

**An Investigation into the Effects of Processing Methods on the
Mechanical Characteristics of High Performance Ferrous P/M
Materials**

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ABSTRACT

The mechanical properties of high performance ferrous P/M materials are influenced by the material composition and processing method. This paper investigates the effects of the ANCORDERNSE™ process, a new, high density, single compaction method, on the mechanical properties of Distaloy® 4800A based materials. The results of this study are discussed with a comparison to the mechanical properties for the same materials developed through single-pressed and double-pressed, double-sintered processing methods. In addition, a case study is performed on a component produced via the ANCORDERNSE method.

INTRODUCTION

Since many of the new applications for P/M require high strength and dynamic properties, either an engineered material or increased density approach is necessary to meet these requirements. Much effort has been devoted within the industry to develop and optimize processes and materials to meet these demanding applications. [1,2,3,4] It is well documented [5,6] that as the density is increased in a P/M component, there is a general enhancement in the mechanical properties. Until recently, the methods available for attaining high densities were either by high temperature sintering, double-press and double-sintering, and liquid phase sintering, each of which have inherent limitations. A new, single compaction method for attaining high densities utilizing the patented ANCORDERNSE process was recently introduced. This method has been shown to provide single compaction, single sintered densities in the 7.25 to 7.55 g/cm³ range without exceeding compaction pressures of 50 tsi (690 MPa) or sintering temperatures of 2300°F (1260°C). [7,8]

The ANCORDERNSE process utilizes a powder binder treatment which has shown good powder flowability and resistance to segregation and is integral with the processing method. Once the powder is

heated to the proper temperature, between 290°F (143°C) and 310°F (155°C), it is fed into a heated die. This requires that the components of the feed and tooling system are heated and/or insulated to provide uniform temperature within +/- 5°F (+/- 2.8°C). Compaction then occurs the same as in a typical P/M process.

This paper examines the effect processing method has on the mechanical properties. Evaluation of the new processing system coupled with double-pressed, double-sintered technology was performed to determine the potential density and property improvements that can be attained. The main focus of the work is with the ANCORDERNSE process and represents a continuing investigation into the optimization of this material system.

EXPERIMENTAL PROCEDURES

Materials

For the initial evaluation, small lab scale test premixes were produced using Distaloy 4800A, a 4%Ni-1.5%Cu-0.5%Mo diffusion alloyed iron powder, as the base material to produce two different compositions by varying the elemental Ni and graphite additions. These materials were produced by either the ANCORBOND® or the ANCORDERNSE method as listed in Table 1. A 500-pound (226 kg.) test premix was made via the ANCORDERNSE method for the second portion of the evaluation. The compositions were chosen for their high mechanical properties where one requires a carburizing heat treatment to achieve the properties and the other utilizes a sinter-hardening response. The conventional premixes had 0.75% Acrawax added as the pressing lubricant while the ANCORDERNSE premixes had 0.6% lubricant.

TABLE 1: Composition of the ANCORBOND processed powders evaluated.

Material Designation	Base Material (Wt. %)			Elemental Additions (Wt. %)			Powder Processing Method
	Ni	Cu	Mo	Ni	Graphite	Lubricant	
A	4	1.5	0.5	2.0	0.3	0.6	ANCORDERNSE
B	4	1.5	0.5	2.0	0.3	0.75	Conventional
C	4	1.5	0.5	4.0	0.8	0.6	ANCORDERNSE
D	4	1.5	0.5	4.0	0.8	0.75	Conventional

Test Specimen Processing

Processing of test specimens and parts was performed on conventional equipment with tooling modified to provide and

maintain the temperature at the +/- 5°F (+/- 2.8°C) required for effective performance of the ANCORDERSE materials. Before compaction of the test pieces, the tooling was allowed to reach steady state temperature conditions. The conventional powders were processed at ambient temperature while the ANCORDERSE powders were heated to the operating temperature with microwave equipment.

For the first portion of the study, compaction was performed between 30 and 50 tsi (415 and 690 MPa). After compaction, test specimens were full sintered at either 2050°F (1120°C) or at 2300°F (1260°C) for 30 minutes at temperature in a 25 v/o N₂ - 75 v/o H₂ atmosphere.

In the second segment of the study, all test shapes were compacted at 45 tsi (620 MPa), unless otherwise specified. For the double-press, double-sinter specimens, the presintering was done in a production 18" (46 cm) mesh belt type furnace in a N₂ based atmosphere with dissociated ammonia additions at 1500°F (815°C) for 20 minutes at temperature. The second compaction step was performed at 45 tsi (620 MPa) under ambient temperature conditions.

The test specimens were separated into two sinterings where one group was full sintered at 2050°F (1120°C) for 25 minutes at temperature in a 24" (61 cm) mesh belt furnace with a N₂ based atmosphere and dissociated ammonia addition. The other group was sintered at 2300°F (1260°C) for 30 minutes at temperature in a 25 v/o N₂ - 75 v/o H₂ atmosphere. For this portion of the experiment, the conventional double-pressed, double-sintered test specimens were sintered at only 2050°F (1120°C).

The carburizing heat treatment was performed in an integral quench furnace utilizing two separate cycles. The first cycle was performed at 1500°F (815°C) with an endothermic atmosphere providing a 0.7% carbon potential. The parts were oil quenched and tempered at 375°F (190°C). The second cycle was performed at 1600°F (870°C) with an endothermic atmosphere at a 0.8% carbon potential. The parts were oil quenched and tempered at 300°F (150°C).

Testing

In all test cases, between 5 and 10 test specimens were evaluated for each test condition. The test specimens were processed and evaluated according to industry standard test procedures. [10] for green strength, green density, sintered and heat treated TRS, heat treated Charpy impact and sintered and heat treated tensile

properties. Tensile properties were developed from ASTM E8 flat, unmachined "dogbone" tensile bars with a 1" gage length.

TRS and tensile testing was performed at a crosshead speed of 0.1 in./min (2.5 mm/mm). A Rockwell Hardness Tester was used for apparent hardness measurements in either the Rockwell C scale or Rockwell B scale. Ejection force was determined on standard TRS specimens. Figure 1 shows the experimental procedure flow chart.

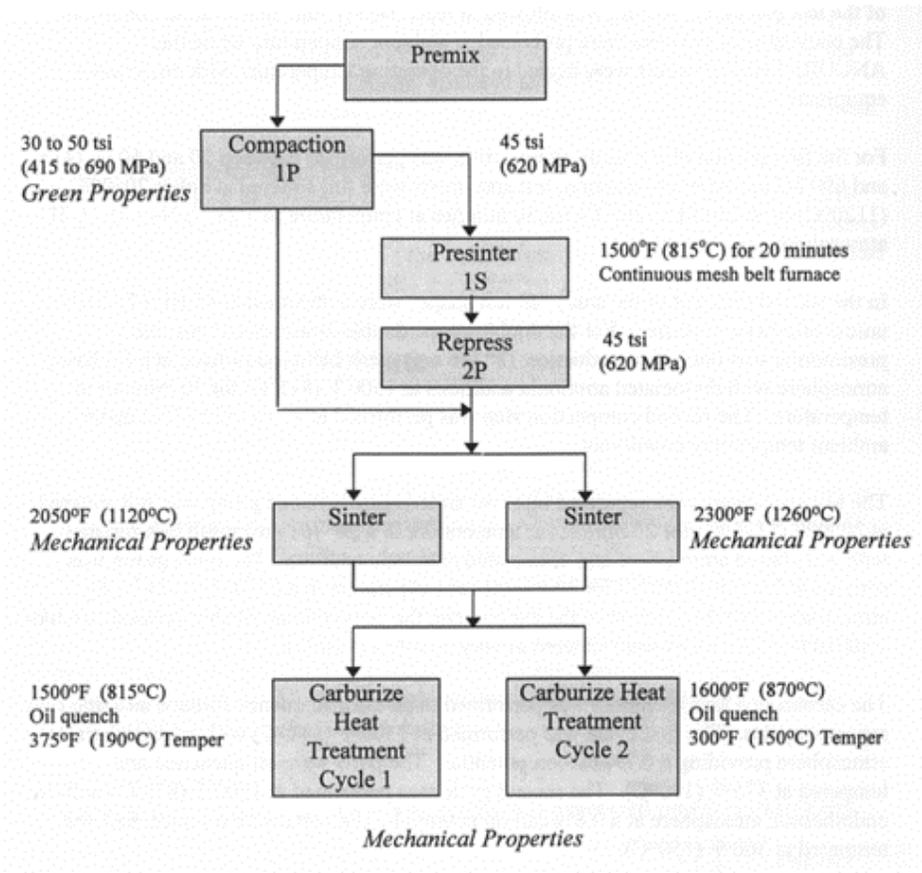


FIGURE 1: Experimental program flow chart.

