

**PROPERTIES OF DIFFUSION BONDED ALLOYS
PROCESSED TO HIGH DENSITIES**

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ABSTRACT

Diffusion bonded alloys have been shown to exhibit excellent properties. Recent advancements in compaction technology have allowed these materials to reach green density levels of over 7.3 g/cm³ in a single compaction process. Various capabilities of this new compaction system will be demonstrated utilizing diffusion bonded alloys. Properties of several diffusion bonded materials will be investigated at these high density levels. A variety of processing techniques will be utilized including high temperature sintering and heat treatment to demonstrate the flexibility of these materials.

INTRODUCTION

Considerable growth in the ferrous powder metallurgy business has been the result of several technological breakthroughs. These initiatives have allowed the manufacturers and users of powder metallurgy parts to expect higher density and premium performance from cost effective alloys and part manufacturing processes. Representative of these technologies from a powder producers perspective are diffusion alloyed powder (Distaloy) and the warm compaction system (ANCORSENSE™*)(1,2).

Diffusion Alloyed Powders

The diffusion alloying process represents a unique method of producing ferrous powders alloyed with various combinations of nickel, copper and/or molybdenum without sacrificing compressibility as might be expected from completely prealloyed materials. Diffusion alloyed materials are manufactured by starting with either a high purity iron powder or a highly compressible prealloyed powder (iron with molybdenum for example) mixed with the desired alloying elements. The alloying elements are then diffused into the surface of the powder during a low

temperature thermal treatment. Since the alloying elements are diffusion bonded onto the surface of the iron powder, the bulk of the material remains generally unalloyed and the powder remains highly compressible. Additionally, the possibility of segregation or dusting of fine alloying additions is eliminated providing a consistent alloy content throughout the part and from part to part.

Sintering of a diffusion alloyed material also provides for a unique microstructure resulting in distinctive physical properties. Typically, when compared to prealloyed or premixed powders of the same composition, the diffusion alloyed powders enjoy an advantage in certain physical properties such as impact energy and fatigue performance.

Material and Compaction System

A patented compaction and material technology, known as ANCORDENSE, has recently been introduced. The system utilizes heated tooling and heated powder to achieve improved green density, higher green strength and improved ejection characteristics. During the compaction process, diagrammed in Figure 1, both the tooling and the powder are heated to about 290°F (140°C). The press ready powder is a derivative of the ANCORBOND®** (3) system utilizing a lubricant/binder system optimized to work at this temperature. The powder can be heated by a variety of heating methods including screw heating (4), microwave heating (5) or a slotted heat exchanger (6). Beyond the application of this relatively low temperature, normal compaction procedures are used. The process has been shown to be applicable to all high performance base materials. A detailed description of how the system operates is available in other papers (7,8,9).

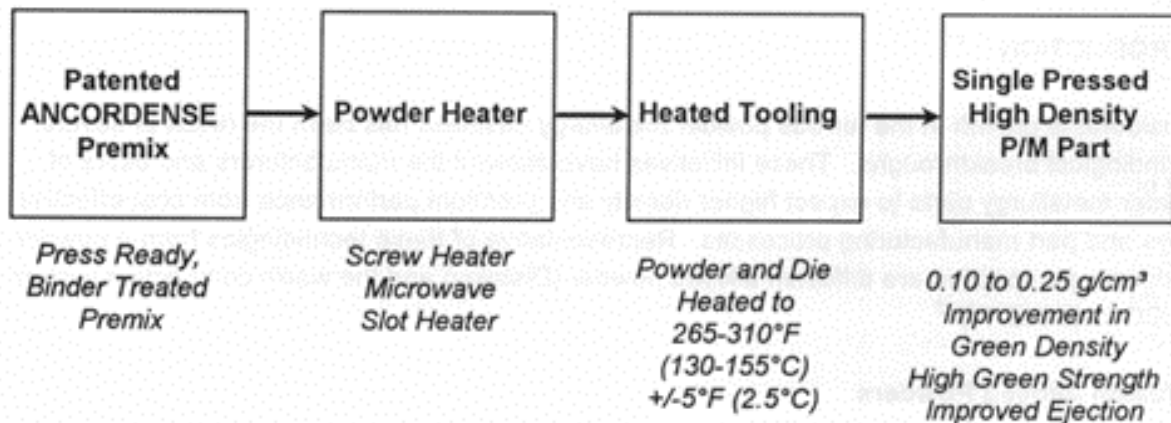


Figure 1: The ANCORDENSE Process

MATERIAL PROPERTIES

Materials

In order to evaluate the advantages of warm processing in combination with diffusion alloyed powders, premixes were prepared with two diffusion alloyed powders according to the chemistries listed in Table I. Both premixes were prepared utilizing the lubricant and binder system required for the warm compaction process.

Table I: Composition of Tested Premixes

Material	Base Iron Grade	Base Material Diffusion Alloyed Components- Nominal Composition			Admixed Components	
		Ni (w/o)	Cu (w/o)	Mo (w/o)	Graphite (w/o)	Lubricant (w/o)
A	Distaloy 4800A/ Distaloy AE	4.00	1.5	0.5	0.5	0.6
B	Distaloy 4600A/ Distaloy AB	1.75	1.5	0.5	0.5	0.6

Preparation and Testing Procedures

The apparent density and flow of the powder were measured under MPIF Standards 03 and 04(10), respectively, with several important modifications. In order to better understand the powder behavior at the desired temperature, both the Hall funnel and the receiving cup were heated to 290°F (145°C) prior to testing. An appropriate amount of powder was weighed and also heated to 290°F (145°C) just prior to testing. The hot powder was then poured into the heated apparatus in order to test the powder at a temperature typical of the warm compaction process.

A variety of test specimens were compacted on a 100 ton hydraulic compaction press utilizing a tool temperature and powder temperature of 290°F (145°C). The die material for all specimens was Crucible CPM 9V. Both the die and powder temperatures were controlled to +/- 5F° (+/- 2.5C°). In all cases, the results of five test specimens were averaged to obtain the reported results. Specimens were tested to evaluate green density according to MPIF Standard 45(10), green strength according to MPIF Standard 15(10), green expansion from die and ejection forces from a nominal 1.25 inches (4.92 mm) by 0.5 inches (12.7 mm) bar compacted to a height of 0.5 inches (12.7 mm).

Sintered properties were obtained by sintering specimens at either 2050°F (1120°C) or 2300°F (1260°C) in an atmosphere of 25v/o N₂ and 75v/o H₂ for 30 minutes at temperature. In all cases sintered carbon values of between 0.48 and 0.50 w/o were maintained. Where appropriate, heat treatment was performed by austenitizing at 1600°F (870°C) for thirty minutes in an endothermic atmosphere with a carbon potential of 0.55%, quenching into 165°F (75°C) oil and tempering at 375°F (190°C) for two hours in air. Sintered density, dimensional change from die according to MPIF Standard 44(10), transverse rupture strength according to MPIF Standard 41(10), and sintered hardness were measured from specimens nominally 1.25 inches (4.92 mm) by 0.5 inches (12.7 mm) compacted to a height of 0.25 inches (6.35 mm). Impact properties were generated utilizing a test specimen of 2.165 inches (55 mm) by 0.394 inches (10 mm) compacted to a height of 0.394 inches (10 mm) and tested according to MPIF Standard 40(10). As-sintered tensile properties were measured on specimens according to MPIF Standard 10(10). Heat treated tensile properties were generated utilizing machined round specimens with threaded ends. These specimens were prepared by compacting blanks, sintering, rough machining, heat treatment and final grinding. The finished test specimens have a gauge diameter of 0.2 inches (5 mm) and a gauge length of 1 inch (25.4 mm).

RESULTS AND DISCUSSIONS

Powder Properties

The apparent density and flow of the powders were measured at 290°F (145°C) as described above and the test results are listed in Table II. This data clearly indicates that the material system provided premixes with the capability to flow rapidly even at the elevated temperature. If traditional lubricants with much lower melting temperatures had been utilized, the lubricants would have melted, become tacky and prevented a free flowing powder.

