

# **HIGH PERFORMANCE FERROUS P/M MATERIALS UTILIZING HIGH TEMPERATURE SINTERING**

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## **ABSTRACT**

Several new and more challenging P/M applications require materials that exhibit higher strength along with improved dynamic property characteristics. To meet these requirements, development efforts focused on material grades capable of achieving high performance properties when sintered at elevated temperature.

Several elements, specifically nickel and copper, were admixed to the water atomized, prealloyed low-alloy steel powders Ancorsteel<sup>®</sup> 85 HP and 150 HP using the patented ANCORBOND<sup>®</sup> process. Tensile and impact performance of the resulting materials have been reviewed along with quantitative metallography of selected as-sintered samples.

The results indicate the type and amount of each admixed element plays an important role in achieving specific characteristics. In addition, the overall performance values are greater for the single press and sinter technique used in this investigation than could previously be achieved using a double press - double sinter process.

## **INTRODUCTION**

The material development program was a cooperative effort to formulate high performance materials that exhibit enhanced properties when sintered at elevated temperature ( i.e. above the conventional 2050° F cycle). Efforts focussed primarily on increased strength and dynamic performance along with compaction characteristics. The high performance materials program established minimum target values of 120,000 psi ultimate tensile strength, 75,000 psi yield strength, unnotched Charpy impact energy of 25 ft. lbf, 3 % elongation, and 40,000 psi rotating bending fatigue (RBF) strength. It was stipulated the properties must be achieved in the as-sintered condition using a single compaction and sinter process without any subsequent thermal or mechanical treatment These properties approximate the typical values of austempered ductile iron and were selected expressly for the unique combination of strength and toughness. Previous efforts to achieve similar performance levels typically involved various combinations of high percentage admixed materials or partially alloyed grades that were processed using a double press - double sinter operation. This approach realized some success but was often found not to be cost effective or was limited to simple geometric shapes.

One of the options considered was to sinter, at elevated temperature, the high compressibility, prealloyed powders, Ancorsteel 85 HP and Ancorsteel 150 HP with admixed additions of nickel and copper. Both grades complement the high temperature process because they can be compacted to high green densities which allows each to realize greater densification from sintering. The selected prealloyed powders exhibit a homogeneous distribution of molybdenum which enhances strength and hardenability without reducing compressibility. Earlier work involving nickel, copper and graphite additions processed at conventional temperature (2050°F) showed promising results <sup>[1,2]</sup>.

Admixing nickel to the referenced prealloyed grades looked particularly promising. Previous investigations using various base powders found nickel diffused more homogeneously when sintered above 2300' F resulting in greater strength and hardenability <sup>[3,4,5]</sup>. Furthermore, investigators found with higher nickel additions the pores in these materials were often located in, or surrounded by, a nickel rich phase which is thought to reduce the transfer of stresses<sup>[6]</sup>, thereby improving dynamic performance. Copper additions were also found to marginally increase tensile, yield and impact strength <sup>[4,5]</sup>. Copper can also control the high shrinkage rate common with greater percentage nickel additions and effectively enhance dimensional stability. However, large pores that remain after sintering and the promotion of grain growth have been reported to off-set any gains realized with copper additions <sup>[3,5]</sup>.

The high temperature sintering process greatly improves interparticle bonding while reducing the number of pores and rounding those that remain. This promotes improved impact energy, fatigue strength and elongation performance for many compositions.

## **EXPERIMENTAL PROCEDURE**

### **Materials**

The materials used in this investigation represent ANCORBOND<sup>[7]</sup> pilot production premixes. This technology bonds admixed elements to the base iron which significantly reduces segregation, dusting loss, and minimizes the material's overall performance variability <sup>[8]</sup>. The base irons used for the study were the prealloyed Ancorsteel 185 HP ( 0.85% Mo.) and Ancorsteel 150 HP ( 1.50% Mo.) grades. Additions of 0.50% Asbury 3203 natural graphite and 0.75% Mallinckrodt XM-Ultrafine zinc stearate were made to each premix. The remaining elemental additions consisted of Inco 123 nickel powder and Alcan 8081 copper powder. The various premix additions and nominal base iron compositions are summarized in Table 1.

### **Test Specimen Preparation**

Tensile specimens conforming to the ASTM E8 unmachined 'dog-bone' configuration were used for testing. Impact properties were developed using both unnotched Izod and Charpy specimens as detailed in ASTM E23. We observed good correlation between test values for both types of specimens. However, we chose to report ambient room temperature Charpy values because this information is more commonly referenced for P/M materials. Dimensional change values and the comparison between green to sintered density were generated from one inch diameter specimens. Samples representing each composition, test procedure and specimen type were single pressed at 30, 40 and 50 tsi compaction pressure.

## **Sintering**

The sintering process used by Remington Powder Metal Products reflects production conditions using a Lindberg high temperature pusher furnace with all specimens placed on fiat ceramic plates. The sintering cycle was 2400° F for 45 minutes at temperature with a relatively slow cooling rate in a multi-zoned, water jacketed cooling section. The protective atmosphere consisted of 75% nitrogen - 25% dissociated ammonia and maintained a -30° C dew point

## **Metallography**

Cross sections for metallographic examination were prepared using exacting procedures to insure a proper surface for quantitative metallography. An automated image analyzer, a Leitz TAS-PIs, was used to determine pore size and shape. Pore size was determined by area (e.g. circular pore with a diameter of 50  $\mu\text{m}$  -- area of 1963  $\mu\text{m}^2$ ) using intervals that ranged between 0 - 2000 $\mu\text{m}^2$  and distinguished by increments of 100 $\mu\text{m}^2$ . Shape assessment was determined using the parameter  $4\pi A/P^2$  ( A - area, P - perimeter ) which equates the relative degree of circularity. For a perfectly circular pore,  $4\pi A/P^2$  equals 1 while irregularly shaped pores have a value less than 1. The specific procedures were detailed in a previous materials development <sup>[9]</sup>.