Metallography

Sections for metallographic examination were cut from TRS test pieces unless otherwise noted. Optical metallography was performed on a Nikon Epiphot. All scanning electron microscopy (SEM) was performed on a JEOL JSM-840 with Kevex system for energy dispersive x-ray analysis.

RESULTS AND DISCUSSION

Compaction (Green Properties)

Compressibility of the premixes was measured between 30 and 50 tsi (415 to 690 Mpa). For the ANCORDENSE processed materials, a 0.17 to 0.25 g/cm³ increase in green density was seen over the conventionally processed premix for the complete range of pressures. The compressibility curves for these four materials are shown in Figure 2.

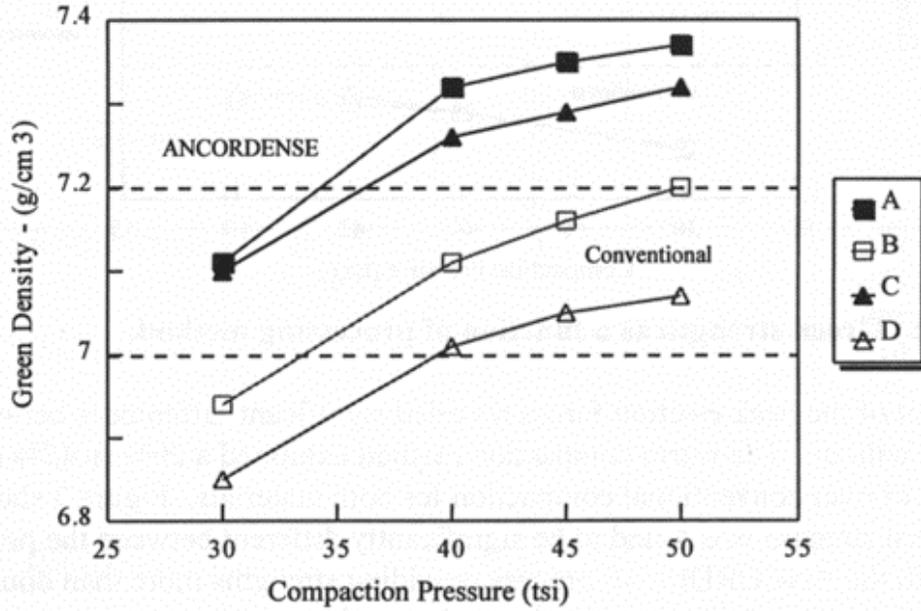


FIGURE 2: Compressibility of the four premix powders.

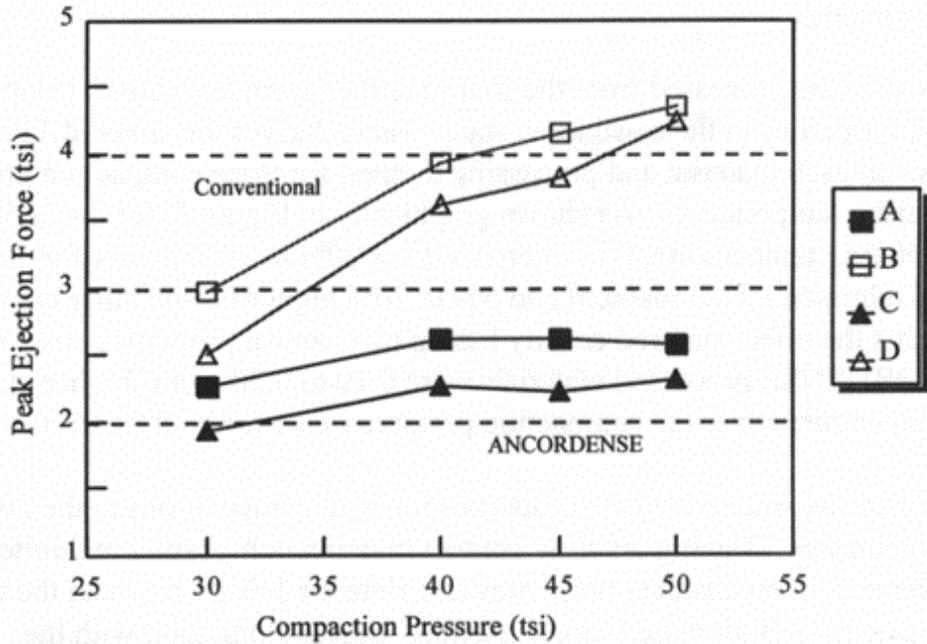


FIGURE 3: Peak ejection force as a function of compaction method.

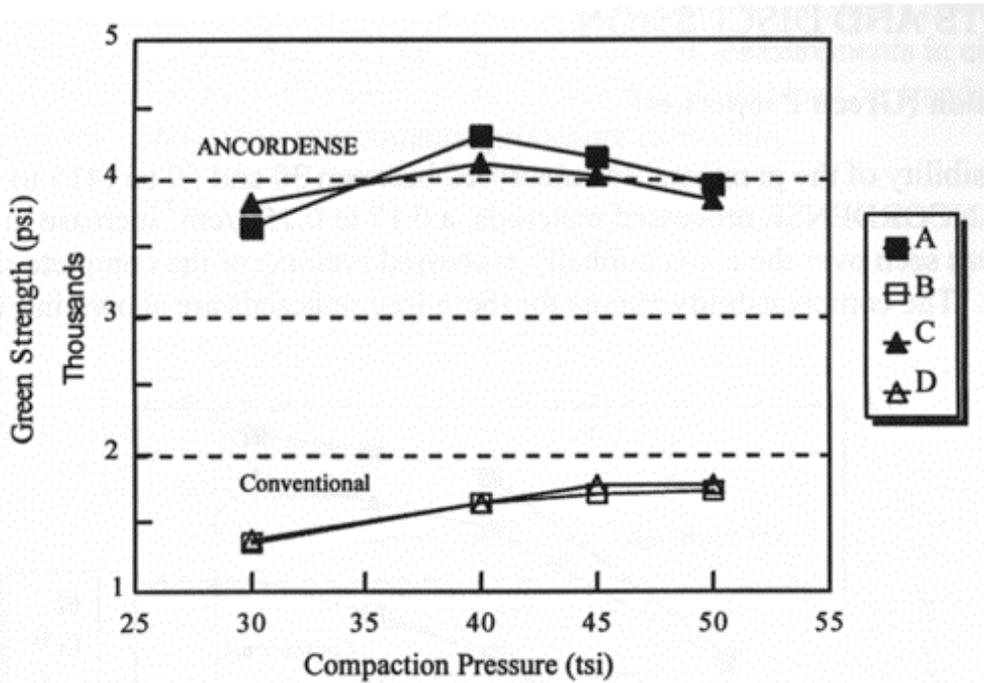


FIGURE 4: Green strength as a function of processing method.

