and Powder Technology, 1-33, (2), 1977, 8.

"Influence of Homogeneity on the Fatigue Properties of Sintered Steels," C. M. Sonsino, G. Schlieper, and W. J. Huppmann, The International Journal of Powder Metallurgy and Powder Technology, 20, (]) 1984, 45.

"Sintering and Heat Treatment of Steels made from a Partially Prealloyed Iron Powder," M. Khaleghi and R. Haynes, Powder Metallurgy, 28, (4) 1985, 217.

"Homogenization in Sintering and its influence on Mechanical Properties of Fe-Cr-C and Fe-Ni-C Steels," P. A. Vityaz and A. Kh. Nasybulin, Powder Metallurgy, 28, (3), 1985, 166.

"Fatigue Properties of Sintered Steels," I. gertilsson, B. Karlsson, and J. Wasen, Modern Developments in Powder Metallurgy, Volume 16, HPIF, 1984, p. 19.

"Alloying Practice in the Production of sintered Steels," P. Lindskog and G. Skoglund, Powder Metallurgy, Third European Powder Metallurgy Symposium 1971, Conference Supplement Part 1, p. 375.

"Alloy Systems Developed for Pressing and Sintering in the Ferrous Field," P. C. Arbstedt, Metals Technology 3, (5), 1976, 214.

"Fatigue Data Analysis," R. C. Rice, Metals Handbook, 9th Edition, Volume 8: Mechanical Testing, p. 695 - 720.

A Reliability Analysis of Fatigue Limits Based on Large Sample Quintal Response Data," R. E. Little, in Mechanical Behavior of Materials, Vol. V, The Society of Materials Science, Tokyo, 1972, p. 471 - 479.

"Tables for Estimating Median Fatigue Limits," R. E. Little, STP 731, ASTM, Philadelphia, 1980.

"Estimating the Median Fatigue Limit for Very Small up-and-down Quantal Response Tests and for S-N Data with Runouts," in Probabilistic Aspects of Fatigue, STP 511, ASTM, Philadelphia, 1972, p. 29 - 42.

Private communication, Ulf Engstrom, Hoganas, AB, Sweden.

	Comp. Press. (tsi)	GD (g/cm^3)	SD (g/cm^3)	Sint. Carbon (Wt. %)	ΔL (%)	Std. Dev. S (n=5)	Apparent Hardness (HRB)	R.T. Impact Energy (ft lbs)	Std. Dev. S (n=5)	$R_{p0.2}$ (ksi)	Std. Dev. S (n=5)	R_m (ksi)	Std. Dev S (n=5)	% Elong. (in 1 in.)	Std.Dev S (n=5)
Elemental	33.2	6.91	6.81	0.53	+0.39	0.01	77	8.5	0.5	52.8	1.3	68.7	1.1	1.9	0.2
Partially Prealloyed	34.2	6.89	6.88	0.52	+0.14	0.00	73	10.5	1.4	60.8	1.9	85.5	1.4	2.6	0.2
Prealloyed	46.5	6.89	6.94	0.51	-0.16	0.01	74	8.0	0.0	64.4	0.8	78.6	2.3	1.8	0.3
Elemental	45	7.16	7.06	0.54	+0.48	0.02	84	12.4	0.4	59.1	2.7	86.0	1.5	2.8	0.2
Partially Prealloyed	45	7.15	7.11	0.54	+0.24	0.00	94	15.8	0.8	62.4	2.7	98.9	1.3	3.5	0.2
Prealloyed	45	6.86	6.90	0.51	-0.18	0.005	79	8.0	0.4	69.8	3.0	86.3	1.9	1.7	0.2
Prealloyed Sumiron 4100S	45	7.03	7.07	0.52	-0.06	0.005	89	10.4	0.5	74.0	1.4	94.7	0.6	2.5	0.3

Table 2: Summary of processing conditions plus impact and tensile properties. (All samples sintered for 30 minutes at temperature in D.A. except for Sumiron 4100S which were sintered at 2300°F.)