Table II: Flow and Apparent Density

Material	Flow at 290°F/145°C (sec/50g)	Apparent Density at 290°F/145°C (g/cm ³)
A	23.9	2.96
B	23.9	2.94

Green Properties

The green properties generated are listed in Table III. Pore free density values for each mix were determined on each mix utilizing a pycnometer and are included in Table III along with a

calculation of the percent of pore free density achieved at 50 tsi (690 MPa). The pore free density represents the density that the green compact would reach if all the pores could be removed. The value for pore free density is greatly affected by the addition of lubricants and graphite which have a much lower specific gravity than iron and thus occupy more volume for a given weight % of addition. Since obtaining green density values close to the pore free density is possible in the warm compaction process, knowing the value for pore free density of a given mix is critical in understanding the potential density that can be achieved by that premix composition.

The data indicates that the green densities for the two materials are essentially identical (Figure 2). These results point out a unique characteristic of the diffusion alloyed materials. Despite the much higher alloy content of material A, the compressibility remains similar to material B. The diffusion alloying process did not significantly affect the compressibility of the base iron. In both cases, the materials reached over 98% of pore free density. The green strength of the material is typical of that expected from warm compacted materials and represents a significant improvement over traditionally compacted materials.

Table III: Green Properties

Matl.	Comp. Press.	Green Density (g/cm ³)	Green Strength	Green Exp. From Die (%)	Peak Ejection Force	Pore Free Density (g/cm ³)	% Pore Free@ 50tsi
A	30(tsi) 415(MPa)	7.14	3945(tsi) 27.2(Mpa)	0.20	2.96(tsi) 40.8(MPa)		
	40 550	7.31	4137 28.5	0.27	3.18 43.9		
	50 690	7.36	4000 27.6	0.33	3.00 41.4	7.507	98.04
B	30(tsi) 415(MPa)	7.14	3912(tsi) 27.0(Mpa)	0.20	2.69(tsi) 37.1(MPa)		
	40 550	7.31	3956 27.3	0.27	2.85 39.3		
	50 690	7.34	3712 25.6	0.33	2.85 39.3	7.475	98.19

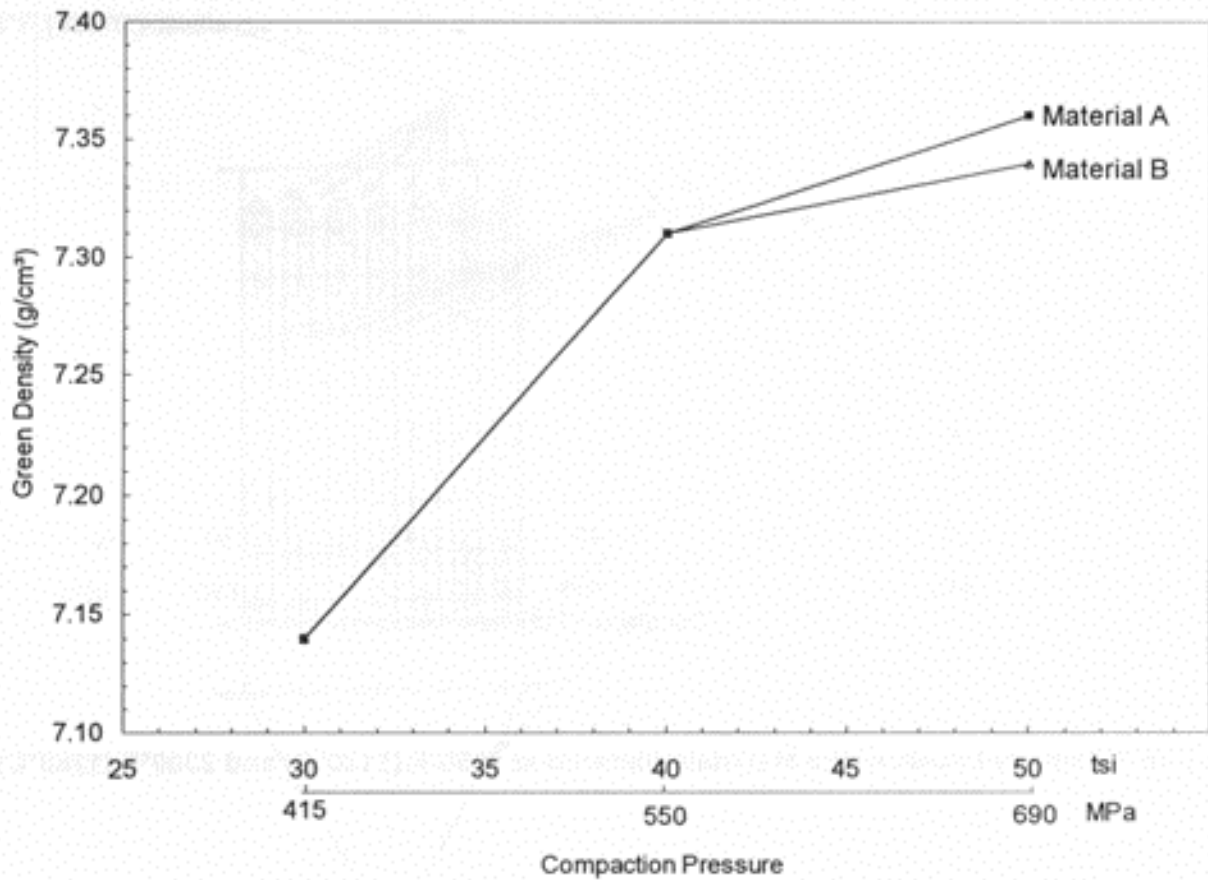


Figure 2: Compressibility Curves

Sintered Properties

Tables IV and V lists the sintered TRS properties obtained at 2050°F (1120°C) and 2300°F (1260°C). The sintered densities obtained by each material are presented in Figure 3. This figure, along with the dimensional change results, indicate that material A shrinks more during sintering than material B. The increased shrinkage is a result of the higher nickel content in material A. At the high density values obtained from the warm compaction process, both materials are highly ductile. As a result, tensile properties are a better indication of relative strength than those obtained from transverse rupture strength testing.