Measurement of the peak ejection forces revealed significant differences between the two processing methods. The warm

compaction method exhibited a 20% to 45% reduction in ejection forces over conventional compaction for both materials. Figure 3 shows these results. Green strength was found to be significantly different between the processing methods with the ANCORDERNSE process providing strengths more than double that of the binder treated materials. Figure 4 shows the results.

Sintered Properties

TRS and tensile bars processed from the four materials were tested to develop mechanical and physical properties in the as-sintered state. Table 2 gives the sintered TRS, density and hardness on each material and processing method for three compaction pressures at the two sintering temperatures. As shown graphically in Figure 5, for the 2050°F (1120°C) sintering temperature, the sintered TRS is 20% to 30% higher for the increased graphite and admixed nickel material and 5% to 10% higher for the other composition. This highlights the effect sintered density has on mechanical properties since the density of the ANCORDERNSE processed materials were 0.10 to 0.20 g/cm³ higher at both sintering temperatures over the compaction pressure range for both materials.

Table 3 gives the as-sintered tensile properties for both compositions at the 2050°F sintering temperature. The higher alloy content material composition exhibited the greatest difference in mechanical properties and sintered density between the two processing methods. Both materials experienced greater elongation with the ANCORDERNSE processing which indicates the strong dependence elongation has on density for a given material composition. Additionally, it was noted that the elongation was higher at the same density for the ANCORDERNSE process than the conventional.

TABLE 2: Comparison of TRS properties for the two processing methods.

Material	Processing Technique	Sintering Temperature	Compaction Pressure (tsi)	Sintered Density (g/cm ³)	TRS (10 ³ psi)	Hardness (HRB)
A	ANCORDERNSE	2050°F (1120° C)	30	7.10	196.5	86
			40	7.31	227.8	95
			50	7.39	234.3	96
B	Conventional		30	6.99	186.6	86
			40	7.17	205.6	90
			50	7.27	224.3	92
C	ANCORDERNSE		30	7.16	211.1	91
			40	7.32	230.4	95
			50	7.38	252.6	95

D	Conventional		30	6.95	162.0	89
			40	7.13	190.2	91
			50	7.21	196.8	94
A	ANCORDENSE	2300°F (1260°C)	30	7.15	224.1	92
			40	7.35	246.1	18 HRC
			50	7.43	268.1	21 HRC
B	Conventional	(1260°C)	30	7.07	210.2	91
			40	7.24	247.2	95
			50	7.33	263.4	21 HRC
C	ANCORDENSE		30	7.25	240.9	27 HRC
			40	7.39	279.7	29 HRC
			50	7.46	297.2	30 HRC
D	Conventional		30	7.10	215.9	23 HRC
			40	7.27	247.9	29 HRC
			50	7.35	269.1	30 HRC

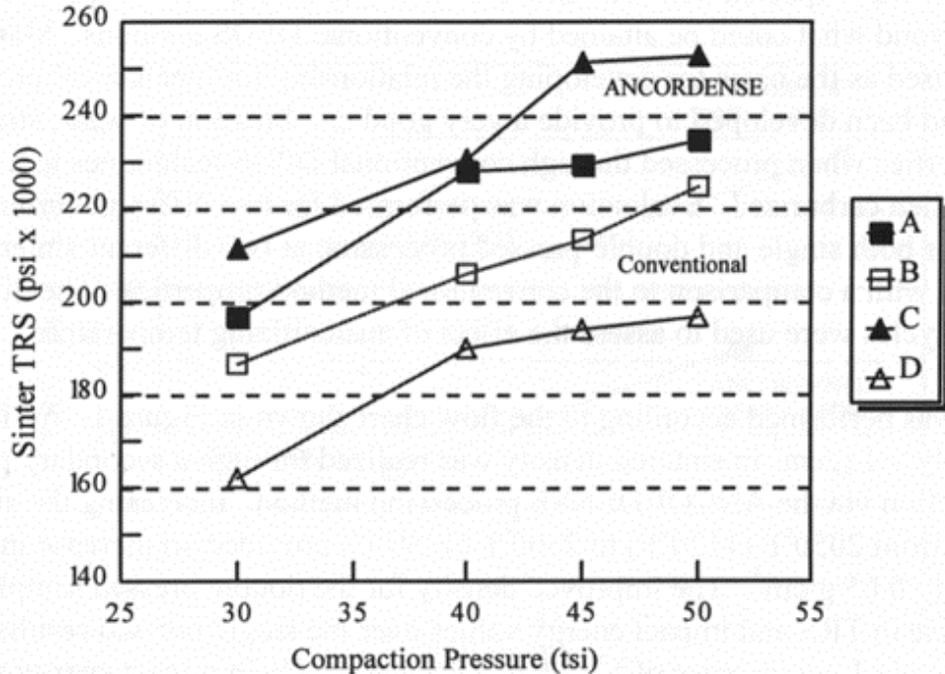


FIGURE 5: Transverse rupture strength as a function processing method (sintered at 2050° F for 30 minutes in 75% H₂/ 25% N₂)

TABLE 3: Sintered tensile properties for the two methods at 2050°F (1120°F).

Material	Processing Technique	Compaction Pressure (tsi)	Sintered Density (g/cm ³)	UTS (10 ³ psi)	0.2% YS (10 ³ psi)	Elong. (%)
A	ANCORDENSE	30	7.22	91.2	54.6	3.3
		40	7.40	103.7	60.3	4.2
		50	7.47	110.7	65.9	4.8
B	Conventional	30	7.11	93.4	61.1	2.7

	1	40	7.23	102.1	65.0	3.0
		50	7.38	111.7	72.3	3.3
C	ANCORDENSE	30	7.21	104.3	52.8	3.1
		40	7.37	122.5	55.0	3.4
		50	7.42	125.5	58.2	3.4
D	Conventional	30	7.02	85.1	46.2	2.1
	1	40	7.18	100.7	50.3	2.6
		50	7.27	108.8	55.0	2.8

The as-sintered carbon contents for each of the materials and sintering conditions were found to vary no more than +/- 0.01% between each of the processing methods for the same material composition. The as-sintered carbon was measured at 0.28% for materials A and B. Materials C and D measured at 0.72%.

Double-Press, Double Sinter (DPDS) Properties

An important aspect of the study was to determine the response of this material system processed via the ANCORDENSE method coupled with double-press, double-sinter techniques. It was expected that densities, and therefore mechanical properties, could be increased beyond what could be attained by conventional DPDS methods. Materials A and B were used as the basis for developing the relationship for mechanical properties because it had been developed to provide a very good combination of wear, strength and impact properties when processed through conventional DPDS techniques to a 7.35 g/cm³ density and then carburized. Evaluation was performed for two different carburizing heat treatments for both single and double-pressed processing at two different sintering temperatures with a comparison to the conventional method properties. The different carburizing cycles were used to assess the effect of austenitizing temperature.

Processing was performed according to the flow chart shown in Figure 1. An increase of approximately 0.1 g/cm³ in sintered density was realized through a secondary pressing after compaction via the ANCORDENSE processing method. Increasing the sintering temperature from 2050°F (1120°C) to 2300°F (1260°C) provided an increase in density of approximately 0.05 g/cm³. The improved density for the double pressed samples provided a rise in TRS and impact energy values over the single pressed results. For carburizing cycle 1, an increase of 12.5% in TRS and 49.4% in impact energy was seen at 2050°F (1120°C) between the SP and DP processes. At 2300°F (1260°C), the TRS and impact energy increased 13.3% and 81.3%, respectively. For cycle 2, an increase of 13.2% in TRS and 31.2% in impact energy was seen at 2050°F (1120°C) and at 2300°F (1260°C) an increase of 8.7% in TRS and 106.7% in impact energy was found. The impact energy of the DP

2300°F (1260°C) sintered samples averaged 41.7 ft-lbf(56.5 J) for cycle 1 and 39.5 ft-lbf(53.6 J) for cycle 2. Some improvements in TRS were seen with an increase in austenitizing temperature, while impact energy was improved slightly with the lower austenitizing temperature. Figures 6 through 9 show the results.