All samples were etched with 2% nital + 4% picral to define the microstructural constituents of selected materials. This provides a quantitative estimate for comparison purposes and contributes to a better understanding of each material's structure / property relationship. Selected samples were tested using a 20 point grid (point spacing of 20  $\mu\text{m}$ ) at 500 diameters magnification. A systematic point count was performed to determine the volume ( $V_v$ ) percent of each microstructural constituent in twenty random fields/sample. The microstructural constituents are illustrated in Figures 1-2 with the total volume percent for each of the selected compositions identified in Table 2. Figures 3 and 4 represent unetched photomicrographs that illustrate pore morphology.

## **RESULTS**

The properties of high temperature sintered materials are listed in Tables 3 and 4. This information is also presented in graphical form in Figures 5-9, with sintered density expressed as a curve plot while tensile, hardness, impact and dimensional change are represented as trend line plots. Performance comparisons between base low-alloy grades for selected properties at a fixed compaction pressure are represented in Figures 10-15. Graphs illustrating the densification that occurs between green and sintered specimens are presented in Figure 16.

Testing was duplicated at Hugeness and Remington with no significant differences observed between results. The reported values represent an average of five determinations per condition. Size change was measured as the difference from the die dimension and reported as a percentage. Yield strength was calculated using a 0.2% off-set and the elongation values represent a 1 inch gauge length.

## **DISCUSSION**

Density and composition are two prominent factors which influence a P/M component's strength and

dynamic performance. Sintered density plays a particularly important role in achieving higher performance levels. To fully take advantage of high temperature sintering, the pressed density should be as high as possible. This results in greater particle surface contact which activates the sintering response, effecting greater densification and improved interparticle bonding. Specimens single pressed to  $>7.10 \text{ g/cm}^3$  using 40 tsi compaction pressure increased  $0.12 \text{ g/cm}^3$  in density when admixed with 4% nickel. This subsequently dropped to a  $0.05 \text{ g/cm}^3$  increase when 1% copper was added to the same material. At the 6% nickel level density increased  $0.18 \text{ g/cm}^3$  and  $0.13 \text{ g/cm}^3$  with a similar 1% copper addition.

Elevated temperature sintering also increases diffusion of the admixed elements which adds to the material's strength and performance. Nickel and copper were both found to have completely diffused within the matrix. Consequently, the characteristics of each element, much like a prealloyed material, were influential in determining the material's overall performance. The selected nickel and copper additions enhanced strength by solid solution alloying along with improving hardenability. Nickel changes the material's transformation characteristics by lowering both the upper and lower critical temperatures and reducing the eutectoid carbon level. This element also acts as a grain refiner which is beneficial when high temperature sintering. The lower austenitizing temperature resulting from the nickel diffusion will likely off-set the higher (6%) temperature required with molybdenum steels. Copper additions can also enhance strength and hardenability when in solution with iron. However, with higher (6%) nickel additions, our findings indicate copper preferentially combines with nickel and adds little to the material's performance.

## **Microstructure**

Three microstructural constituents were observed in the materials examined. Nickel rich martensite and colonies of divorced pearlite were predominant with relatively small percentages of bainite comprising the balance. No appreciable amounts of ferrite or nickel rich areas were found that were not martensitic. The higher degree of nickel diffusion, with its previously stated influence on transformation, along with the prealloyed molybdenum and admixed graphite apparently provide sufficient hardenability to form greater percentages of martensite at relatively slow cooling rates. The 6% nickel addition formed greater percentages ( $>60\%$ ) of martensite than the 4% addition. The 1% copper addition at the higher nickel level had very little effect on transformation. However, with 4% nickel, the copper addition appears to have been more influential resulting in a 35% increase in martensite transformation when added to the Ancorsteel 150 HP base steel powder.

Tempering both tensile and impact specimens at  $400^\circ\text{F}$  for one hour had no beneficial effect on performance. This may be associated with a relatively low stress martensite formation in the nickel rich areas. Presumably, some refined austenite in the nickel rich phase may be a contributing factor. However, in production environments or more highly stressed conditions, a tempering operation may be appropriate considering the material's ability to transform to high percentages of martensite at relatively slow cooling rates.

All pores appear to be randomly distributed and not necessarily surrounded by a nickel rich phase. The pore size analysis indicates the 6% nickel materials show a greater number of overall pores in comparison to 4% nickel additions. However, the greater percentage of these fell below  $100\mu\text{m}^2$  in area. When 1% copper was added to either nickel addition a greater percentage of larger pores was

evidenced (Fig. 17-18). Pore shape analysis found a normal distribution regarding the degree of circularity (Fig. 19-20). This relates to the higher percentage nickel addition and resulting diffusion leaving many small pores that are somewhat elongated. However, all the pore edges are well rounded and appear to be migrating towards one another and showing a tendency to coalesce. These findings suggest that as nickel increases the total pore count increases, primarily because of the as-pressed, fine particle arrangement of nickel.

### **Tensile Properties**

The prealloyed molybdenum content and the complete diffusion of admixed elements had a significant influence on strength. The best overall tensile performance was achieved with a 6% nickel addition to Ancorsteel 85 HP. When single pressed at 50 tsi compaction pressure this material exhibited a 197,200 psi ultimate tensile strength, 145,200 psi yield strength and 38 URC apparent hardness with 2.8% elongation. A slightly greater graphite addition of 0.6% could potentially increase both tensile and yield strength to exceed 200,000 psi and 150,000 psi, respectively. Clearly, these values are equal or greater than typical double pressed - double sintered and/or quenched & tempered properties. For example, MPIF Standard 35 references typical properties for FN- 0405-180HT as 185,000 tensile strength with an apparent hardness of 40 HRC and < 0.5% elongation at a sintered density of 7.4 g/cm<sup>3</sup> achieved using a DP-DS process. If, on the other hand, the graphite addition had been decreased to 0.4% the elongation would likely be greater than the targeted 3.0% minimum yet still exceed by a significant margin the high performance material program's strength requirements.

In comparing the 4% vs 6% nickel additions, we found the additional 2% nickel increased yield and tensile strength by an average of 45% for both prealloyed base low-alloy grades. The greater percentage of nickel, however, did not dramatically improve elongation when added to either base steel powder. The 1% copper addition at the 6% nickel level had little influence other than a slight increase in yield strength with the 0.85% molybdenum steel. However, the 4% nickel - 1% copper addition increased strength by 12-20% with the 1.50% molybdenum grade showing the highest values at 165,310 psi tensile and 124,900 psi yield strength. The copper addition consistently reduced the elongation performance but did stabilize the dimensional response by reducing the degree of shrinkage.