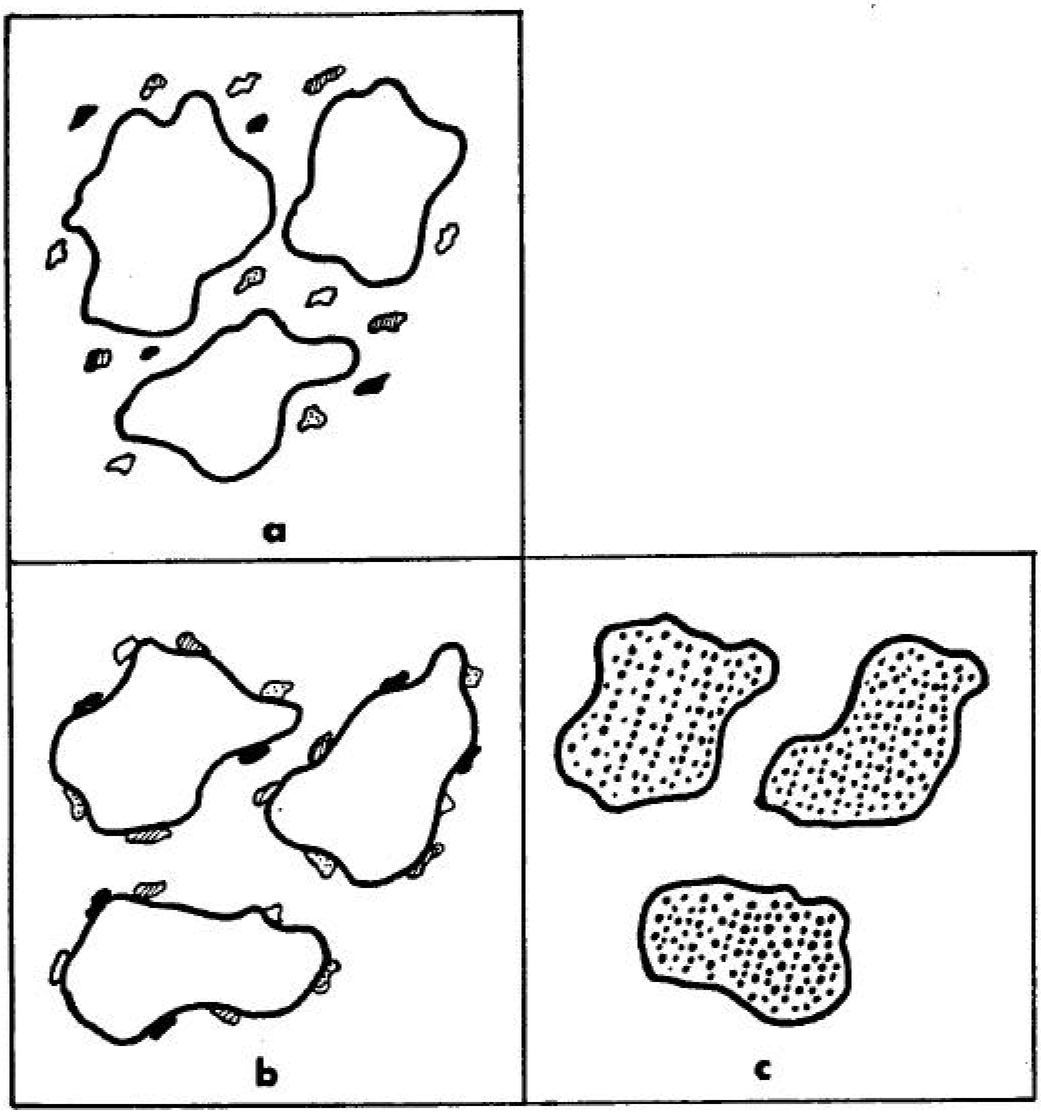


Figure 1: Alloying Methods in Ferrous P/M - schematic

a = elemental admixture
b = partially prealloyed powder
c = completely prealloyed powder