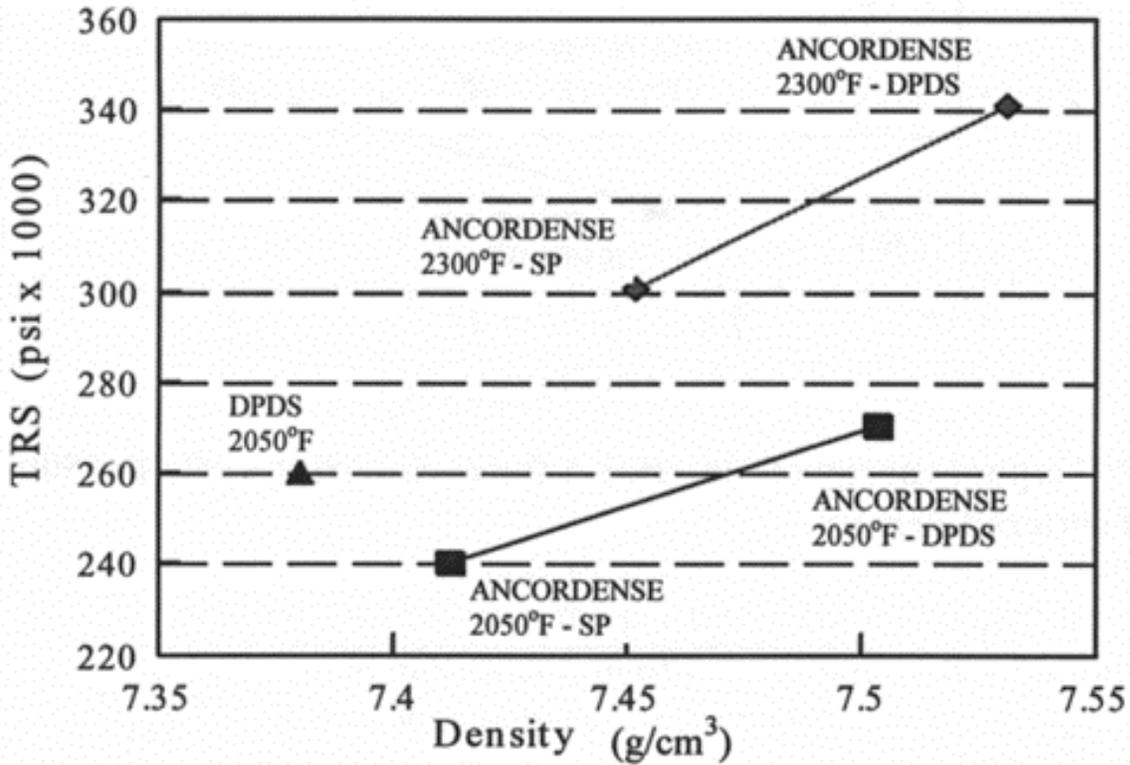


FIGURE 6: Transverse rupture strength of material A for carburizing cycle 1 (1500°F) as a function of sintered density.