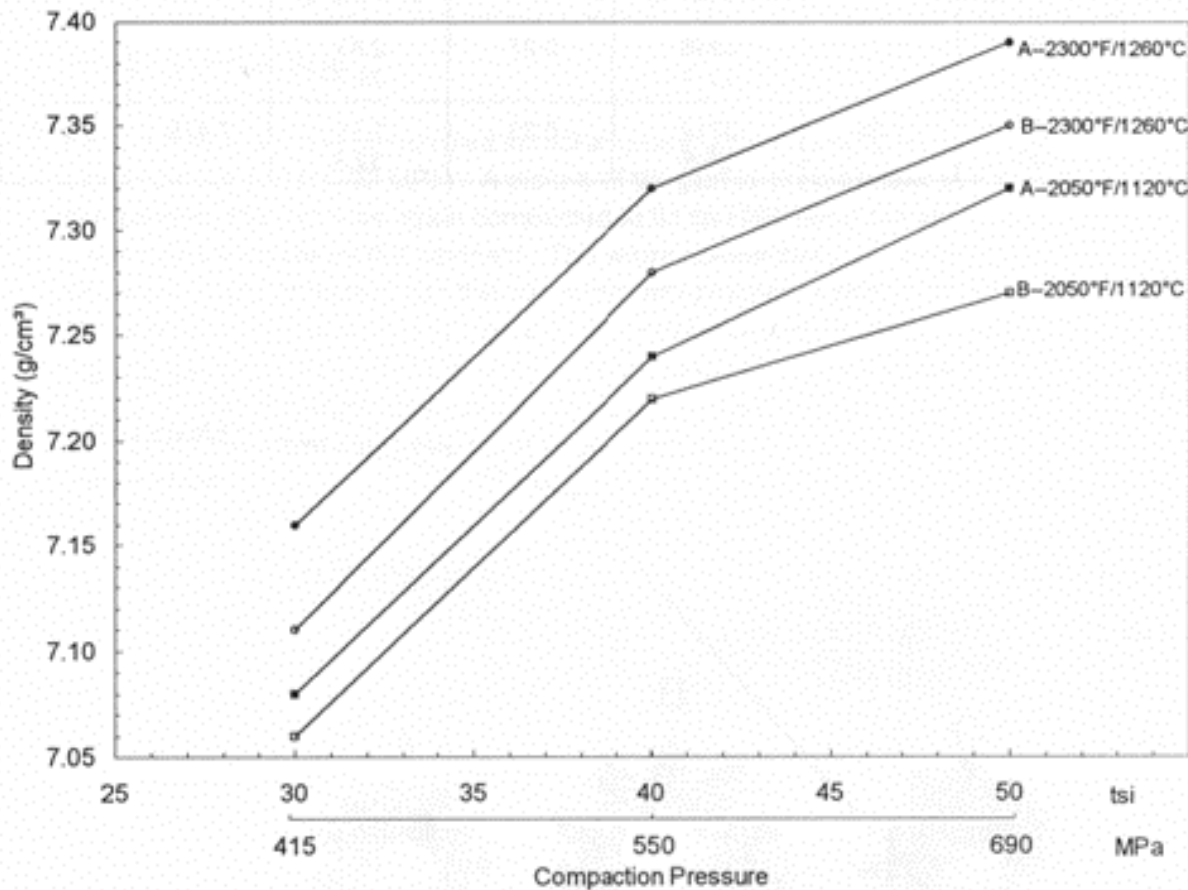


Figure 3: Density Levels of the Materials Sintered at 2050°F (1120°C) and 2300°F (1260°C)

Table IV: Sintered Properties from Transverse Rupture Bars Sintered at 2050°F (1120°C)

Matl.	Compaction Pressure	Sintered Density (g/cm ³)	TRS	Dim. Chg. From Die (%)	HRB
A	30(tsi) 415(MPa)	7.08	216.7(10 ³ psi) 1494(MPa)	0.04	86
	40 550	7.24	237.1 1635	0.14	92
	50 690	7.32	244.8 1688	0.17	93
B	30(tsi) 415(MPa)	7.06	181.7(10 ³ psi) 1253(MPa)	0.24	81
	40 550	7.22	205.3 1415	0.32	86
	50 690	7.29	216.6 1493	0.38	87

Table V: Sintered Properties from Transverse Rupture Bars Sintered at 2300°F (1260°C)

Matl.	Compaction Pressure	Sintered Density (g/cm ³)	TRS	Dim. Chg. From Die (%)	HRB
A	30(tsi) 415(MPa)	7.16	223.2(10 ³ psi) 1539(MPa)	-0.23	21C
	40 550	7.32	261.0 1800	-0.08	25C
	50 690	7.39	275.2 1897	0.00	26C
B	30(tsi) 415(MPa)	7.11	167.6(10 ³ psi) 1156(MPa)	0.05	80
	40 550	7.28	195.9 1351	0.14	85
	50 690	7.35	203.5 1403	0.19	86

As-Sintered Tensile Properties

Results obtained from dogbone tensile testing of the two materials are listed in Tables VI (2050°F/1120°C) and VII (2300°F/1260°C). The 0.2% offset yield strength, ultimate tensile strength and elongation are presented in Figures 4, 5, and 6, respectively. As expected, the data indicates that the strength of material A is significantly higher than that of material B. Additionally, material B exhibits a slightly higher elongation at a given sintering temperature than material A. Both of these findings are consistent with the higher alloy content in material A which provides greater hardenability and thus higher strength and lower ductility. In all cases, the strength of the materials is dependent strongly on density. In all but one case, the strength values are enhanced by increasing the sintering temperature. However, material B showed slightly lower ultimate tensile strength for a given density at the higher sintering temperature.

The elongation results are more complex. In most cases for a given density, the materials sintered at the lower temperature exhibit slightly higher elongation values. This may be due to more complete alloying occurring at the higher sintering temperature and thus more uniform and hardenable microstructure. In material B, the elongation also drops slightly at the higher density when the materials are sintered at the lower temperature. This effect is thought to be the result of a rapid increase in yield strength at higher densities while the ultimate tensile strength increases only slightly. The result is a lower ratio of ultimate strength to yield strength and thus lower elongation. The effect has been observed in a variety of ANCORSENSE materials and, as noted in this set of experiments, appears to be more prominent at lower sintering temperatures.

Table VI: As-Sintered Tensile Properties for the Materials

Sintered at 2050°F (1120°C)

Matl.	Sintered Density (g/cm ³)	0.2% Yield Strength	Ultimate Tensile Strength	Elongation (%)	HRB
A	7.06	55.7(10 ³ psi) 384(MPa)	92.7(10 ³ psi) 639(MPa)	2.1	90
	7.25	59.0 406	108.0 744	2.5	94
	7.32	65.4 450	113.8 784	2.4	96
B	7.04	52.0(10 ³ psi) 358(MPa)	83.2(10 ³ psi) 573(MPa)	2.5	82
	7.21	54.7 377	93.0 641	2.9	86
	7.27	56.3 388	95.7 659	2.7	91

Table VII: As-Sintered Tensile Properties for the Materials Sintered at 2300°F (1260°C)

Matl.	Sintered Density (g/cm ³)	0.2% Yield Strength	Ultimate Tensile Strength	Elongation (%)	HRB
A	7.14	67.8(10 ³ psi) 467(MPa)	107.0(10 ³ psi) 737(MPa)	2.0	91
	7.33	75.7 521	124.9 861	2.8	93
	7.37	76.3 526	134.1 924	3.0	98
B	7.11	56.6(10 ³ psi) 390(MPa)	83.4(10 ³ psi) 575(MPa)	2.3	83
	7.28	60.9 419	94.0 648	2.8	89
	7.35	62.5 430	101.9 702	3.8	89