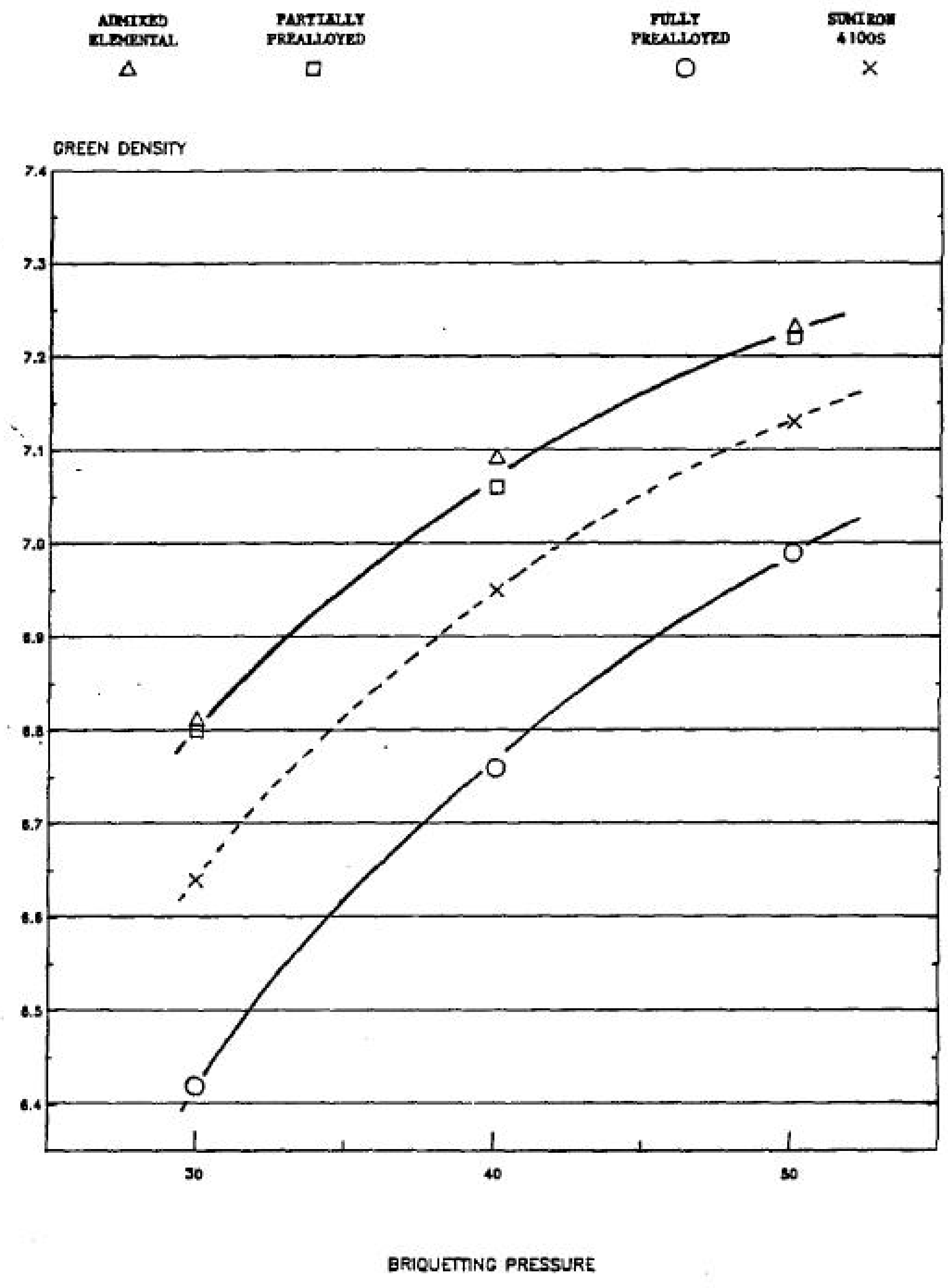


Figure 2: Compressibility curves for the various powders. (Each material contained 0.6 wt. % graphite and 0.5 wt. % Acrawax.)