### **Hardness and Impact Properties**

The hardness increased with greater nickel additions, which coincides with the tensile performance. Copper additions also increased hardness particularly in conjunction with 4% nickel, whereas it had a lesser impact with 6% nickel additions. Even though both base low-alloy grades exhibit similar hardness values, as represented by the test specimens, we would expect that under actual production conditions the 1.50% prealloyed molybdenum grade would exhibit greater hardenability and would be more appropriate for larger mass parts. Additionally, we found the as-sintered hardness shows a reasonably good correlation with the percentage of martensite formed for each of the examined conditions.

It has previously been documented that density and pore shape are most influential in determining impact performance. To the greater extent, our results parallel these findings. Nickel enhances impact energy because it adds toughness, generally results in a rounded - fine pore distribution and increases density when high tempered sintered. The 6% nickel premixes outperformed the 4% addition by a

range of 25-38% for both base low-alloy grades. The 1% copper, when added to either nickel level or base low-alloy powder, shows no benefit, and in some cases results in lower values. This is most likely caused by the formation of voids at the prior copper sites which result in large pores after sintering.

## **CONCLUSIONS**

When high temperature sintered, the selected materials exceeded the tensile and impact requirements established for the high performance material program. The primary objective of developing materials that exhibit high strength yet maintains good toughness characteristics has essentially been satisfied. It is anticipated that the fatigue performance of selected materials will also meet the required minimum performance level established for the program. Fatigue testing is currently in progress.

The high temperature sintering process in conjunction with the selected materials provides the capability for PM component manufacturers to advance to a higher, and increasingly more sophisticated parts development plateau. Previous restrictions involving multiple pressing operations or costly secondary processes to increase performance are no longer necessary. Significant improvements in part design and related manufacturing economics can also be realized by using stronger, tougher materials to redesign existing parts by reducing cross sections or to increase component durability.

## **ACKNOWLEDGEMENTS**

The authors would like to thank B. Dodson and S. Bridges of Remington Powder Metal Products for their valued assistance in preparing and testing the specimens. We also appreciate the cooperation of R. Fitzpatrick and C. Gamble for their help in mechanical property testing, and grateful to T. Murphy and S. Kolwicz for their assistance in microstructural evaluation and quantitative metallography.

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**Table 1 - Bonded elements and base low-alloy chemistry**

A	B	C	D
4 % Nickel	4 % Nickel 1 % Copper	6 % Nickel	6 % Nickel 1 % Copper

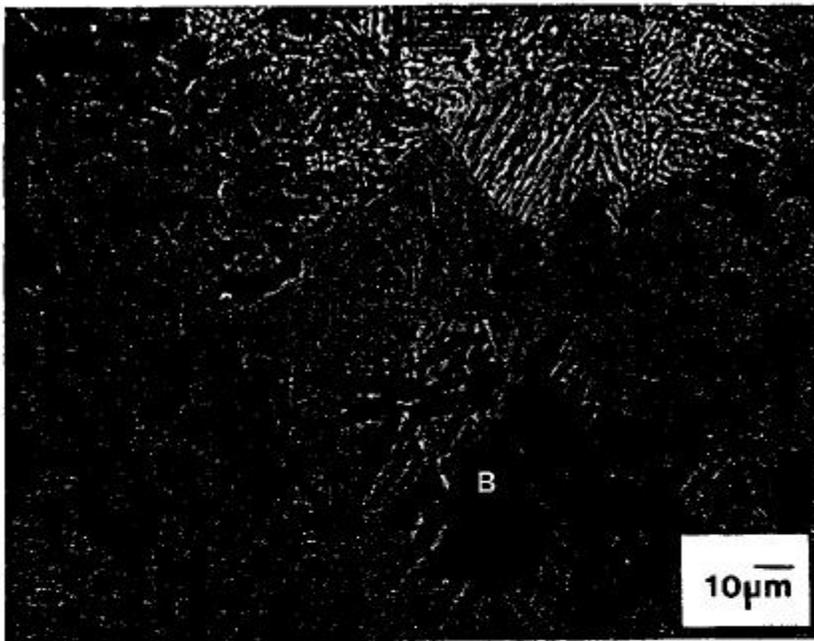
Plus 0.5% graphite and 0.75% zinc stearate

**Nominal, wt%**

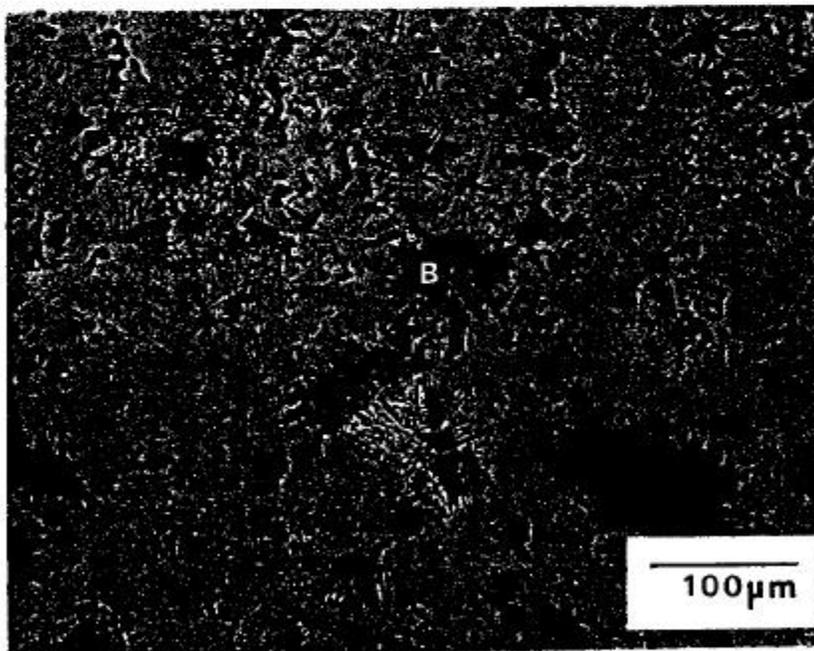
Ancorsteel Grades	Carbon	Manganese	Molybdenum	Oxygen
85 HP	<0.01	0.14	0.85	0.07
150 HP	<0.01	0.14	1.50	0.07