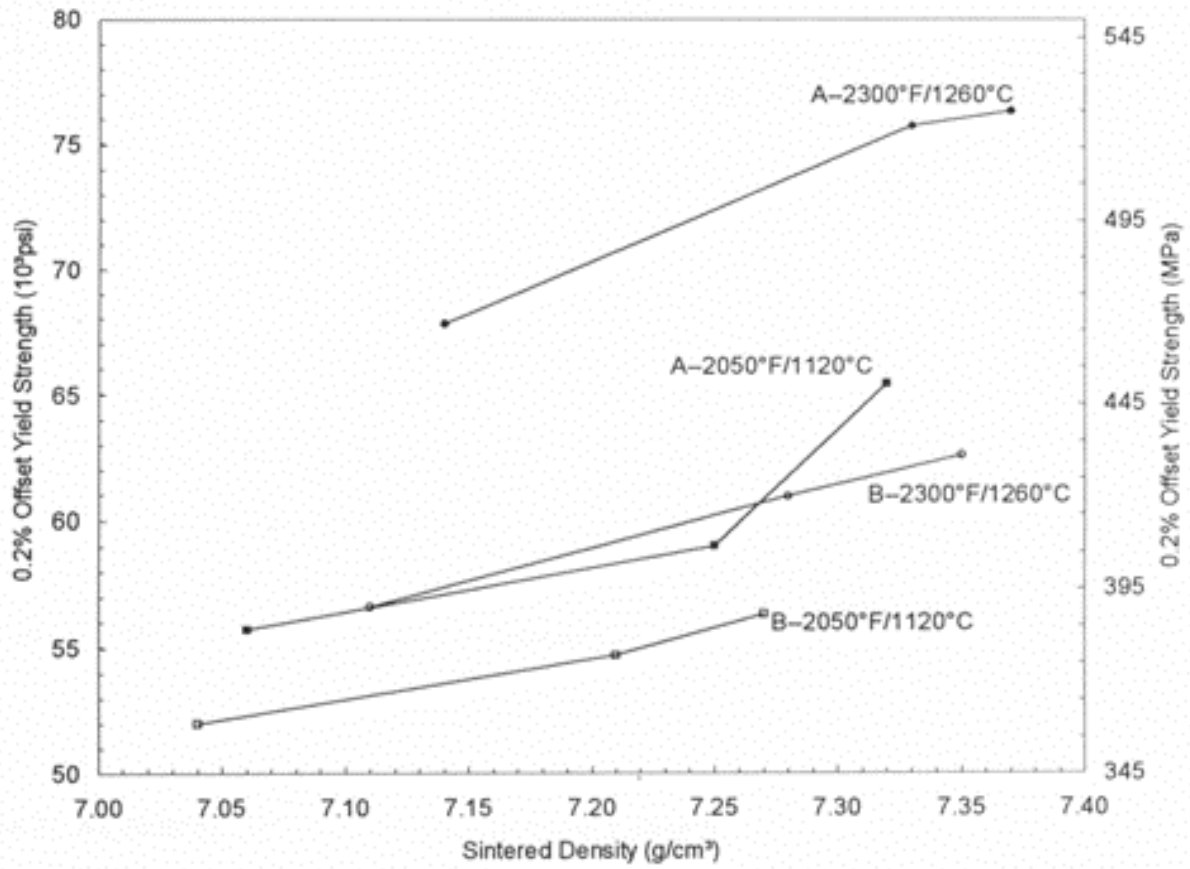


Figure 4: As-Sintered 0.2% Offset Yield Strengths

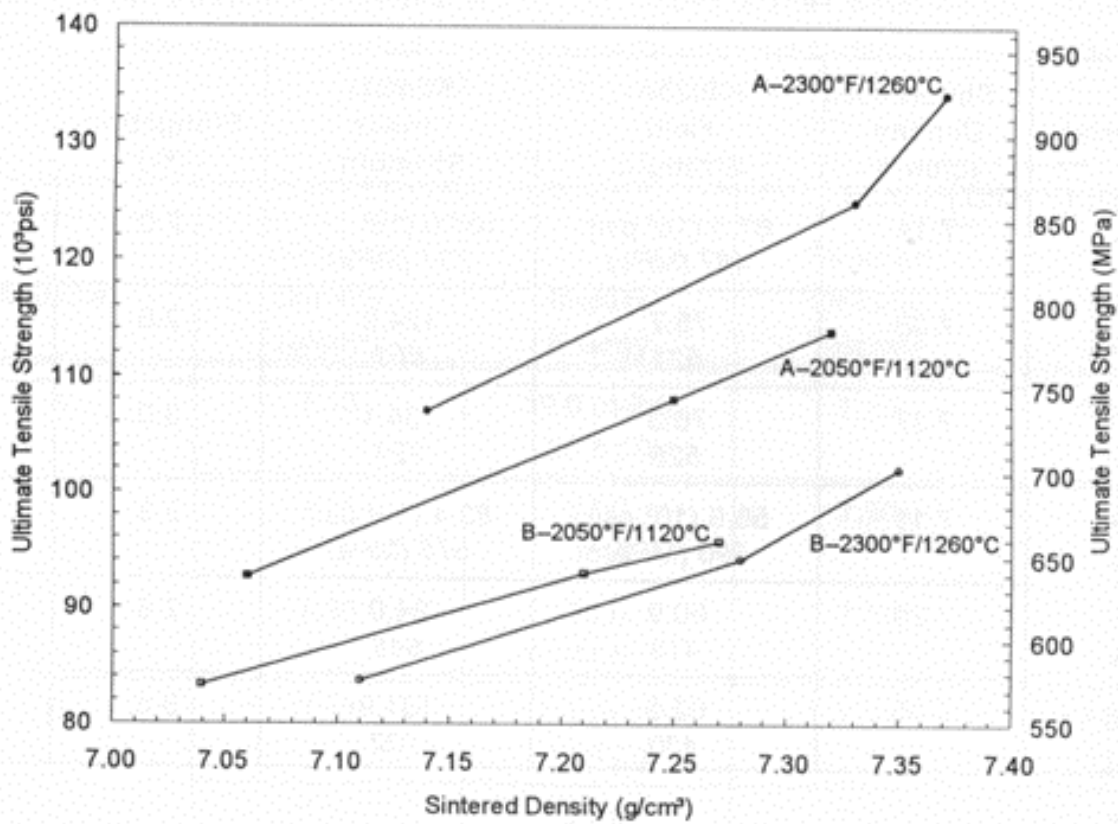


Figure 5: As-Sintered Ultimate Tensile Strengths

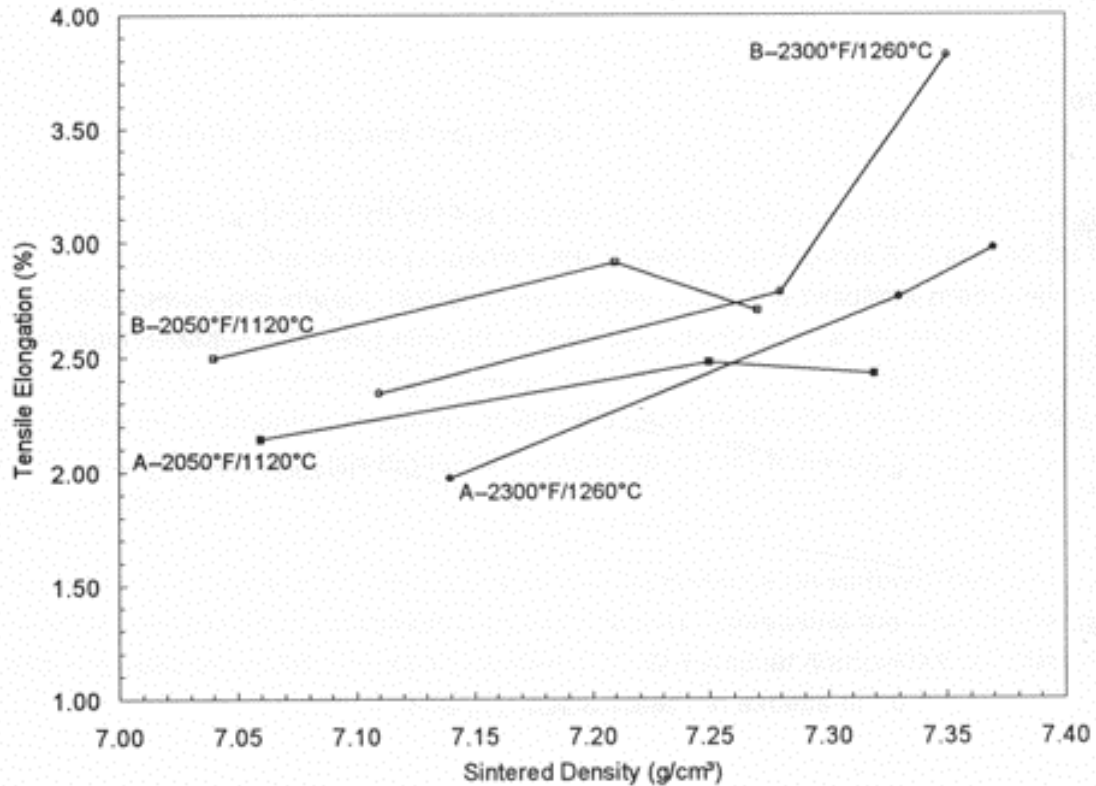


Figure 6: As-Sintered Tensile Elongation Values

As-Sintered Impact Properties

Results of un-notched charpy impact testing of as-sintered materials are listed in Table VIII and are presented as a function of sintered density in Figure 7. In all cases, the impact properties are significantly improved by increasing density. In the case of material A, impact properties are slightly decreased when the material is sintered at higher temperatures. This is most likely due to a similar explanation as was noticed with elongation. A more complete alloying as a result of increased sintering temperature results in a harder microstructure and decreases the impact properties. Material B, with a smaller alloy content, indicates the expected increase in impact resistance with increasing sintering temperature.