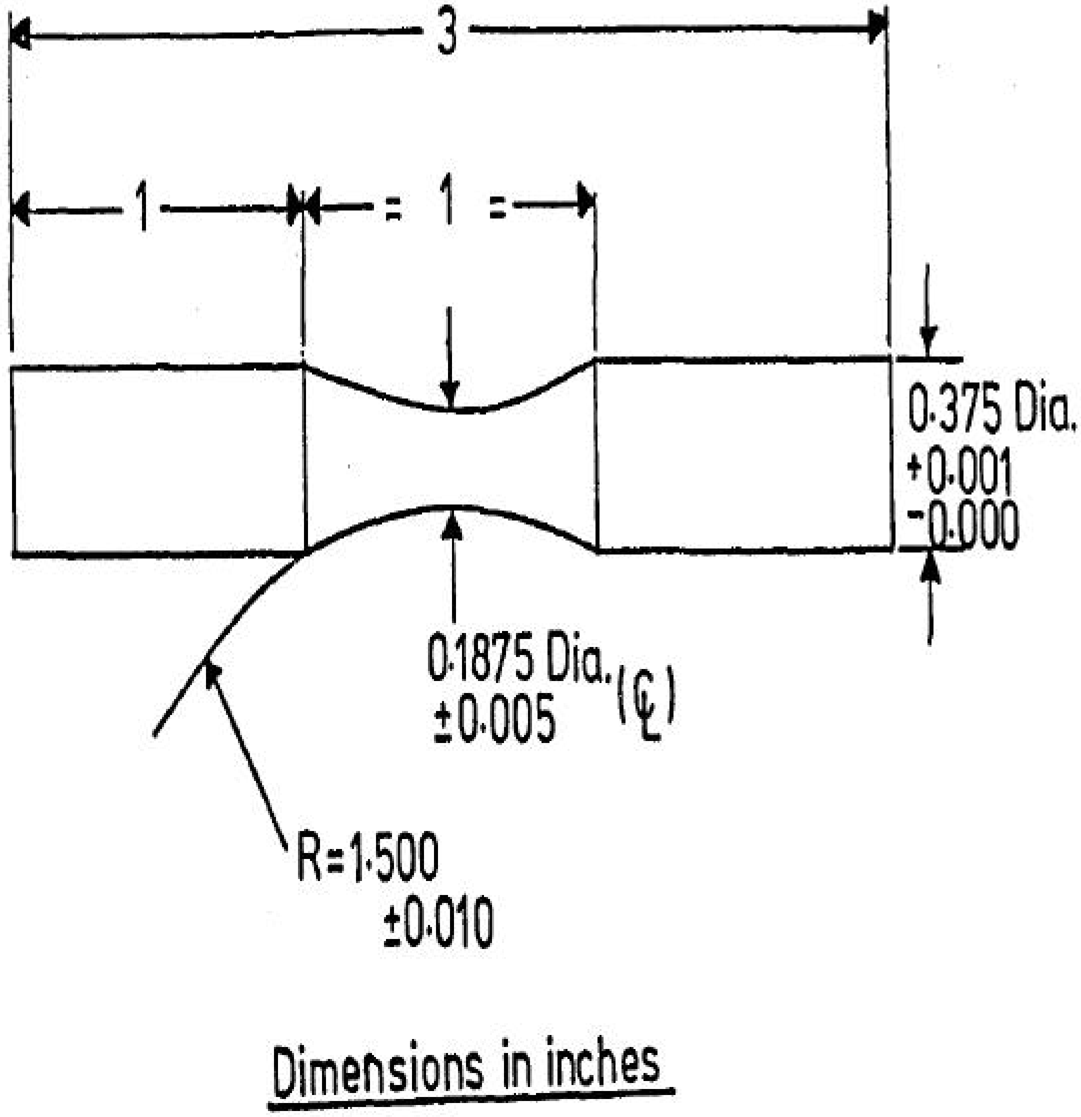


Figure 3: Rotating Bending Fatigue (RBF) Test Piece

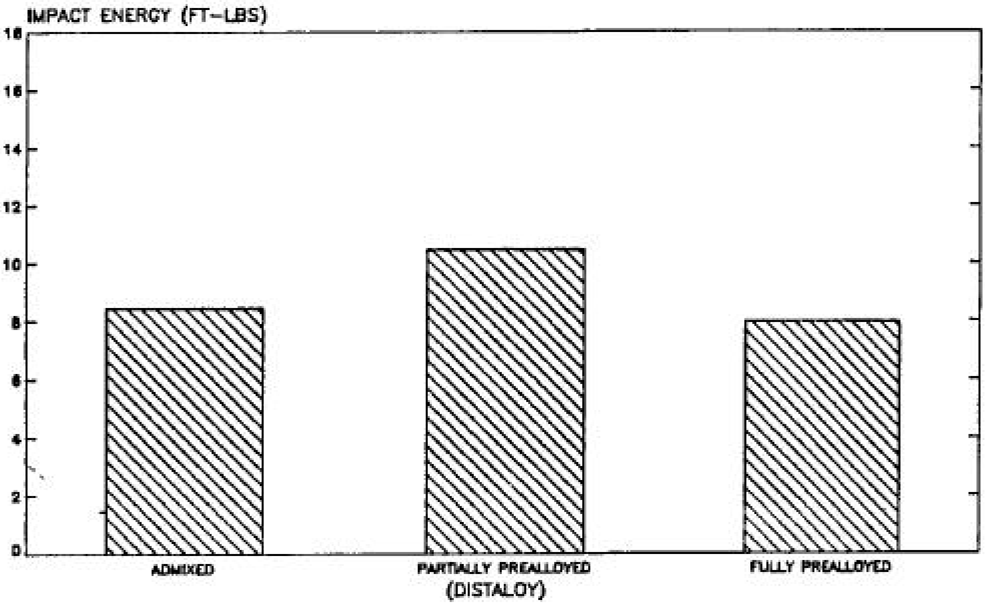


Figure 4: Room temperature unnotched Charpy impact energy data for samples pressed to a green density of 6.9 g/cm^3. (Sintered in dissociated ammonia for 30 minutes at a temperature of 2050°F.)

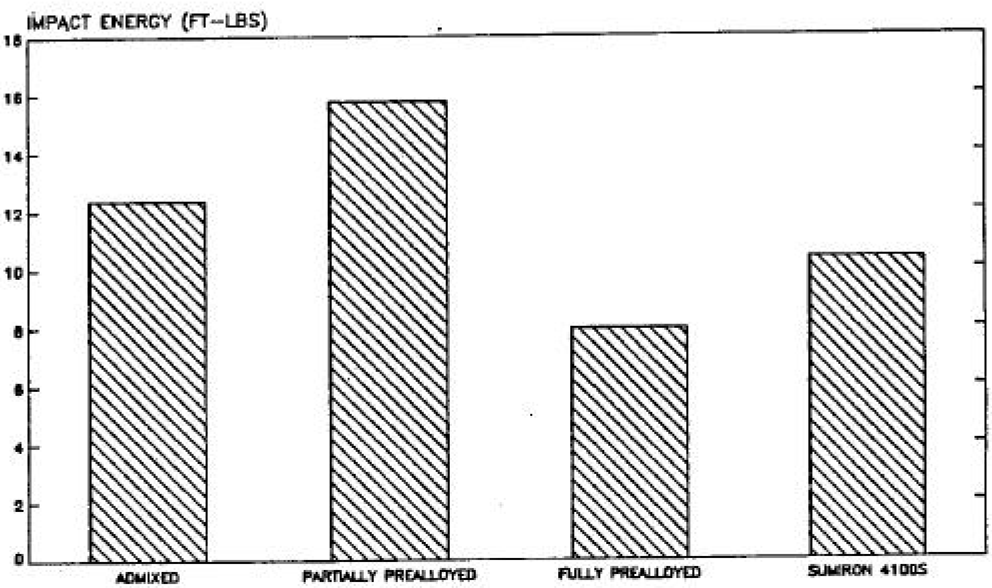


Figure 5: Room temperature unnotched Charpy impact energy data for samples compacted at 45 tsi. (Sintered in dissociated ammonia for 30 minutes at 2050°F - Sumiron 4100S, 30 minutes at 2300°F.)