**Table 2 - Volume percent of microstructural constituents.  
Specimens compacted at 40 tsi.**

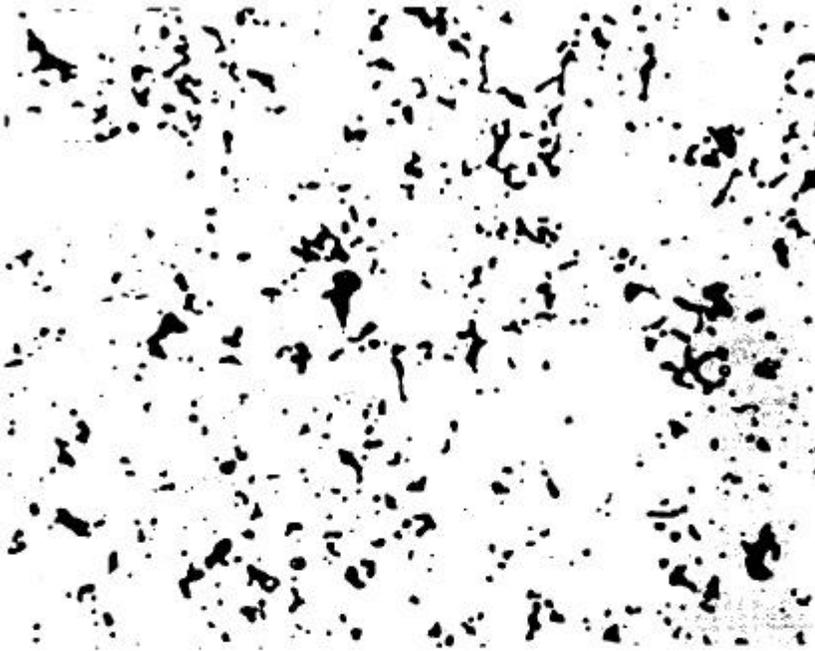
Composition	Divorced Pearlite	Martensite	Bainite	Estimated Porosity
85HP+6NI	26.75	62.25	6.00	5.00
85HP+6NI+1Cu	19.50	69.50	3.00	8.00
150HP+6NI	25.50	58.25	10.50	5.50
150HP+4NI	65.00	23.50	5.00	6.50
150HP+4NI+1Cu	24.50	58.25	8.00	9.25
85HP+4NI+1Cu	37.25	48.25	7.75	6.75



**Figure 1 - Photomicrograph of 0.85% Mo + 6% Ni + 0.5% gr etched with 2% nital + 4% picral at 500X magnification. Specimens were pressed to 40 tsi compaction pressure. ( DP - Divorced Pearlite, M - Martensite, B -Bainite )**



**Figure 2 - Photomicrograph of same material at 200X magnification showing constituent distribution and relative degree of uniformity.**



**Figure 3 - Unetched photomicrograph of 0.85% Mo + 6% Ni + 0.5% gr specimen pressed to 40 tsi compaction pressure. Shows fine pore distribution with rounded pore perimeters. Magnification 200X.**



**Figure 4 - Same as Figure 3 but with 1% copper addition. Larger pores are readily evidenced.**

**Table 3 - Sintered Performance of 0.85% Molybdenum Grade**

Admixed Additions	Compaction Pressure, tsf	Tensile psi	Yield psi	Elongation %	Impact ft.lbf.	Hardness HRC	Size Change, %	Density
4% Ni	30	100380	78290	1.9	12	8	-0.77	7.04
	40	116700	86820	2.5	20	15	-0.58	7.25
	50	131520	96140	3.0	24	23	-0.47	7.35
4% Ni-1% Cu	30	119370	96260	1.5	13	18	-0.48	6.97
	40	137580	103220	2.1	18	25	-0.34	7.18
	50	146980	106700	2.2	22	26	-0.33	7.30
6% Ni	30	150820	116290	1.8	19	33	-1.07	7.07
	40	182270	135860	2.5	26	37	-0.83	7.29
	50	197200	145200	2.8	33	38	-0.70	7.41
6% Ni-1% Cu	30	154770	125200	2.0	18	31	-0.83	7.04
	40	180900	143840	2.2	26	37	-0.62	7.25
	50	198580	157630	2.5	30	40	-0.48	7.36

**Table 4 - Sintered Performance of 1.50% Molybdenum Grade**

Admixed Additions	Compaction Pressure, tsf	Tensile psi	Yield psi	Elongation %	Impact ft.lbf.	Hardness HRC	Size Change, %	Density
4% Ni	30	107200	91200	1.5	13	12	-0.63	7.00
	40	132340	103040	2.2	18	23	-0.51	7.22
	50	139820	109340	2.4	24	26	-0.41	7.35
4% Ni-1% Cu	30	125760	105380	1.3	14	26	-0.51	6.96
	40	152800	116890	1.8	19	32	-0.36	7.16
	50	165310	124900	1.9	25	35	-0.24	7.30
6% Ni	30	144950	115060	1.7	17	30	-0.96	7.04
	40	179470	133480	2.2	24	36	-0.81	7.29
	50	198680	154140	2.5	32	39	-0.69	7.41
6% Ni-1% Cu	30	141480	116350	1.5	18	32	-0.80	7.01
	40	171220	128490	1.7	26	38	-0.61	7.23
	50	185450	141560	1.9	30	40	-0.47	7.35