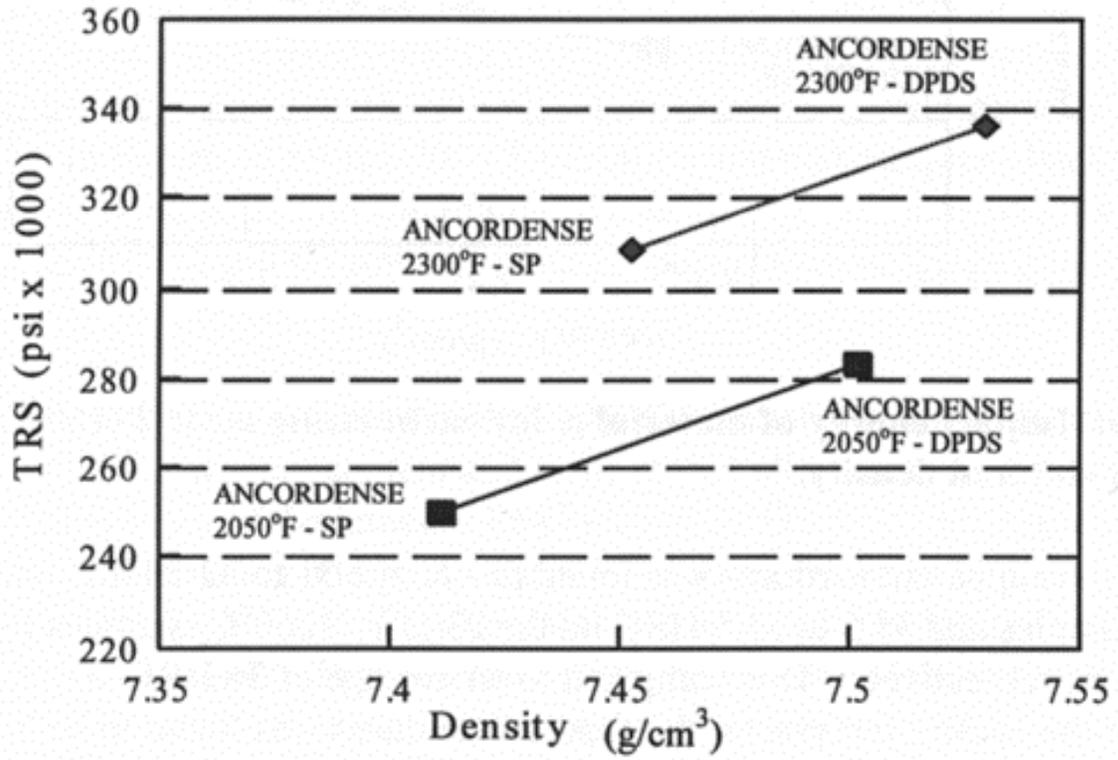


FIGURE 7: Transverse rupture strength of material A for carburizing cycle 2

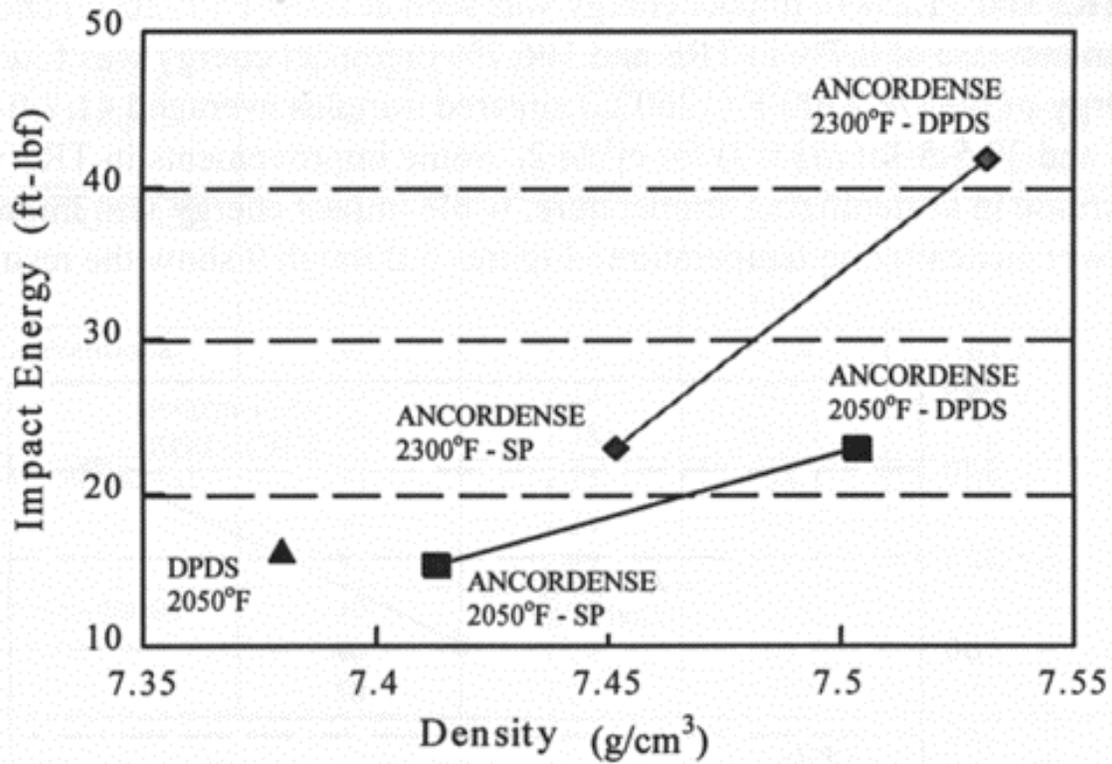


FIGURE 8: Impact energy of material A for carburizing cycle 1 (1500°F) as a function of sintered density.

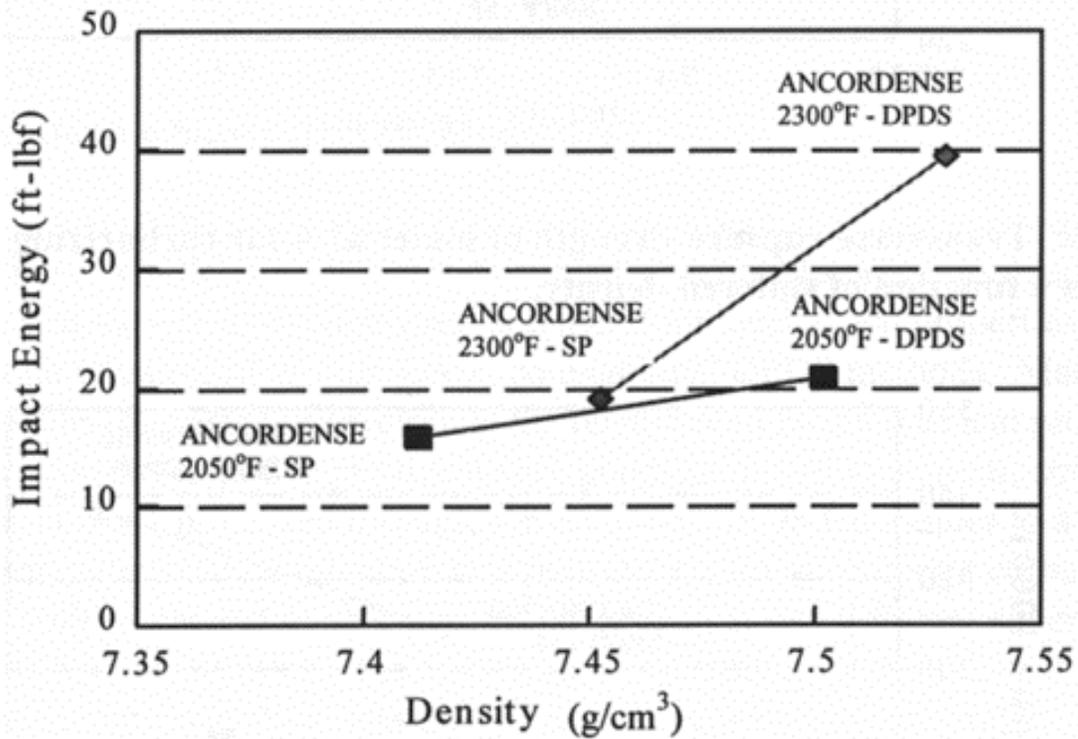


FIGURE 9: Impact energy of material A for carburizing cycle 2 (1600°F) as a function of sintered density.

For cycle 1, the apparent hardness was found to average 31 to 32.5 HRC on the 2050°F (1120°C) samples and 35.5 to 33.5 HRC on the 2300°F (1260°C) samples for the SP and DP processes, respectively. This compared to an average of 36 HRC on the conventional DPDS. For the cycle 2 test pieces, the apparent hardness was found to be higher at 39.5 to 40.5 HRC on the 2050°F (1120°C) samples and 41.5 to 37 HRC on the 2300°F (1260°C) samples for the SP and DP processes, respectively, due to the increased carburization with the higher austenitizing temperature. The particle hardness in the case regions reflected the basic trend found with the apparent hardness.

Material Characterization

Initially, the powders were examined using the SEM. The particles were irregular in shape and exhibited the typical low level of fine particles of the ANCORBOND treatment as compared to conventional premixes. Uniformly distributed diffusion bonded particles were present across the base iron particle surfaces. The effects of the powder binder treatment were evident as small particles (< 10µm) uniformly dispersed and adhered to the base particles. Backscattered electron imaging (BEI) was coupled with secondary electron imaging (SEI) to distinguish between the different elements. Energy x-ray dispersive analysis (qualitative) was performed to verify the elements present.

After compaction, the green strength samples were analyzed on the fracture surface and polished cross section. Examination of the fracture surface revealed mechanical interlocking as the predominant strengthening method. The increased densification achieved with the ANCORDENSE process provided a significant increase in interlocking between particles than the conventional compaction method. No intergranular fracture was evident with the fracture path exhibiting non-linear behavior (irregular fracture path).

The polished cross section revealed that particle deformation occurred during the compaction process with close contact between the particle surface apparent throughout. This indicates that significant particle movement, plastic deformation and rearrangement transpired. This was unlike typical contact between surfaces seen with conventional compaction which generally occur at points of asperity. It should be noted that the powder mass approaches maximum theoretical pore free density (> 97%)

characterizing the particle packing as being very efficient during the ANCORDEENSE compaction process.

Analysis of the as-sintered samples revealed a micro structure consisting of pearlite, ferrite, martensite, bainite and nickel-rich regions. The high nickel regions were prevalent around porosity indicating that the nickel diffusion was predominantly surface diffusion as shown in Figure 12 for the 2050°F (1120°C) sintered samples. Nickel diffusion along grain boundaries and smoothing or rounding of the pores was also apparent. For the 2300°F (1260°C) sintered samples, pore rounding and degree of diffusion was more pronounced.

A substantial volume of ferrite was present throughout the micro structure. Pearlite was found in regions of very low alloy content and some higher alloy areas as evidenced by the morphology and spacing of the lamellar Fe₃C platelets. The low alloy regions formed colonies of pearlite in alternating and relatively parallel strips (plates) of Fe₃C and proeutectoid ferrite. The higher alloy regions exhibited divorced, randomly oriented Fe₃C platelets in a ferrite matrix. This pearlite was in the vicinity of the nickel rich areas with some located in the nickel diffusion region of the iron particles with lamellar pearlite in the core of the particles. This showed the influence of local concentration of alloy elements in solid solution in the austenite on the nucleation and growth of the pearlite.

Cross sections of the carburized samples revealed microstructures consisting of tempered, high carbon martensite and nickel-rich regions in the case with a gradual transformation to mixed martensite, bainite, pearlite, ferrite and nickel-rich regions in the core. The composition gradients were verified by EDS, showing little change from the sintered state. Fracture analysis of the tensile and TRS bars was performed, with the core and case regions compared. Figure 13 shows a case region in the SP 2050°F (1120°C) sintered bar.