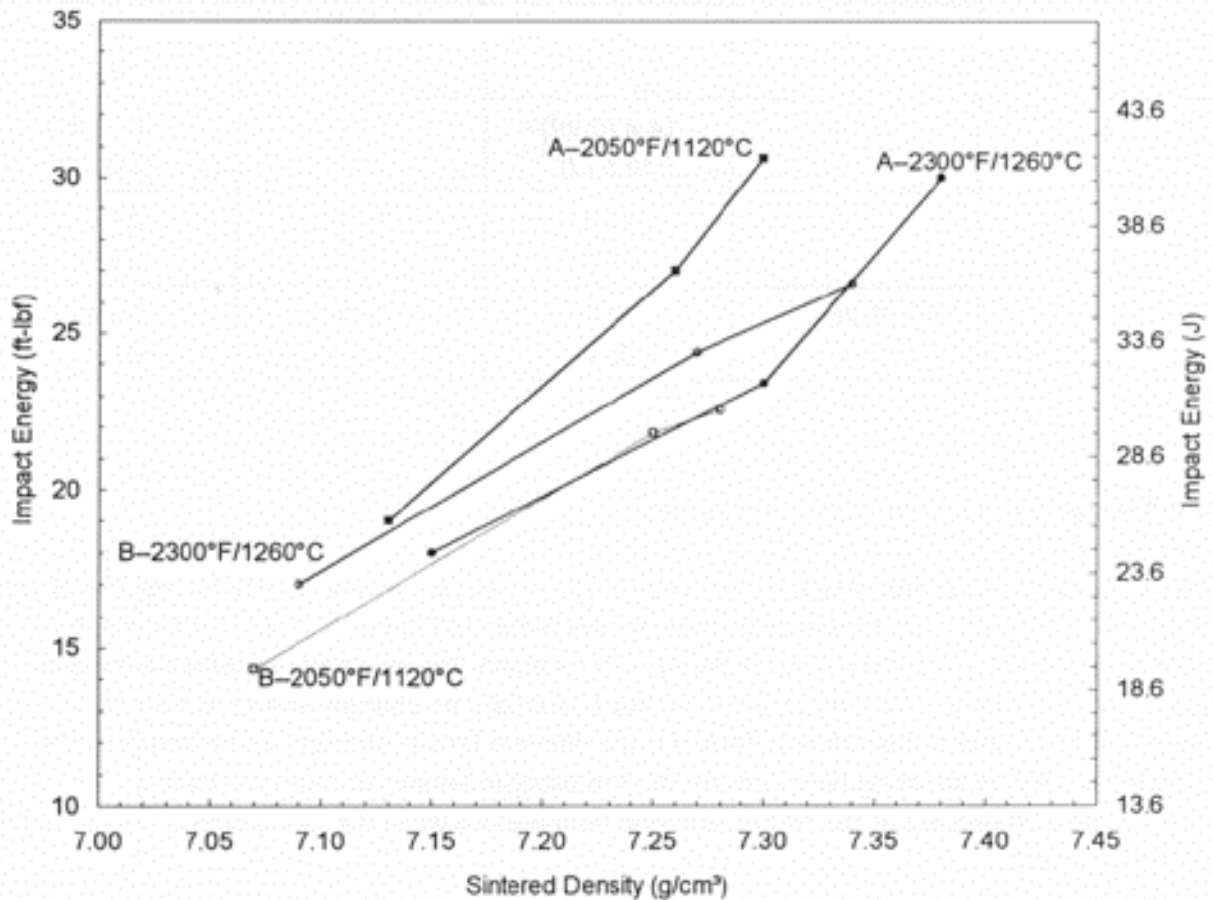


Figure 7: As-Sintered Un-Notched Charpy Impact Values

Table VIII: As-Sintered Impact Properties for the Materials Sintered at 2050°F (1120°C) and 2300°F (1260°C)

Matl.	2050°F (1120°C) Sinter		2300°F (1260°C) Sinter	
	Sintered Density (g/cm ³)	Impact Energy (ft-lbf) (J)	Sintered Density (g/cm ³)	Impact Energy (ft-lbf) (J)
A	7.13	19.0(ft-lbf) 25.8(J)	7.15	18.0(ft-lbf) 24.4(J)
	7.26	27.0 36.6	7.30	23.4 31.7
	7.30	30.6 41.5	7.38	30.0 40.7
B	7.07	14.4(ft-lbf) 19.5(J)	7.09	17.0(ft-lbf) 23.0(J)
	7.25	21.8 29.6	7.27	24.4 33.1
	7.28	22.6 30.6	7.34	26.6 36.1

Heat Treated Tensile and Impact Properties

The tensile and impact properties of heat treated specimens are available only for material B and are listed in Table IX. The tensile properties are presented in Figures 8, 9 and 10 for tensile strength, elongation and impact, respectively. Once again, the resultant properties are highly dependent on density. The yield strength shows essentially no change when the material is sintered at the higher temperature. However, the ultimate tensile strength and elongation are highly influenced by sintering temperature with the ultimate tensile strength increasing approximately 20 percent at the higher sintering temperature while the elongation is improved by about 75 percent.

Heat treated impact values also indicate a strong dependence on sintered density and are excellent for the strength level exhibited by the material. Increasing the sintering temperature resulted in greatly improved impact properties with the samples compacted at 50 tsi (690 MPa) and sintered at 2300°F (1260°C) showing impact values in excess of 16 ft-lbf (22 J).

Table IX: Heat Treated Properties of Material B

Sint. Temp.	Sint. Dens. (g/cm ³)	0.2% Offset Yield Strength	Ultimate Tensile Strength	Elongation (%)	Impact Energy
2050°F/ 1120°C	7.06	141.9(103psi) 978(MPa)	158.3(103psi) 1091(MPa)	1.1	8.5 (ft-lbf) 11.5(J)
	7.11	157.9 1088	176.6 1217	1.1	8.8 11.9
	7.27	174.9 1205	188.6 1300	1.2	10.2 13.8
2300°F/ 1260°C	7.11	154.5(103psi) 1065(MPa)	190.3(103psi) 1312(MPa)	1.8	9.0 (ft-lbf) 12.2(J)
	7.28	166.9 1150	209.1 1441	2.0	15 20.3
	7.35	174.6 1203	220.5 1520	2.1	16.2 22.0