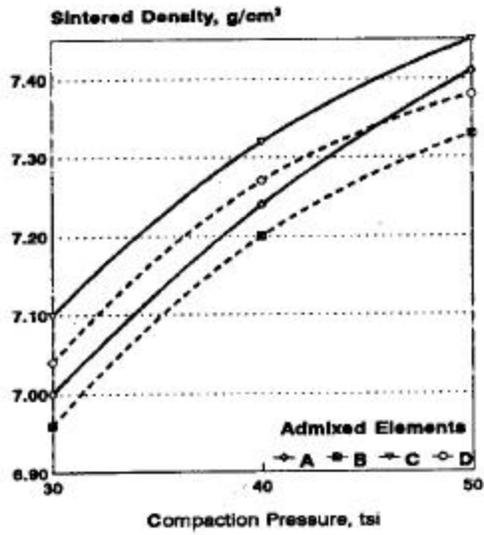


Figure 5A - 0.85% Molybdenum Grade

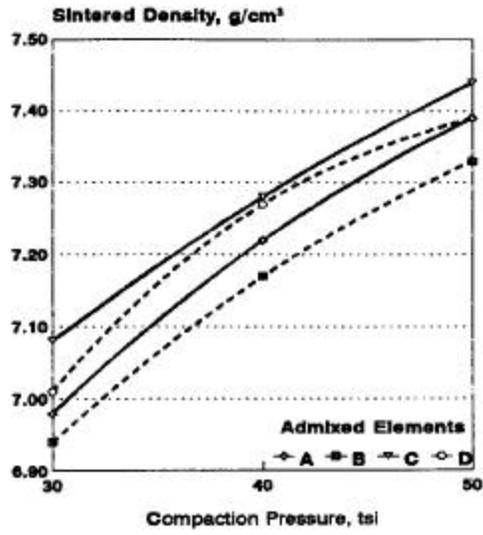


Figure 5B - 1.50% Molybdenum Grade

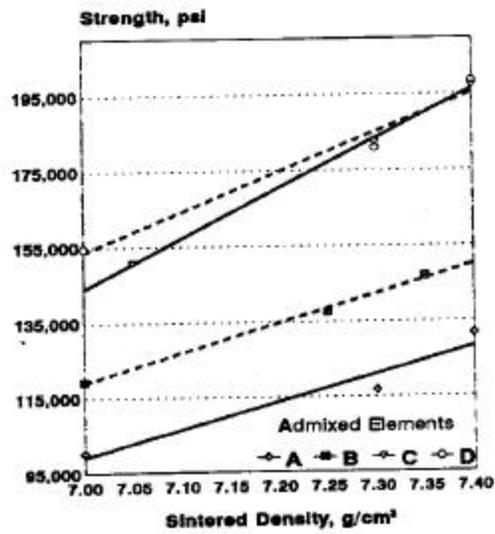


Figure 6A - Ultimate Tensile for 0.85% Mo Grade

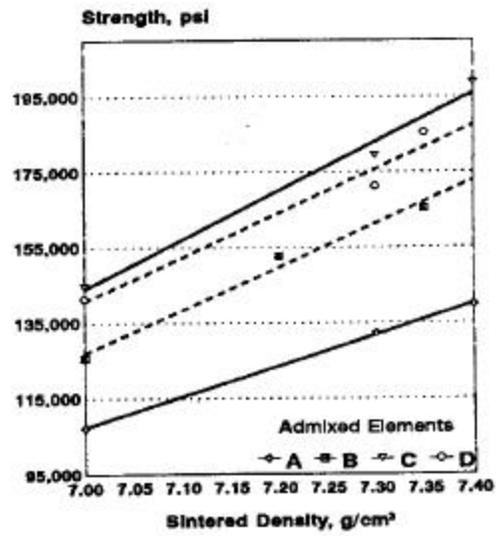


Figure 6B - Ultimate Tensile for 1.50% Mo Grade

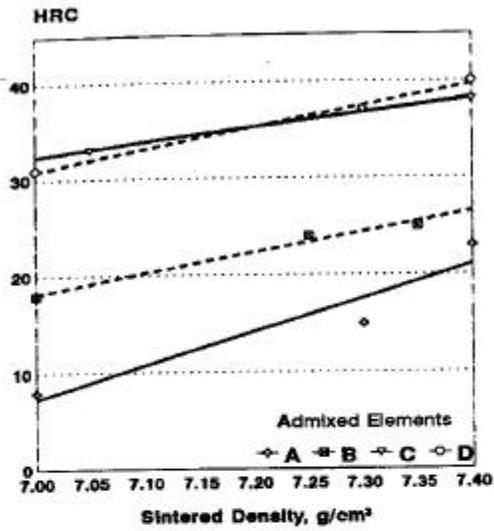


Figure 7A - Sintered Hardness for 0.85% Mo Grade

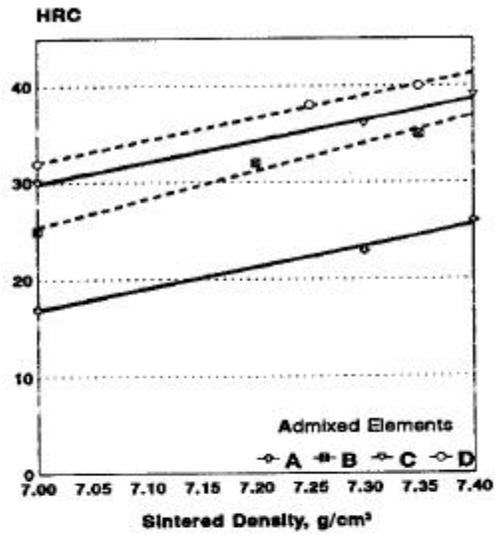


Figure 7B - Sintered Hardness for 1.50% Mo Grade

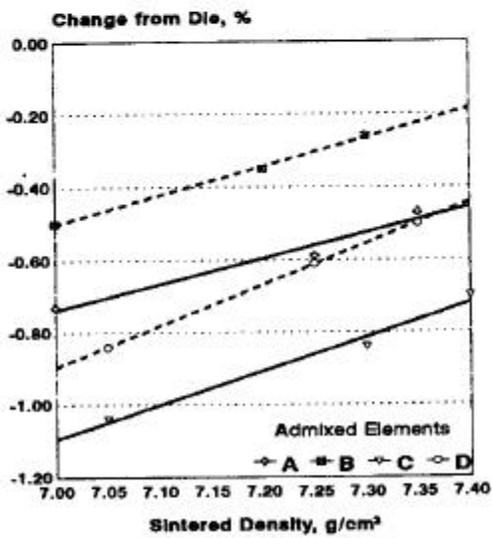


Figure 8A - Dimensional Change for 0.85% Mo Grade

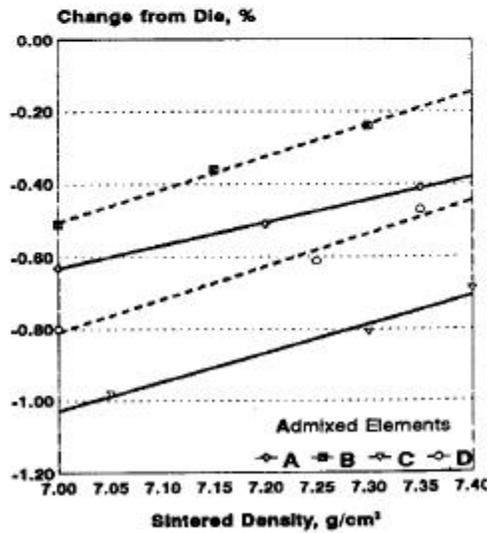