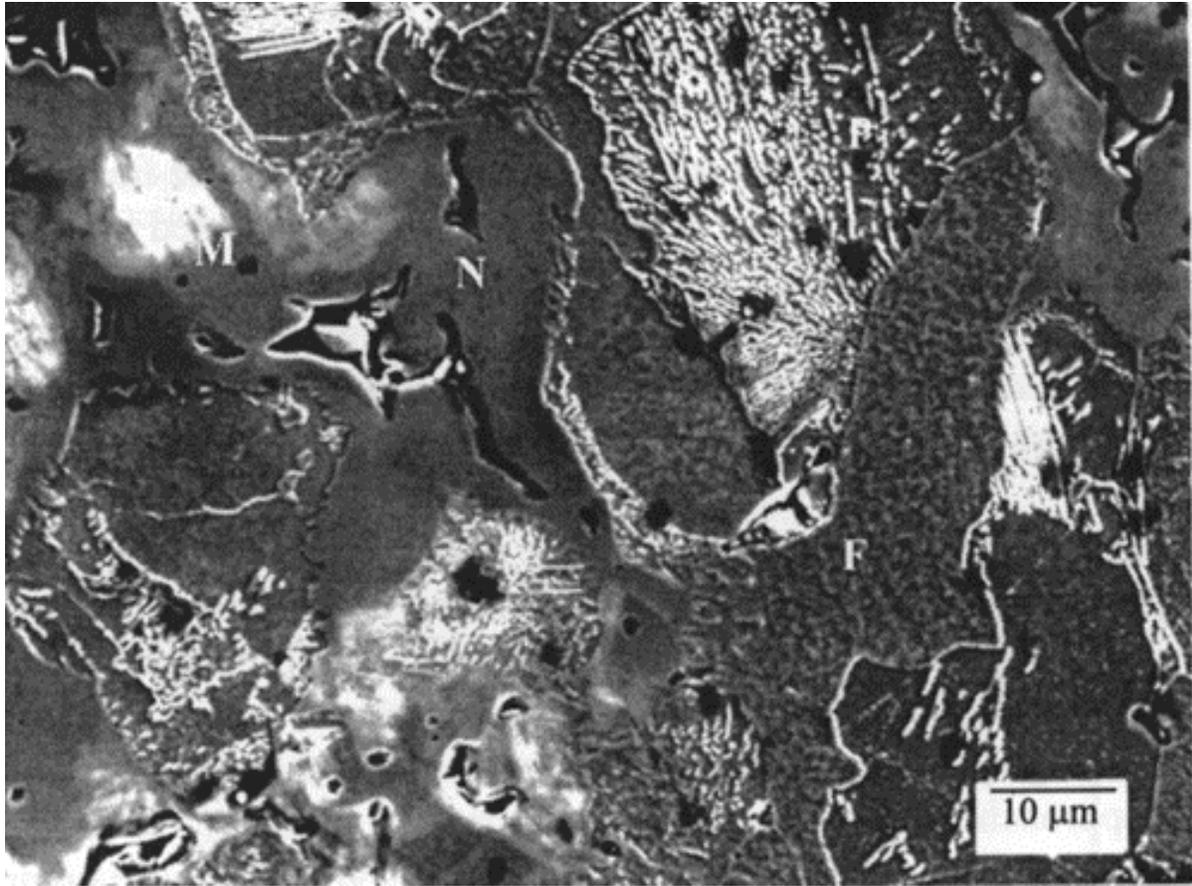


FIGURE 12: (SEI) Sinter microstructure of material A single pressed and sintered at 2050°F (1120°C). (N: nickel-rich, P: pearlite, F: ferrite, M: martensite).

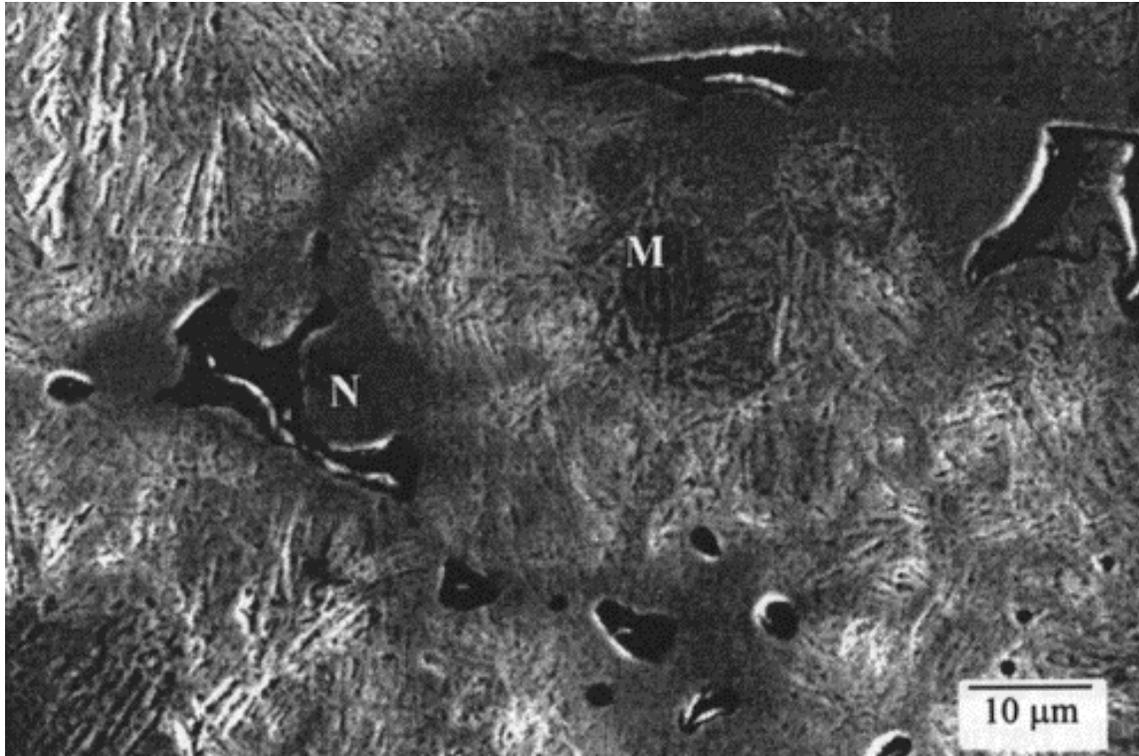
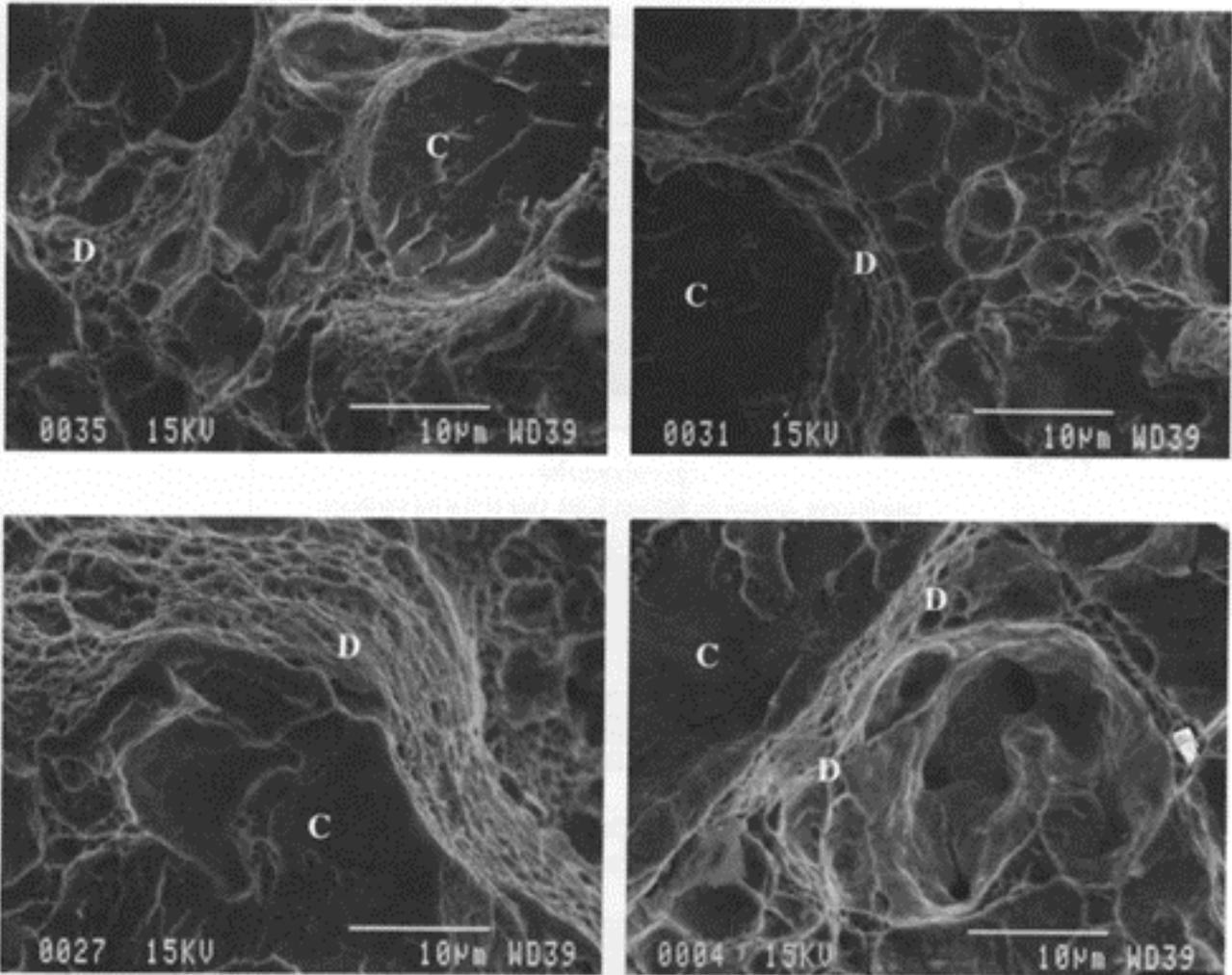