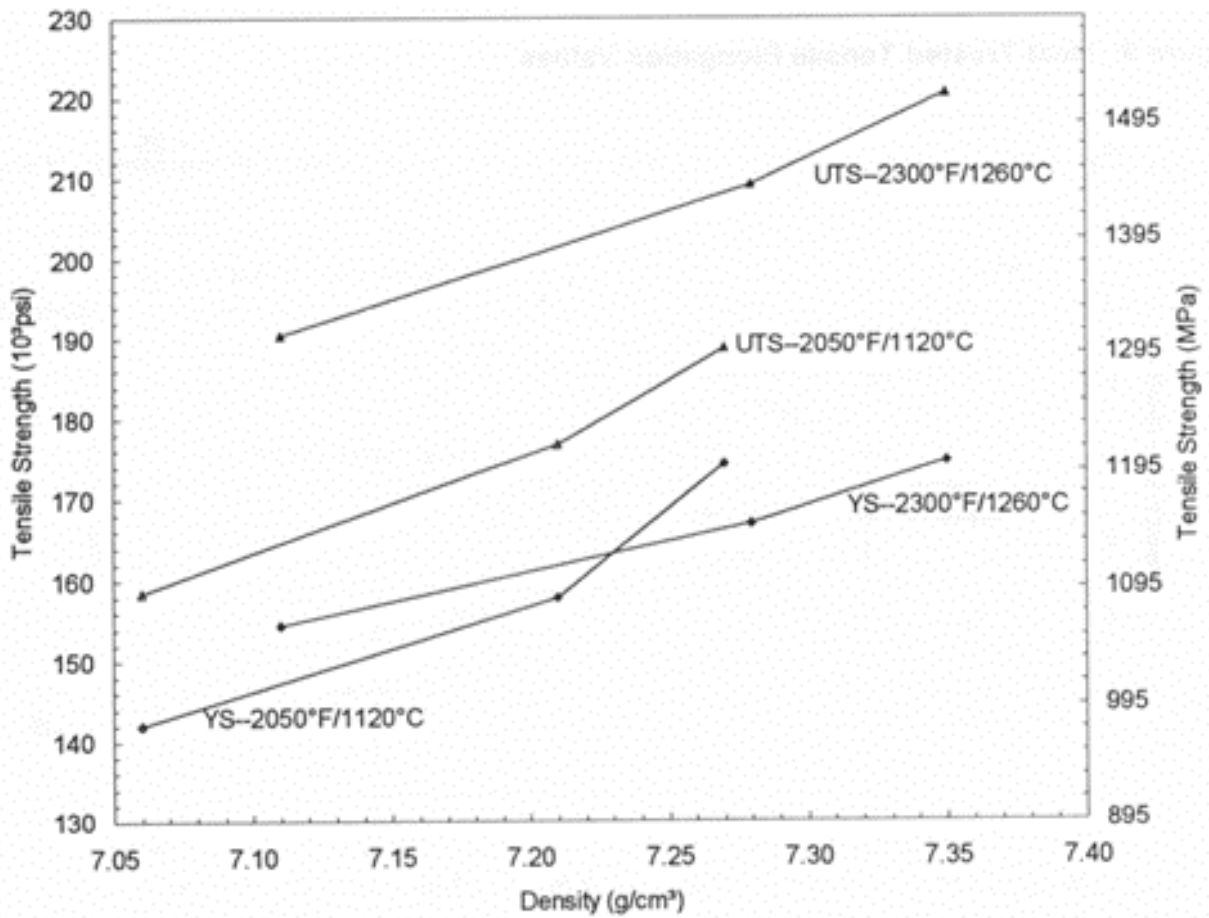


Figure 8: Heat Treated Tensile Strengths

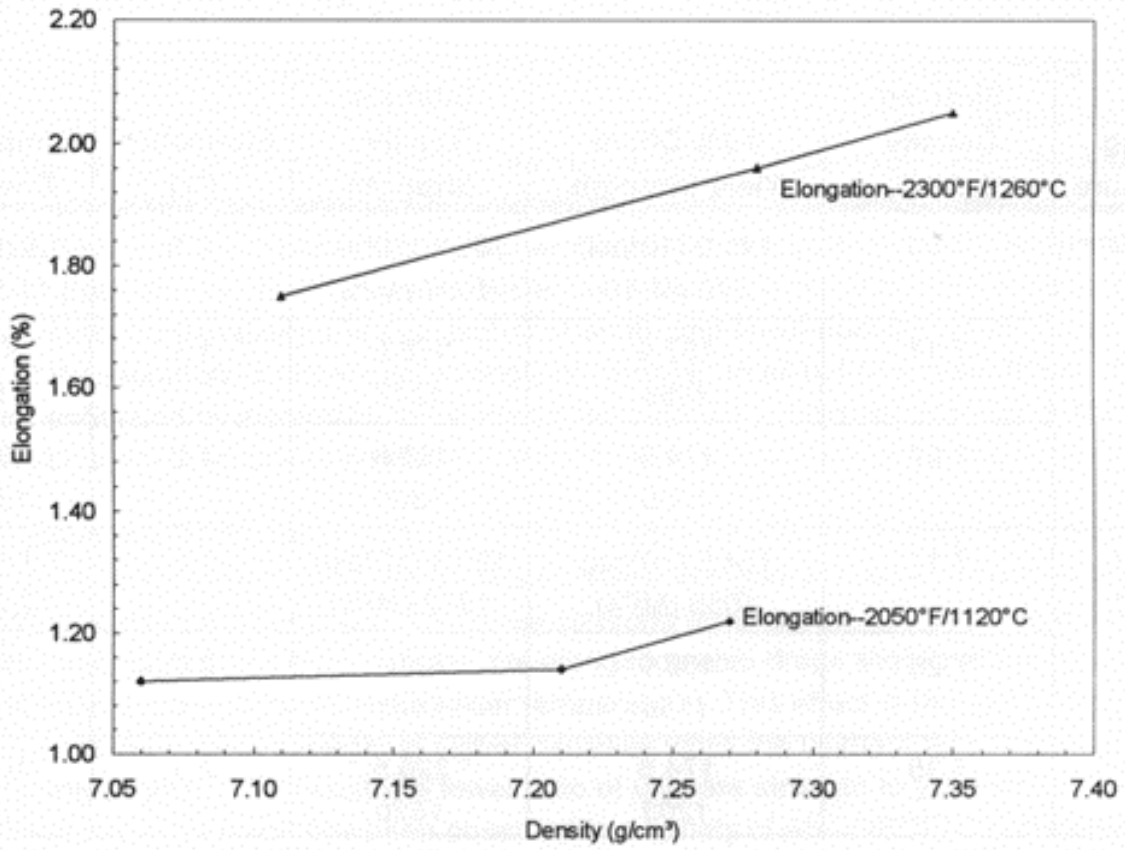


Figure 9: Heat Treated Tensile Elongation Values

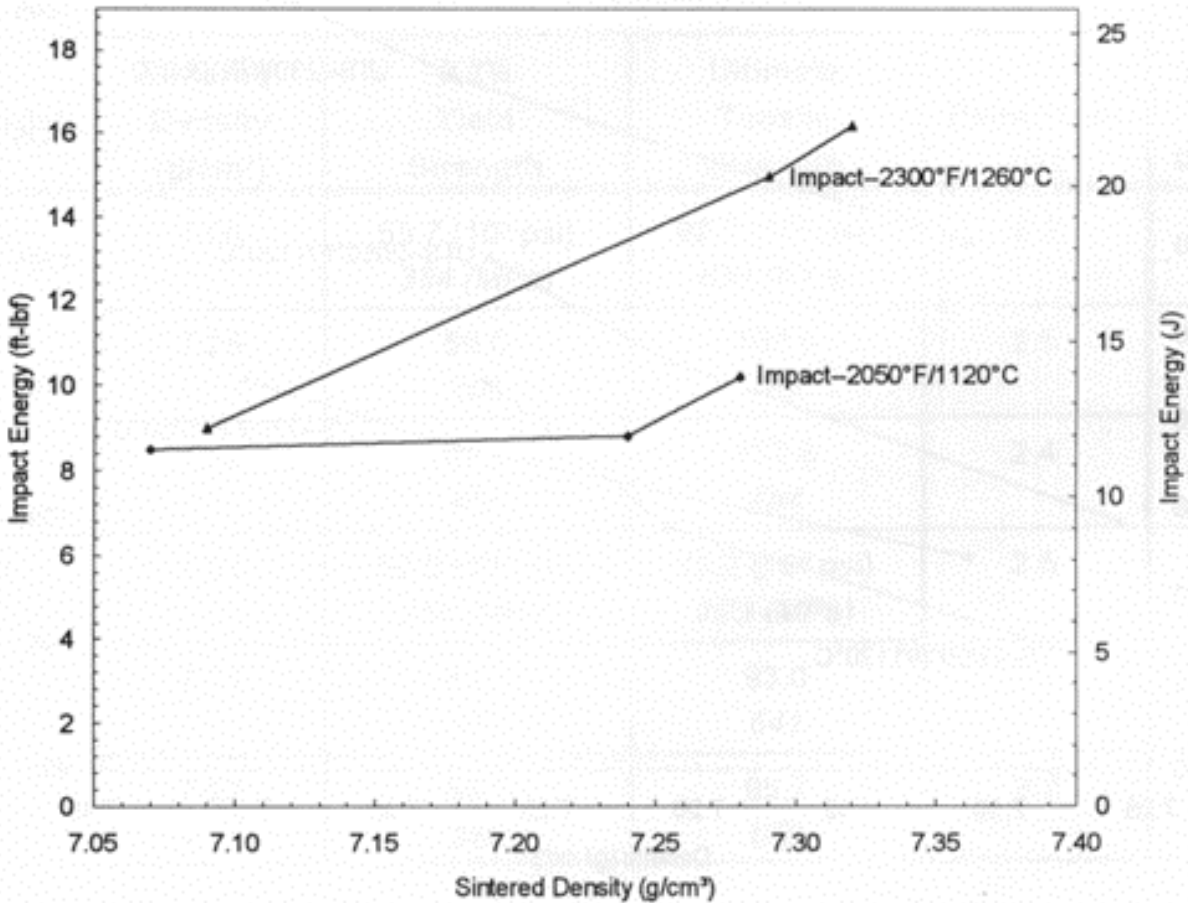


Figure 10: Heat Treated Impact Values

CASE STUDIES

The warm compaction process has been shown to be commercially viable(11). In order to further understand the warm compaction process and how it performs in actual production, several parts were compacted on production equipment to evaluate the process's capability.

Trials 1-3

Three parts were run utilizing a Dorst 45-ton mechanical press equipped with a powder heating system as shown in Figure 11. This powder heating system consists of a specially designed heat exchanger. In this design, the powder is filled into 0.4 inches (10 mm) slots which are heated by oil to the required temperature. As the powder is consumed by the compaction process, a level indicator in the hopper below the heater signals the valve in the bottom of the heater which then opens and closes to keep the powder in the hopper at a constant level. The heated powder is then feed through a hose into the shuttle. Both the

hose and shuttle are heated and insulated to ensure against heat loss. This heating unit has been shown to easily meet the requirement of maintaining powder temperature control to within $\pm 5^{\circ}\text{F}$ ($\pm 2.5^{\circ}\text{C}$).