Figure 8B - Dimensional Change for 1.50% Mo Grade

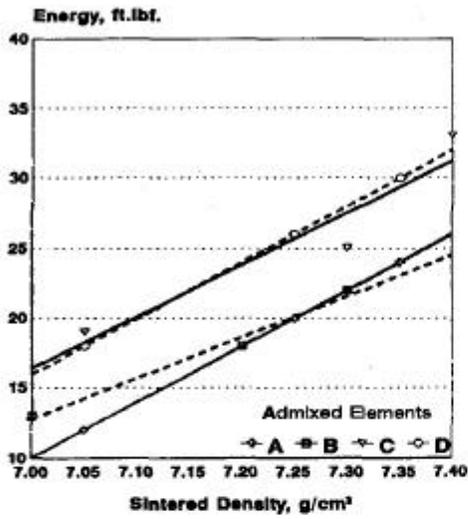


Figure 9A - Charpy Impact for 0.85% Mo Grade

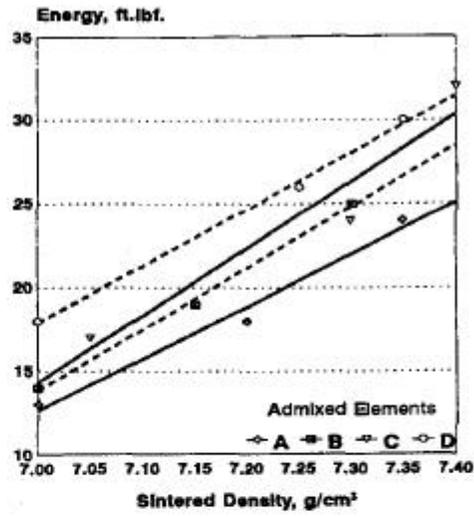


Figure 9B - Charpy Impact for 1.50% Mo Grade

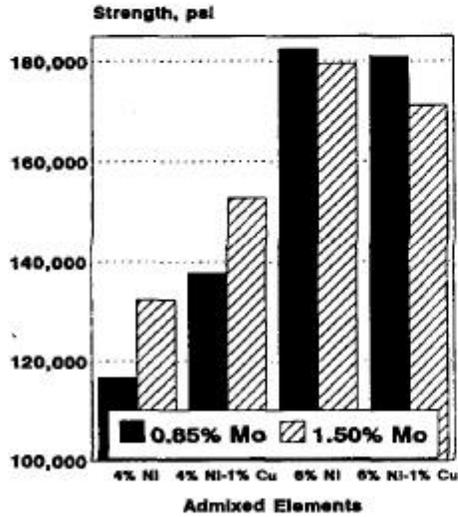


Figure 10 - Tensile Comparison using 40 ksi specimens

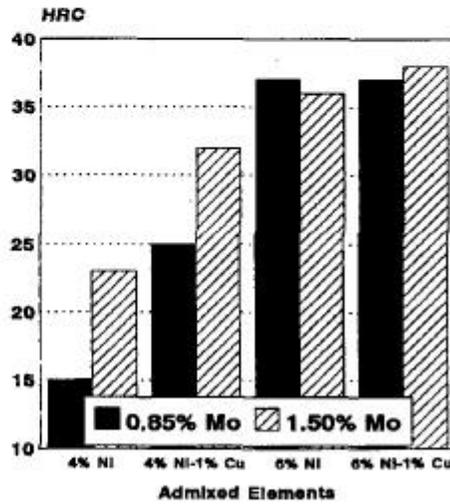


Figure 11 - Hardness Comparison using 40 ksi Specimens

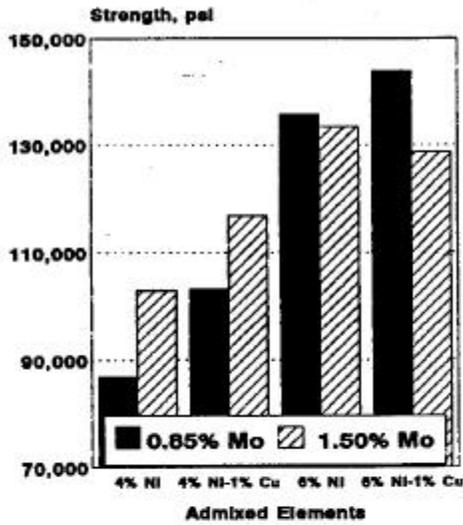


Figure 12 - Yield Comparison using 40 ksi specimens

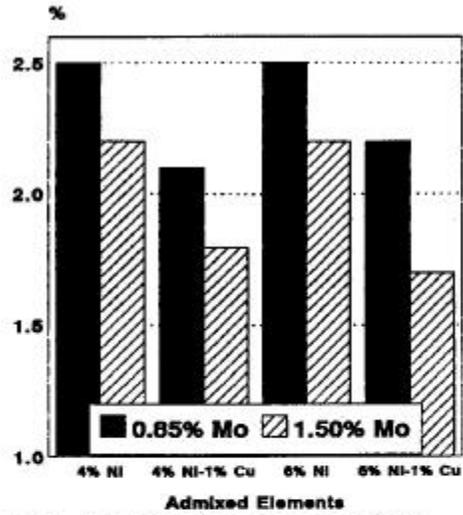


Figure 13 - Elongation Comparison using 40 ksi specimens.