FIGURE 13: (SEI) Carburized microstructure of material A single pressed and sintered at 2050°F (1120° C) in the case region. (N: nickel-rich, M: martensite).

SEM analysis of the TRS fracture surfaces revealed mixed mode (both transgranular and ductile rupture) fracture in the case region and only ductile rupture in the core for all samples. The DP samples exhibited a greater amount of transgranular fracture in the case with the 2300°F (1260°C) sintered parts having the highest level. In addition, both DP samples had larger ruptured bond area fractions than the SP samples. The ductile rupture regions in the case areas, which were at prior particle bonds, indicate that the surface diffusion of nickel provided toughening and ductility to the interparticle bonds. The significant increase in bond area fraction for the double pressed parts provided the improvement seen in mechanical properties. Figure 14 shows the shear fracture surfaces from the TRS bars for the 1600°F (870°C) carburizing heat treatment.



Direction of shear load ----->

FIGURE 14: SEM fractographs of TRS bars carburized via cycle 2 in the case region. SP on the top, DP on the bottom with the 2050°F sinter on the left and the 2300°F on the right. Shows transgranular cleavage (C) and ductile rupture (D).

Production Parts Processing

A component was produced via the single pressed ANCORDENSE processing method using material A to a density of 7.4 g/cm^3 . The part was single level with complex O D and I.D. (core rod) shapes. Production equipment was utilized after modification to provide the necessary temperature control. Compacts were pressed at about 50 tsi (690 MPa) to two different thicknesses, 0.278 in (7.06 mm) and 0.678 in (17.22 mm). The surface area of the part was 1.789 in^2 (11.54cm^2). After sintering at 2050°F (1120°C),

the parts were then carburized and tempered as described for cycle 2.