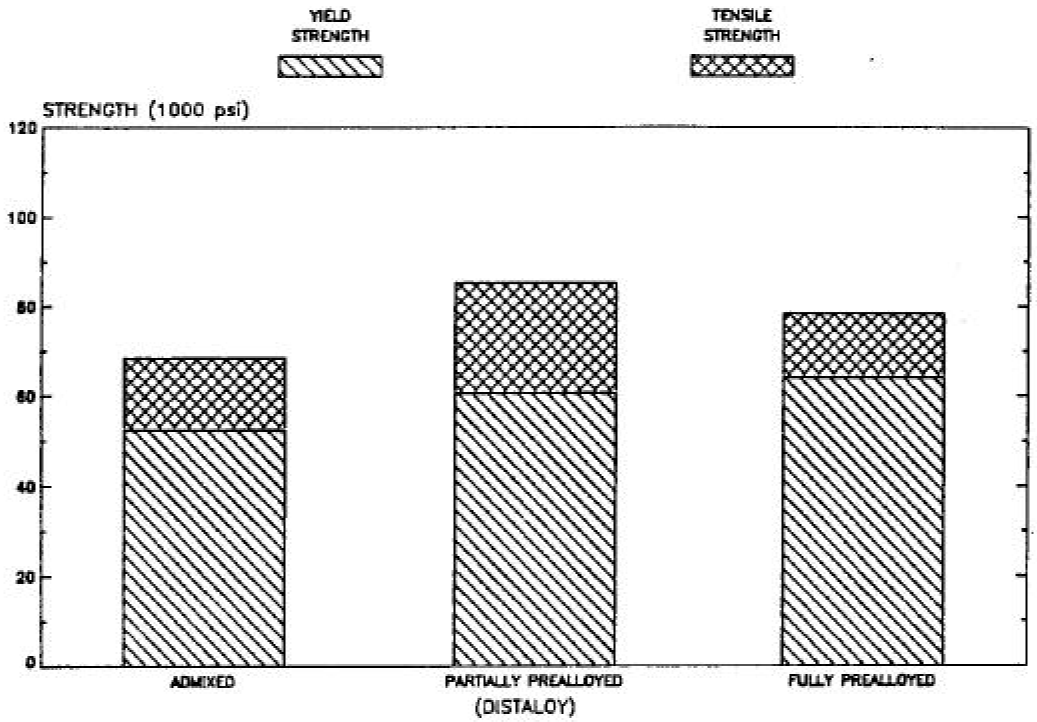


Figure 6: 0.2% offset yield strength ($R_{p0.2}$) and UTS data (R_m) for samples pressed to a green density of 6.9 g/cm^3. (Sintered in dissociated ammonia for 30 minutes at 2050°F.)

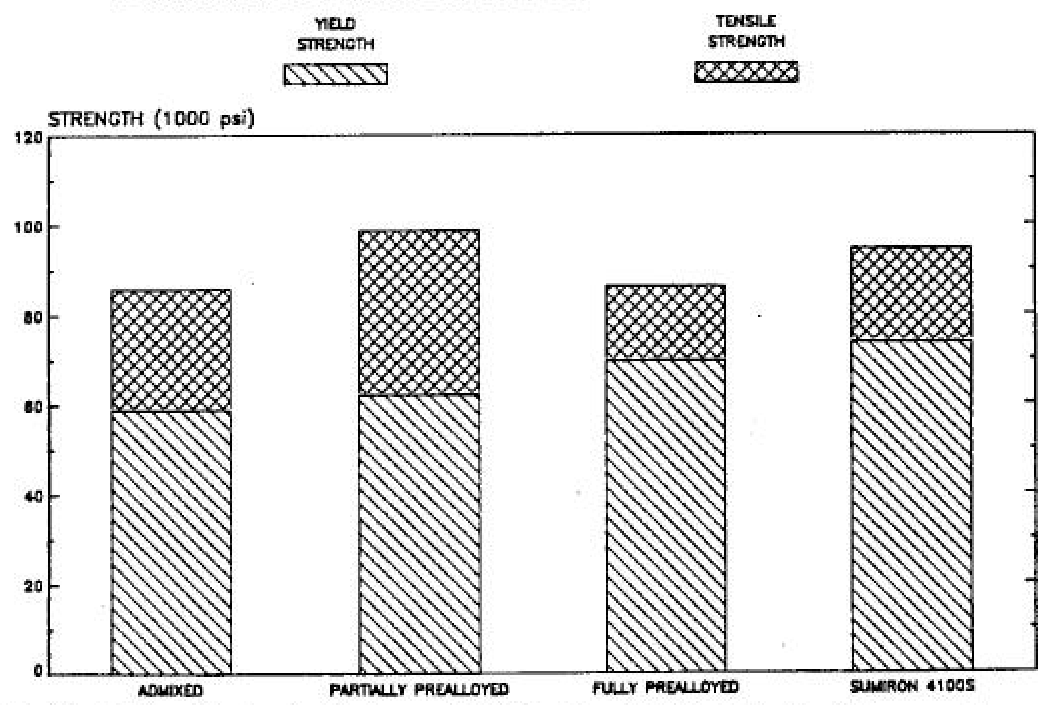


Figure 7: 0.2% offset yield strength ($R_{p0.2}$) and UTS data (R_m) for samples compacted at 45 tsi. (Sintered in dissociated ammonia for 30 minute at 2050°F - Sumiron 4100S, 30 minutes at 2300°F.)