In these three trials, a premix identical in composition to material A was used (Distaloy 4800A {Distaloy AE} + 0.5 w/o graphite + 0.6 w/o lubricant). An automatic weighing system was utilized to evaluate the weight variability of the parts. For these three trials a powder temperature of 265°F (130°C) and a die temperature of 300°F (150°C) was maintained.

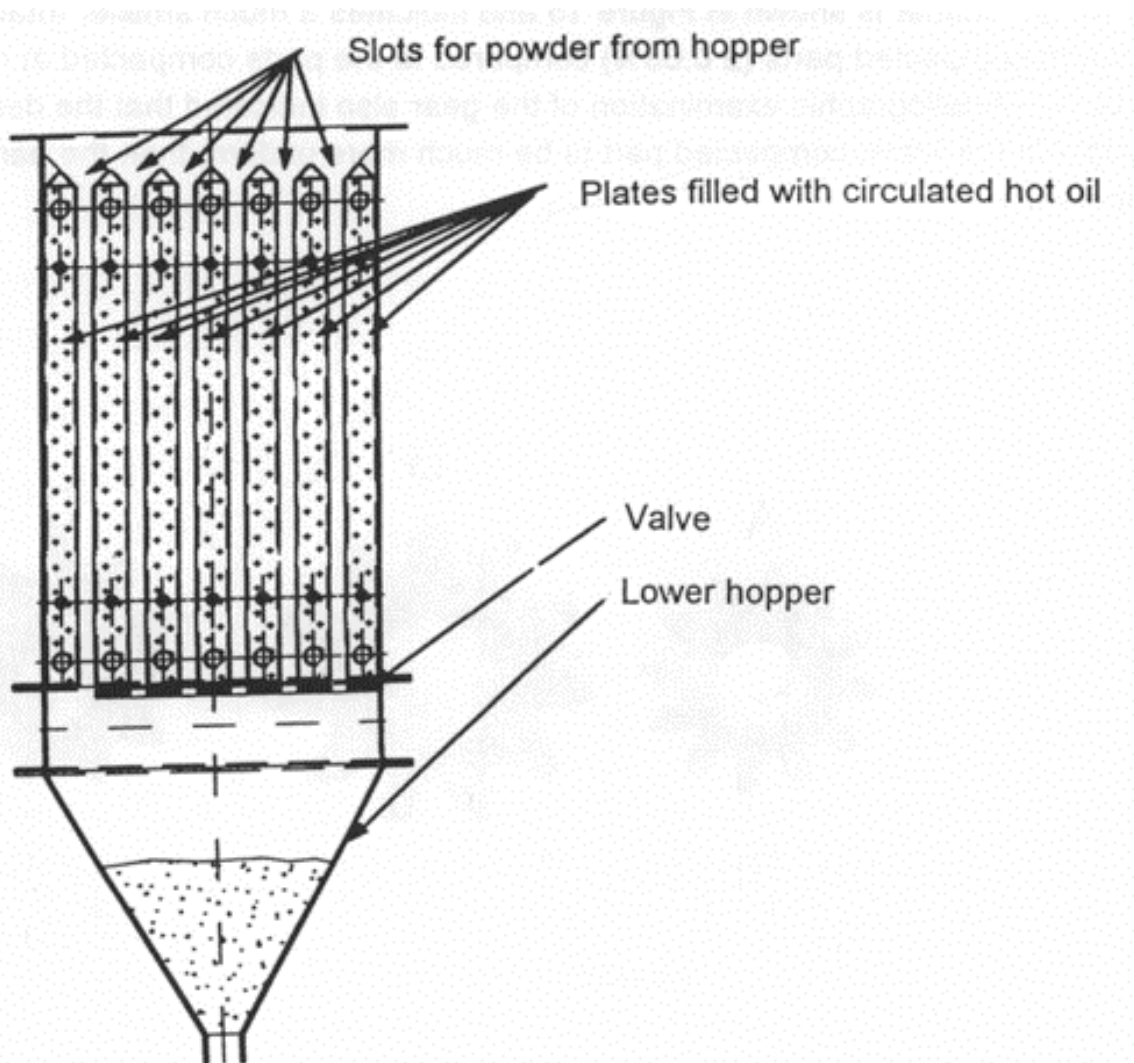


Figure 11: Powder Heating Unit Utilized in Part Trials

Trial 1--Gear

In the first trial, a common gear shape (Figure 12-part A) with a nominal weight of 15 grams was compacted at 51 tsi (700 MPa). A

green density of 7.35 g/cm^3 was achieved and metallography again indicated that excellent density distribution throughout the part was achieved. Total weight scatter through the run was good at $\pm 0.79\%$ as shown in Figure 13.

Trial 2--Oil Pump Gear

A second test was made with an oil pump gear with a nominal weight of 47.5 grams. This part is shown in Figure 12 (part B). Again, the part was compacted at 51 tsi (700 MPa) and a green density of 7.35 g/cm^3 was achieved. Each part was weighed and the results are shown in Figure 14. Total part weight variability was $\pm 0.43\%$ and, again, the density distribution throughout the part was more consistent than that found previously in conventionally processed materials.

Trial 3--Sprocket Gear

For the third trial, several hundred sprocket gears, with a nominal weight of 14 grams, were compacted at 51 tsi (700 MPa). A picture of the part is shown below in Figure 12 (part C). As a comparison, the part was again compacted at 51 tsi (700 MPa) but at room temperature and 0.8w/o zinc stearate as the lubricant. The warm compacted part obtained an average green density 0.15 g/cm^3 higher than the conventionally processed part (7.37 g/cm^3 versus 7.22 g/cm^3). The weight scatter is shown in Figure 15 and indicates a much smaller total weight variation for the warm compacted parts ($\pm 0.63\%$) compared to the parts compacted at room temperature ($\pm 1.02\%$). metallographic examination of the gear also indicated that the density distribution throughout the warm compacted part to be much more uniform than the part compacted at room temperature.