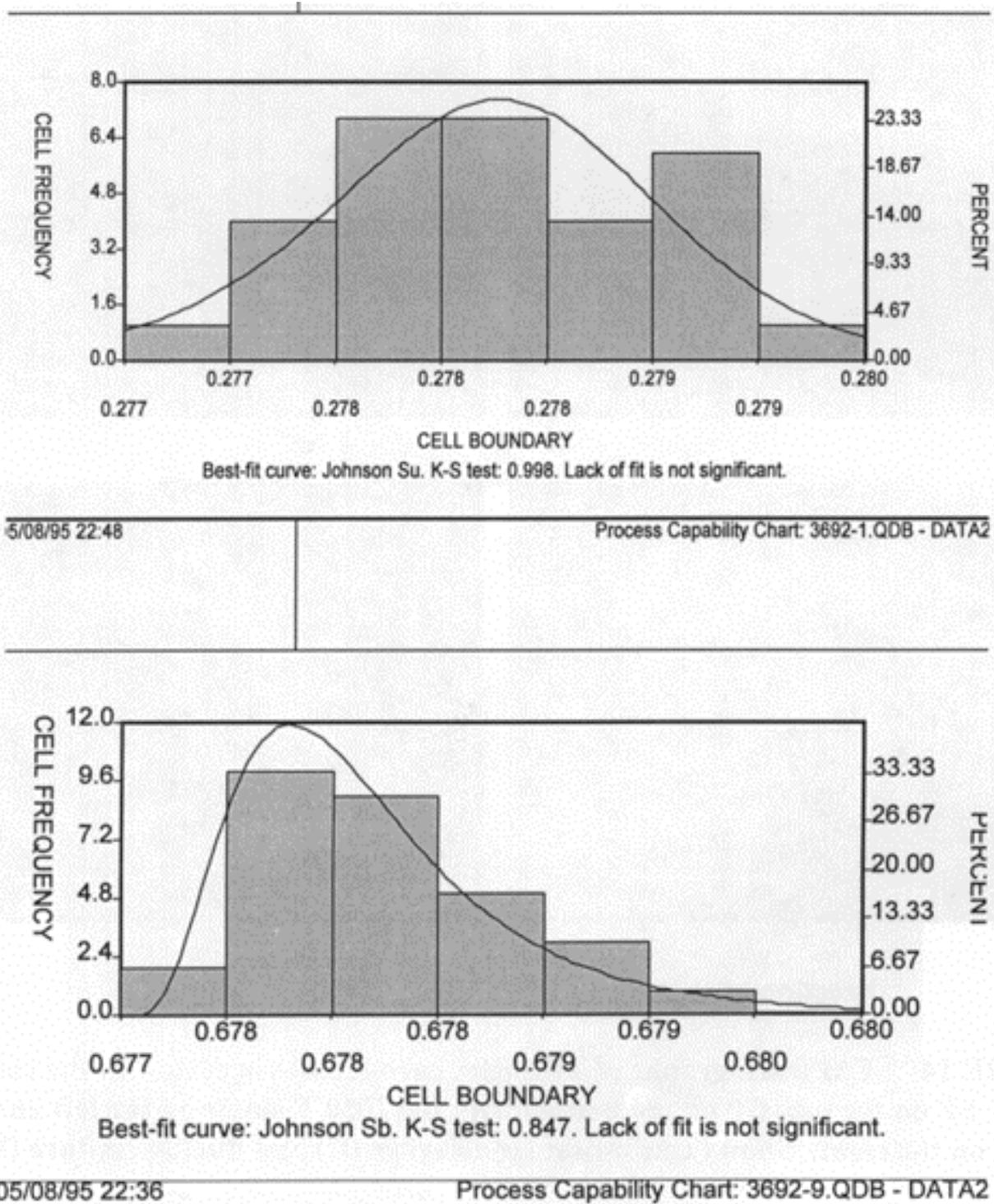
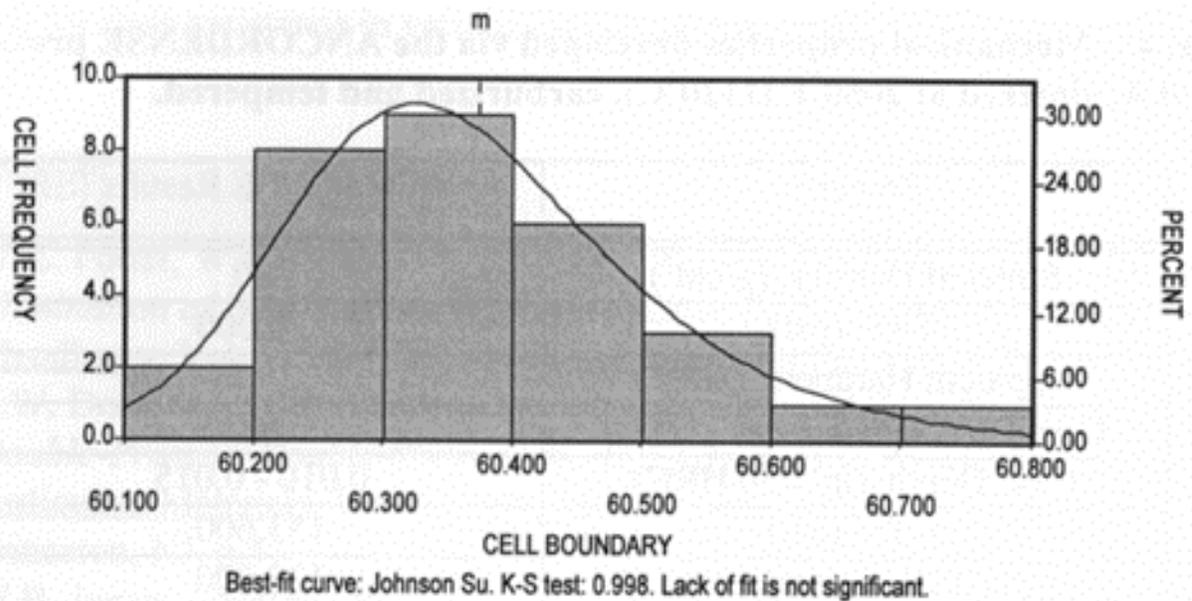
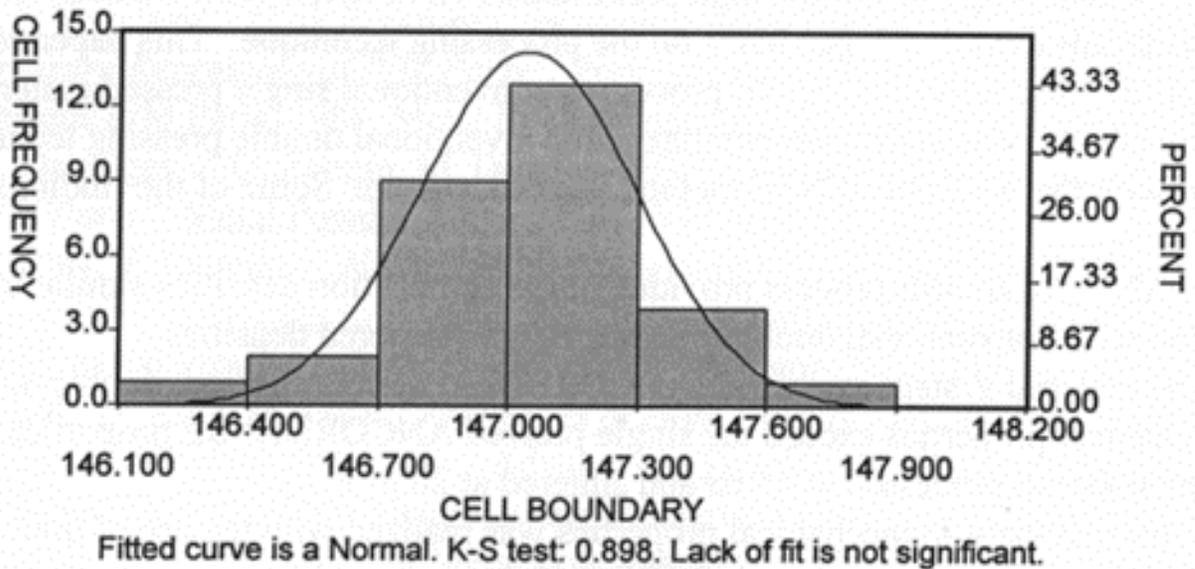


FIGURE 15: Green thickness process capability analysis for the production part.



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Process Capability Chart: 3692-1.QDB - DATA1



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Process Capability Chart: 3692-9.QDB - DATA1

FIGURE 16: Green weight process capability analysis for the two thicknesses of the production part (0.278 in thickness part weight at top).

The press tooling was stabilized at the operating temperature, then run to bring the weight and thickness into target. Once the controls were met, the press was run on automatic to develop capability of the process for the two thicknesses. The histograms shown in figures 15 and 16 represent the thickness and weight capability for the two press thicknesses. Both showed acceptable deviation and distribution in the measured characteristics with the weight varying 1.45 grams for the thicker parts and 0.57 grams for the thinner parts.

The parts were then sintered and carburized. TRS and tensile bars were processed to the same density and processed with the production parts. The mechanical properties developed are listed in Table 4.

TABLE 4: Mechanical properties developed via the ANCORDERSE process with material A, sintered at 2050°F (1120°C), carburized and tempered.

Property	ANCORDERSE Results (SP)
Sintered Density (g/cm ³)	7.41
TRS (psi)	276,000
Apparent Hardness (HRC)	36.2
Case Particle Hardness (HRC)	62
Case Depth (in.>50HRC)	0.010 - 0.015
UTS (psi)	153,600
0.2% Yield Strength (psi)	128,000
Elongation (%)	1.3

Conclusions

The mechanical properties of the high performance ANCORDERSE Distaloy 4800A based materials exhibit a dependence on the processing technique. This paper compared the single pressed ANCORDERSE process to conventional single pressed materials. Also, the effects of sintering temperature and conventional double pressing techniques coupled with the ANCORDERSE method were examined. Some of the findings include

1. The ANCORDERSE process provides single compaction densities similar to those found with conventional double-pressed, double-sintered densities.
2. In the material system examined, ANCORBOND double-pressed, double-sintered mechanical properties exceed the single pressed

ANCORDENSE properties for the same material processed at 45 tsi and sintered at 2050°F.

3. An improvement in mechanical properties was realized with the use of double-press techniques coupled with the ANCORDENSE process, with a greater improvement seen with the 2300°F (1260°C) sinter for either process.

4. Green strength was improved significantly (over 100%) over conventional processing.

5. For a conventional carburizing cycle, the impact energy was improved by 31% for 2050°F (1120°C) sintering and 106% for 2300°F (1260°C) sintering with double-pressing the ANCORDENSE material A as compared to the single pressed ANCORDENSE material.

6. Densities in excess of 7.5 g/cm³ are attainable with conventional sintering temperatures when double-press techniques are coupled with the warm compaction process.

7. Commercial parts production is viable with acceptable weight and thickness tolerances achievable with adequate temperature control.

ACKNOWLEDGMENTS

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