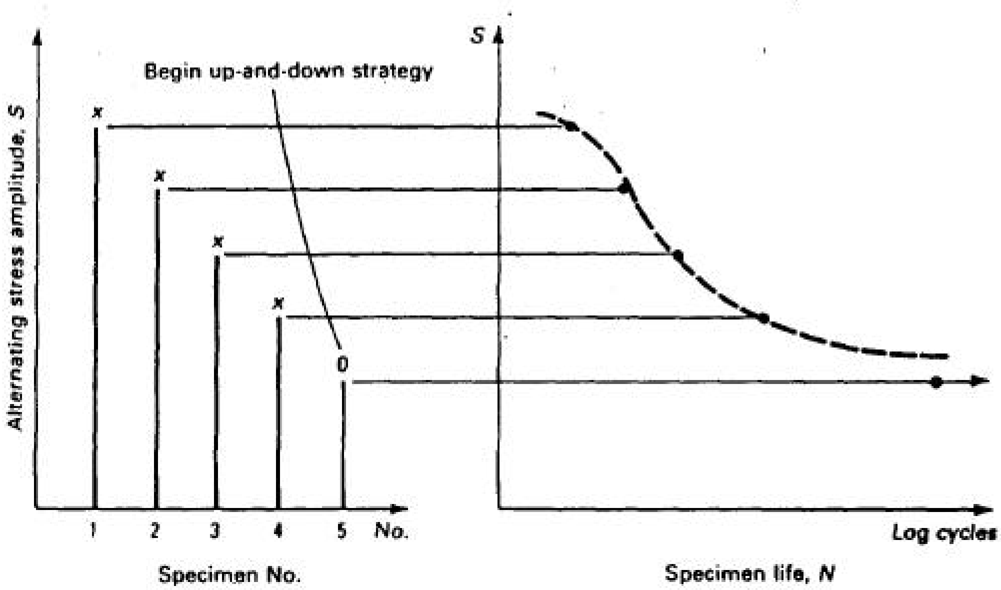


Figure 8: Schematic of S/N data generated as a preliminary test for subsequent two point program beginning with up and down strategy.

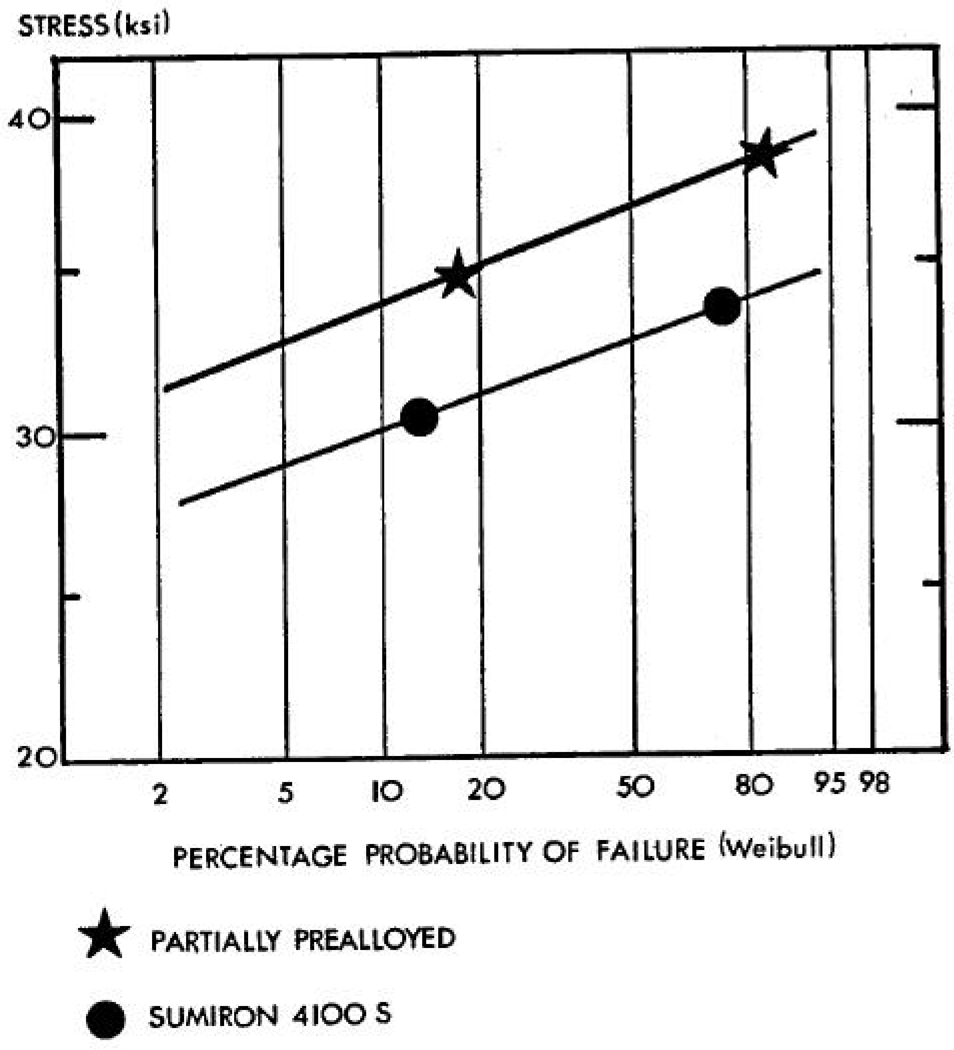


Figure 9: Stress versus the percentage probability of failure in rotating bending fatigue. (Samples compacted at 45 tsi and sintered for 30 minutes at temperature in a dissociated ammonia atmosphere. The partially prealloyed material was sintered at 2050°F and the Sumiron 4100S was sintered at 2300°F.)