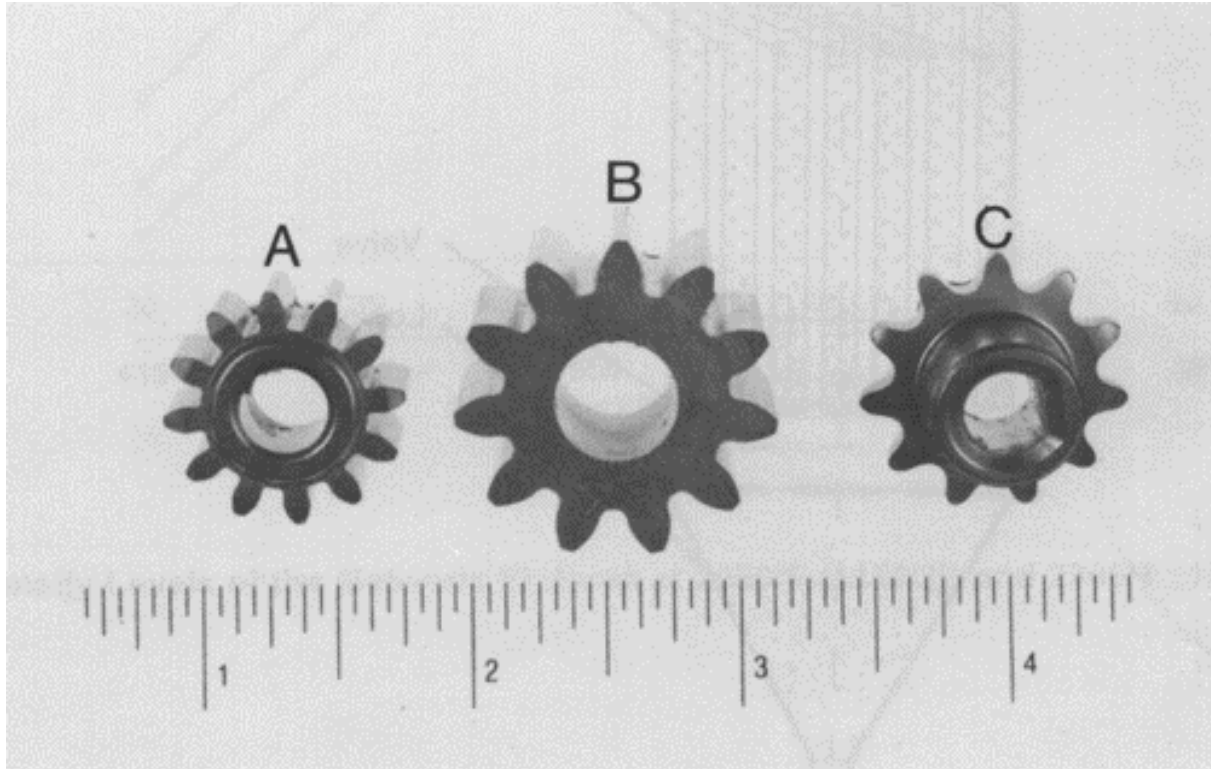


Figure 12: Trial Parts

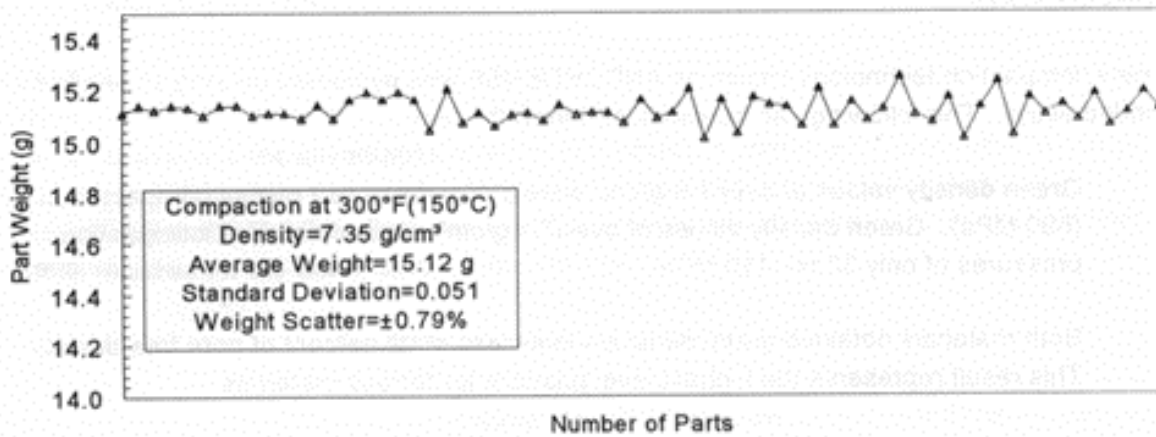


Figure 13: Weight Variability from Trial 1 (Part A)

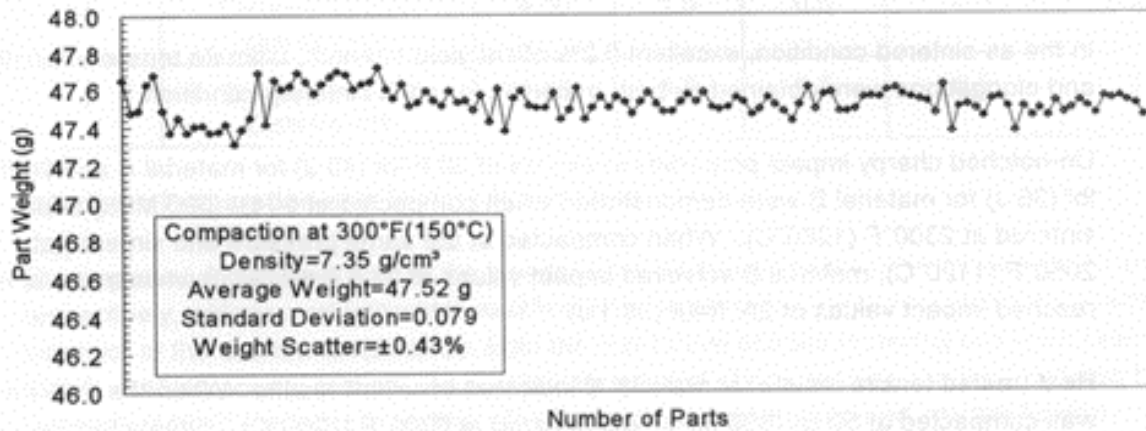


Figure 14: Weight Variability from Trial 2 (Part B)

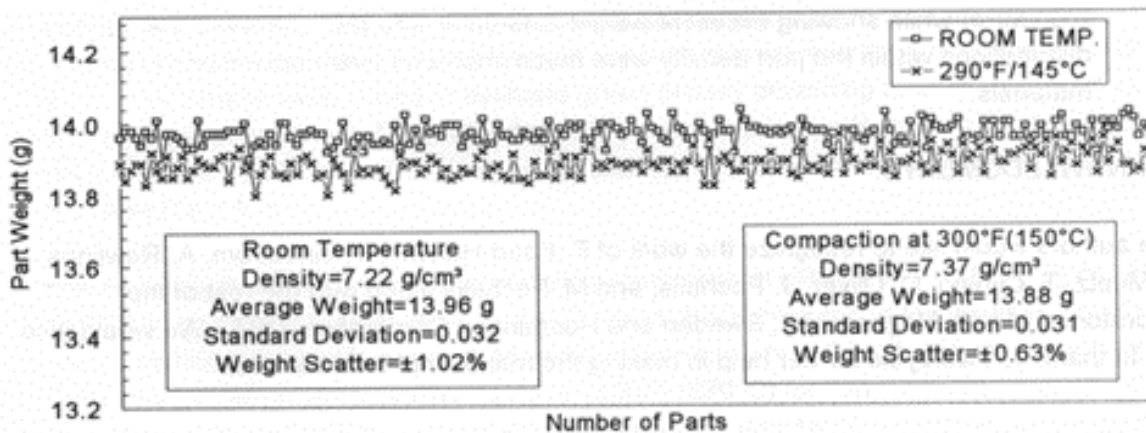


Figure 15: Weight Variability from Trial 4 (Part C)

CONCLUSIONS

A new compaction technology known as ANCORDENSE was evaluated utilizing several diffusion bonded alloys. The following can be concluded from the test:

1. Green density values of over 7.3 g/cm^3 were achieved on test pieces compacted at 50 tsi (690 MPa). Green density values of over 7.1 g/cm^3 were achieved at compaction pressures of only 30 tsi (410 MPa).
2. Both materials obtained green densities in excess of 98 percent of pore free density. This result represents the highest level possible for ferrous materials.
3. Green strengths in excess of 3700 psi (25.5 MPa) were demonstrated in both materials

4. Densities of 7.32 g/cm³ and 7.29 g/cm³ were achieved for materials A and B, respectively, when sintered at 2050°F (1120°C). Densities in excess of 7.35 g/cm³ obtained by both materials when sintered at 2300°F (1260°C).

5. In the as-sintered condition, excellent 0.2% offset yield strength, ultimate tensile strength, and elongations were obtained by both materials for each sintering condition.

6. Un-notched charpy impact properties in excess of 30 ft-lbf (40 J) for material A and 26 ft-lbf (36 J) for material B were demonstrated when compacted at 50 tsi (690 MPa) and sintered at 2300°F (1260°C). When compacted at the same pressure and sintered at 2050°F (1120°C), material B achieved impact values of 22.6 ft-lbf (30 J) while material A reached impact values of 26. ft-lbf (36.1 J).

7. Heat treated tensile results for material B indicated excellent results. When the material was compacted at 50 tsi (690 MPa) and sintered at 2300°F (1260°C), ultimate tensile strengths of 220,000 psi (1520 MPa) were achieved.

8. Trials on a variety of parts demonstrated the capability of the warm compaction system in a production environment. Green density levels of 7.35 g/cm³ were achieved at 51 tsi (700 MPa) while showing excellent weight control. It was also observed that density distributions within the part density were much improved over conventionally processed materials.

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Notes

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