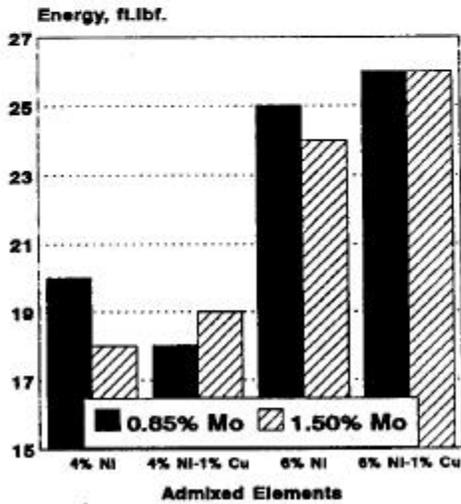


Figure 14 - Charpy Comparison using 40 ksi Specimens

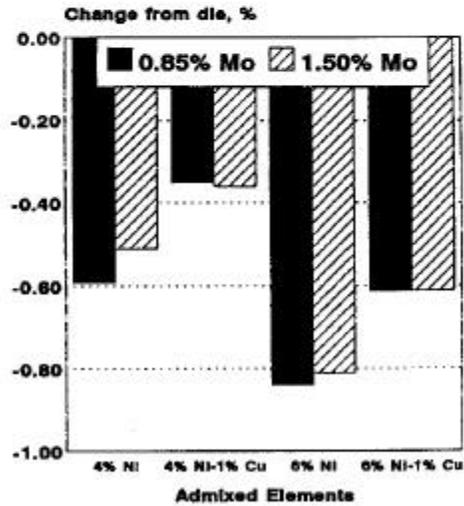
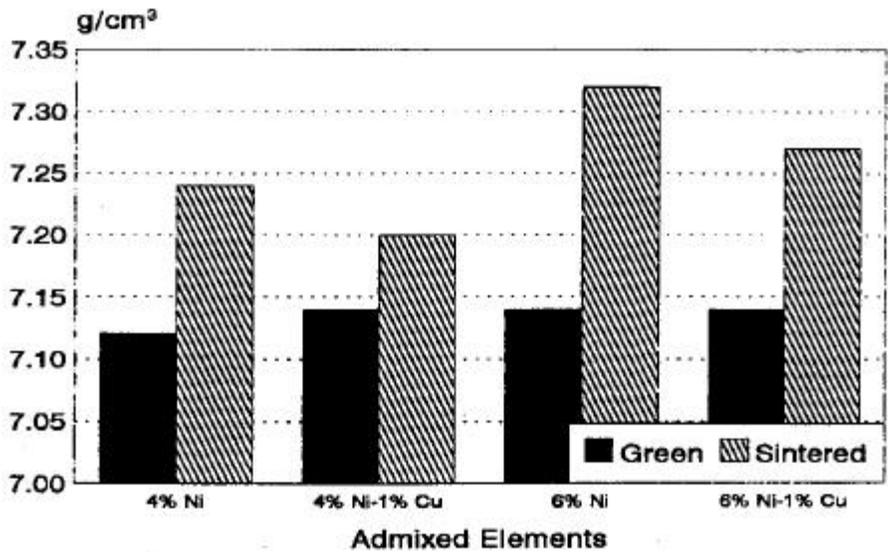
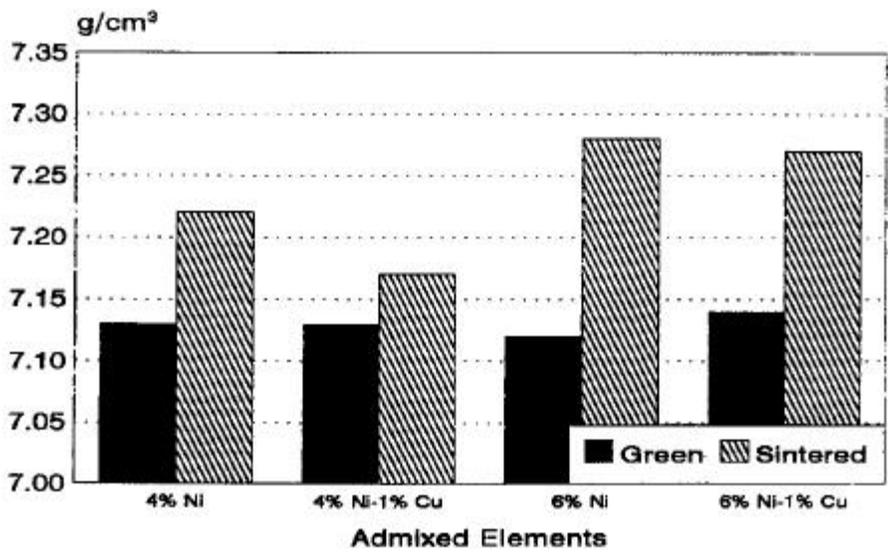


Figure 15 - Size Change Comparison using 40 ksi specimens.