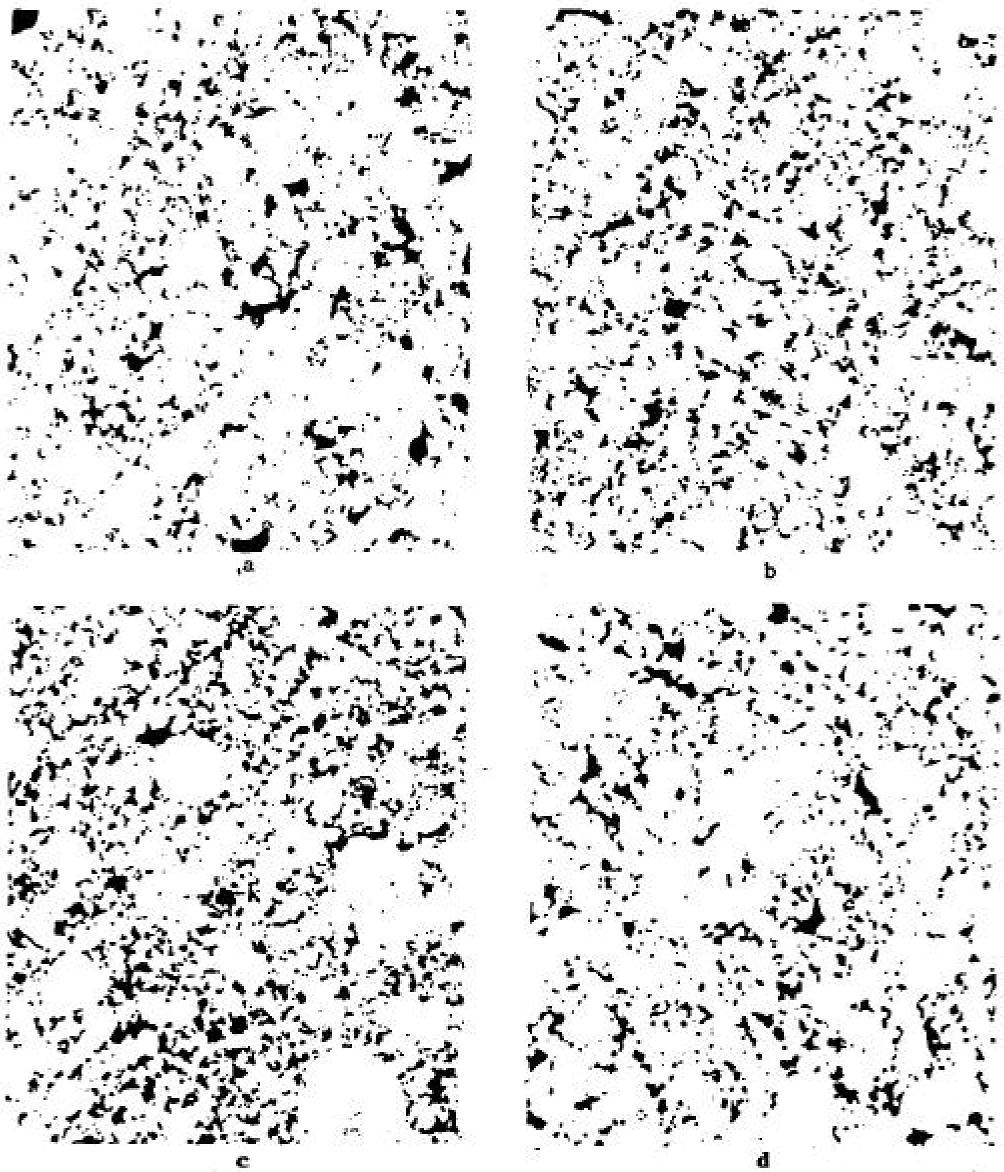
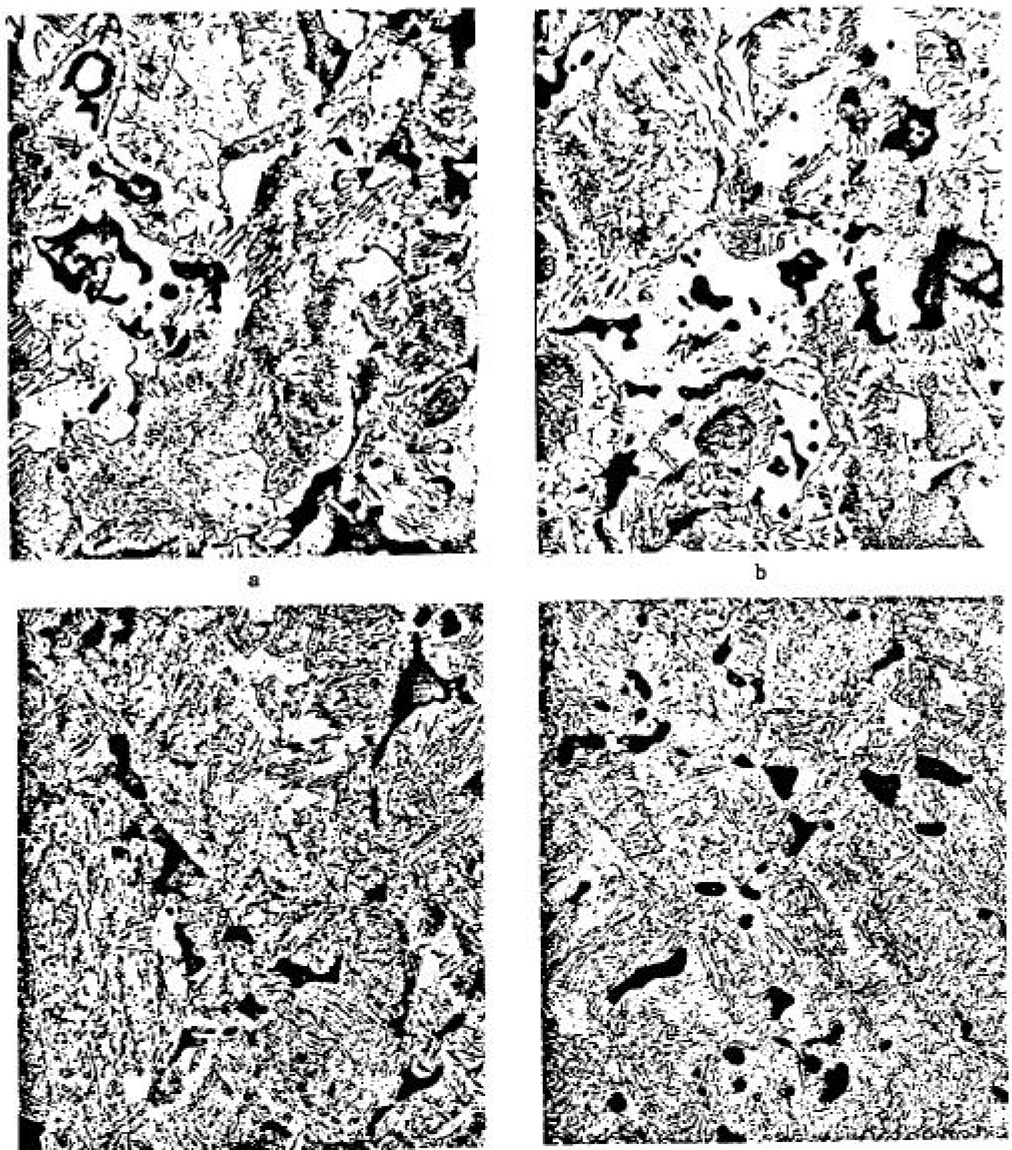


Figure 10: Photomicrographs of the unetched microstructures of the various samples: x100. (Samples compacted at 45 tsi and sintered at 2050°F for 30 minutes at temperature in a dissociated ammonia atmosphere – Sumiron 4100S sintered at 2300°F.)

a = elemental admixture
b = partially prealloyed material
c = prealloyed material
d = Sumiron 4100S



a b c d

Figure 11: Photomicrographs of the etched microstructures of the various samples: x500. (Samples compacted at 45 tsi and sintered at 2050°F for 30 minutes at temperature in a dissociated ammonia atmosphere - Sumiron 4100S sintered at 2300°F.)

a = elemental admixture
b = partially prealloyed material
c = prealloyed material
d = Sumiron 4100S

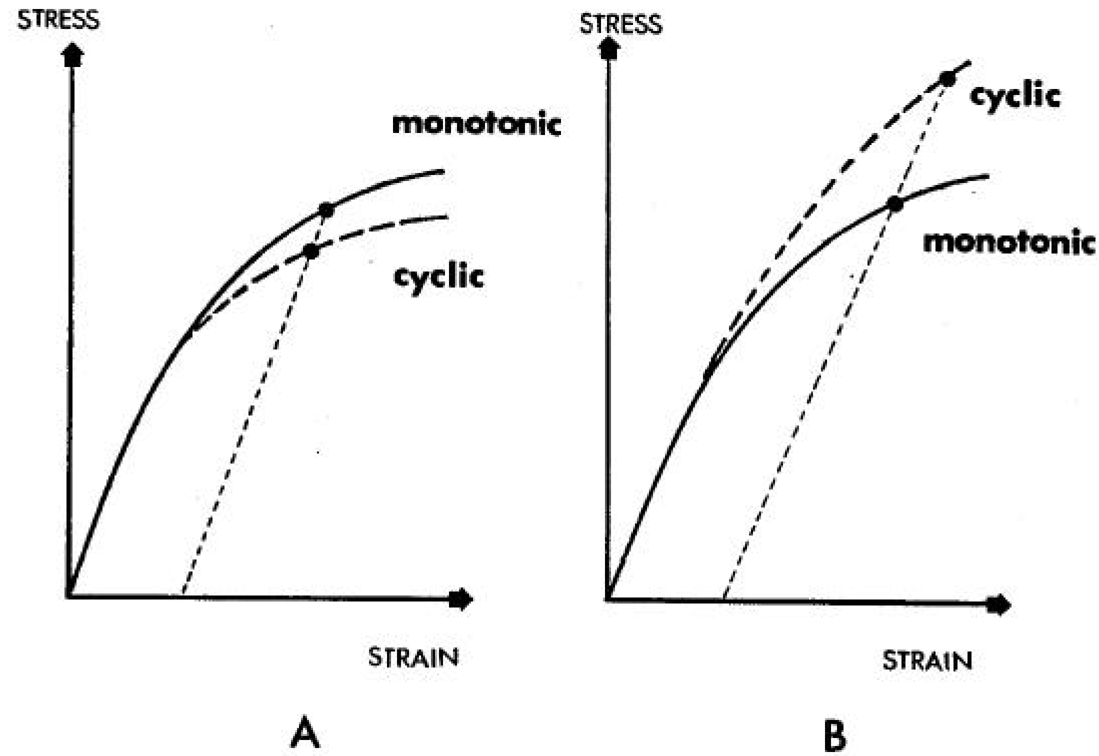


Figure 12: Monotonic and cyclic stress–strain curves.
A = cyclic softening
B = cyclic hardening

	Wt. % Ni	Wt. % Cu	Wt. % Mo	Balance
Elemental Admixture	1.89	1.79	0.66	Fe
Partial Prealloy	1.77	1.60	0.59	Fe
Prealloy	1.81	1.57	0.57	Fe
	Wt. % Cr	Wt. % Mn	Wt. % Mo	Balance
Sumiron 4100S (prealloy)	1.10	0.70	0.31	Fe

Table 1: Chemical Analyses of the Alloys