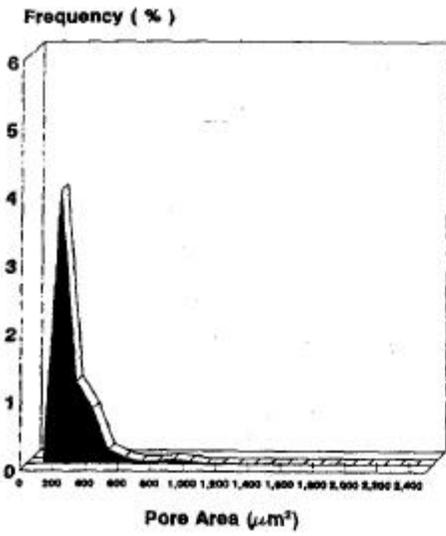
Specimens at 40 tsi Compaction Pressure

Figure 16A - Green to Sintered Density Comparison for 0.85% Mo Grade



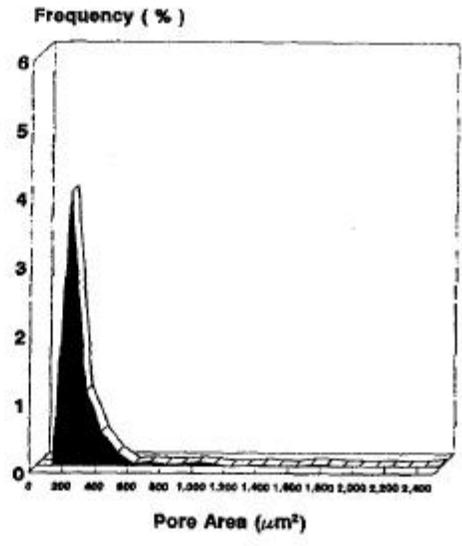
Specimens at 40 tsi Compaction Pressure

Figure 16B - Green to Sintered Density Comparison for 1.50% Mo Grade



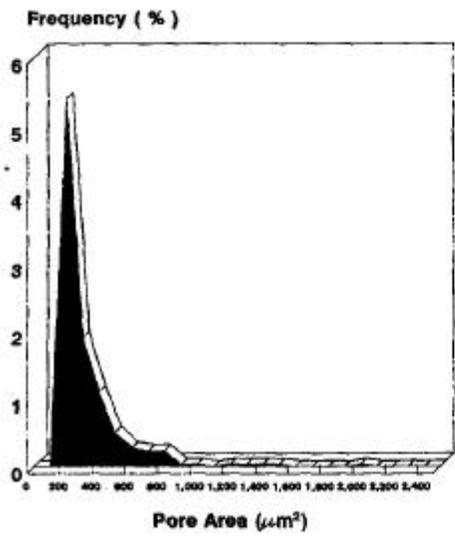
Found 93.5% less than 100 $\mu\text{m}^2$   
Total Pores - 3540

Figure 17A - 0.85% Mo + 6% Ni + 0.5% gr



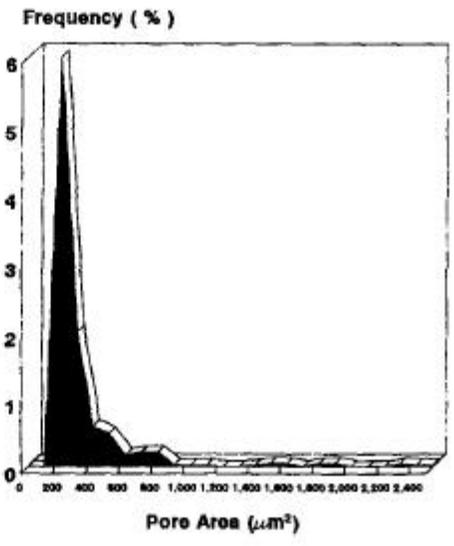
Found 93.7% less than 100 $\mu\text{m}^2$   
Total Pores - 3314

Figure 17B - 0.85% Mo + 6% Ni + 1% Cu + 0.5% gr



Found 90.2% less than 100 $\mu\text{m}^2$   
Total Pores - 2987

Figure 18A - 1.50% Mo + 4% Ni + 0.5% gr



Found 89.8% less than 100 $\mu\text{m}^2$   
Total Pores - 3121

Figure 18B - 1.50% Mo + 4% Ni + 1% Cu + 0.5% gr

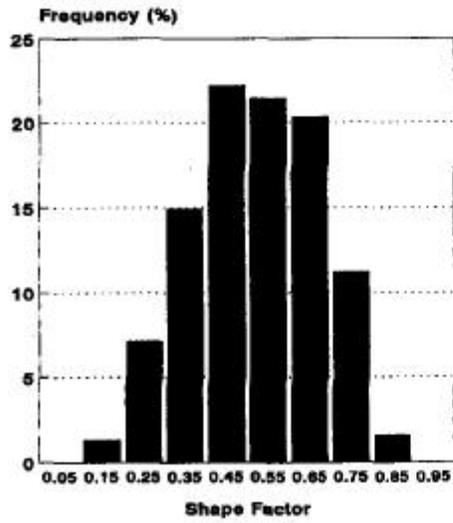


Figure 19A - Prealloyed 0.85% Mo + 6% Ni + 0.5% gr

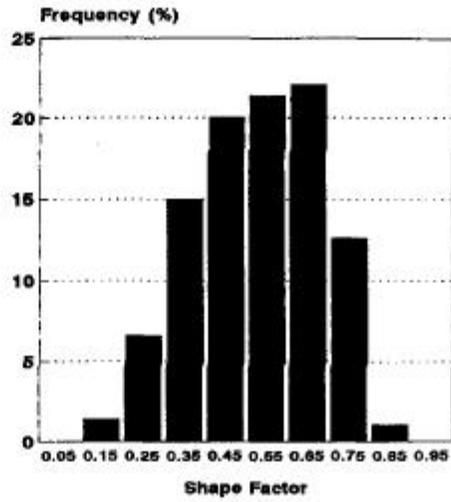


Figure 19B - Prealloyed 0.85% Mo + 6% Ni + 1% Cu + 0.5% gr

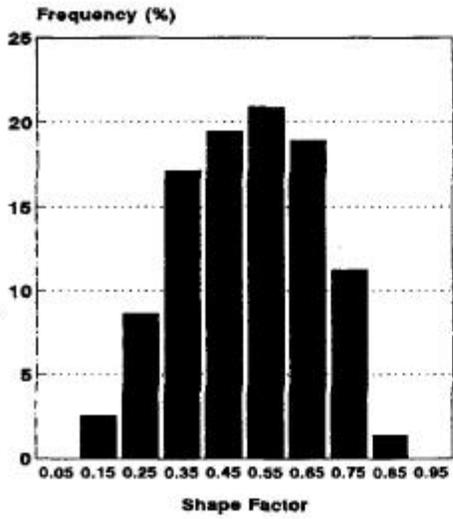


Figure 20A - Prealloyed 1.50% Mo + 4% Ni + 0.5% gr

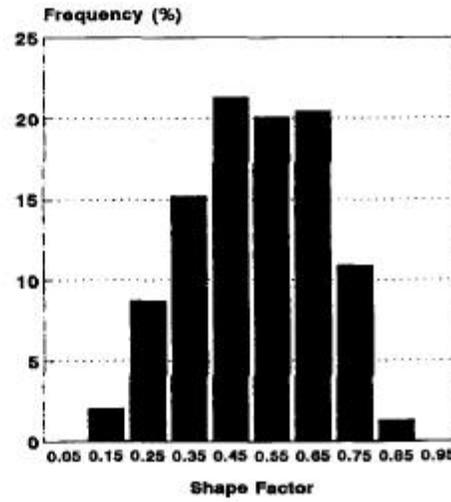


Figure 20B - Prealloyed 1.50% Mo + 4% Ni + 1% Cu + 0.5% gr