ADVANCED PROPERTIES OF HIGH DENSITY FERROUS POWDER METALLURGY MATERIALS

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Presented at PM^2 TEC '95
May 14-17, 1995 - Seattle, WA USA

ABSTRACT

The introduction of the ANCORDENSE™* system has provided significantly higher density levels than previously possible in a single press/single sinter operation. This paper will explore the role that higher density has on mechanical properties. Various properties will be evaluated, including transverse rupture strength, tensile strength, and impact. Additionally, the effect of other processes, such as high temperature sintering and heat treatment, will be addressed.

INTRODUCTION

The introduction of an advanced compaction and premix technology, known as ANCORDENSE[3,4], has provided a means to produce high green and sintered density parts while increasing green strength and reducing ejection forces. The process involves compacting heated powder in heated tooling. Both the powder and the die are heated in a range of 265°F to 310°F (130°C to 155°C) with the temperature being controlled to +5°F (+2.5°C) during the process. The compaction process is diagramed in Figure 1,

Other than the addition of a powder heater and heated dies, this new technology utilizes traditional compaction presses. Currently, there are several methods available for heating powder, including heated screws[s], microwave[6] and slotted heat exchangers[7]. The lubricant/binder system specifically engineered for this process is designed to provide optimal performance at the specified temperature range. The lubricant system provides a free flowing powder at temperature, while providing excellent lubricity and reduced ejection forces.

EXPERIMENTAL PROCEDURE

Premixes were prepared using the lubricant system developed for warm compaction from a variety of base materials as listed in Table I. These materials were chosen to demonstrate the applicability of ANCORDENSE technology to a wide variety of base materials, from highly compressible pure iron and prealloy compositions to less compressible prealloyed materials.

The flow and apparent density of the powders were measured utilizing a modified Hall flow cup. In order to evaluate the premixes properties at the elevated compaction temperature, both the powder and the flow cup were preheated to 290°F (145°C) prior to testing. Once the powder temperature was stabilized, the tests were carried out in the traditional manner.

Green properties of the materials were obtained by compacting bars to a nominal height of 0.5 inches (12.7mm) high in accordance with MPIF Standard 458. The compaction process was carried out with both the die and powder heated to 290°F (145°C), at pressures of 30, 40 and 50 tsi (415, 550 and 690 MPa). The specimens were then measured and tested to determine green density and green strength according to MPIF Standards 45 and 10, respectively. The green expansion in the length direction was also determined.

Sintered properties were determined on transverse rupture bars compacted in the same manner as described for the green bars above except that the specimens were compacted to a nominal height of 0.25 inches (6.35 mm). The samples were then sintered in an atmosphere of 75 v/o hydrogen and 25 v/o nitrogen for 30 minutes at temperatures of 2050°F (1120°C) and 2300°F (1260°C) with one exception. Material D41 was sintered at 2350°F (1290°C) and tempered at 375°F (190°C) for one hour in air. The higher sintering temperature was utilized to reduce the oxides present in the manganese and chromium ferroalloy additions.

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*ANCORDENSE is a trademark of the Hoeganaes Corporation.

Following sintering, the bars were measured and tested to determine sintered density, dimensional change from die size in the length direction, sintered hardness and transverse rupture strength in accordance with MPIF Standards 44 and 41.

As-sintered tensile results were determined on dogbone tensile bars compacted and sintered in a similar fashion to the TRS specimens above. The bar geometry and the test methods used to determine 0.2% offset yield strength, ultimate tensile strength and tensile elongation conform to MPIF Standard 10. Impact properties were measured in accordance with MPIF Standard 40.

RESULTS AND DISCUSSIONS

Powder Properties

The apparent density and flow of the various premixes are listed in Table II. The data indicates that excellent flow properties are maintained by the lubricant system at elevated temperatures. The properties are on par with those expected with ANCORBOND®** materials.

Table II: Powder Properties of Premixes Used in Tests

Material	Apparent Density at 290`F (145`C) (g/cm^3)	Flow at 290 `F (145`F) (sec/50g)
D10	2.93	28.8
D85	3.06	26.4
D20	2.98	24.3
[:)15	3.12	22.6
D41	2.95	23.4

Green Properties

Table Ill lists the green properties of the various materials. Figure 2 show compaction curves for each of the five materials. The results are generally as expected with the less compressible material (D20) demonstrating the poorest compressibility of the group. However, the warm compaction process still allows a density of 6.95 g/cm^3 at 30 tsi (415 MPa) and over 7.25 g/cm^3 at 50 tsi (690 MPa). The premixes made with the highly compressible molybdenum based materials all show similar compressibility. All three materials obtained green density levels in excess of 7.0 g/cm^3 at 30 tsi (415 MPa) and in excess of 7.3 g/cm^3 at 50 tsi (690 MPa). The D85 premix indicated the best green density of the group, obtaining a green density of 7.37 g/cm^3 at 50 tsi (690 MPa). This represents 98.4% of the pore free density. With current technology, this level of remaining porosity is the lowest that might be expected. Unexpectedly, the iron based premix (D10) indicated a slightly poorer green density at 50 tsi (690 MPa) than the molybdenum based prealloy-based compositions. The material still obtained a green density of 7.29 g/cm^3 at 50 tsi (690 MPa) and showed the highest green density of the group (7.14 g/cm$^{3)}$ at 30 tsi (415 MPa).

The green strength levels of the materials are shown in Figure 3. As anticipated the green strength levels of all materials are quite high, ranging from over 3200 psi (22.1 MPa) to over 4500 psi (31.0 MPa). It is interesting to note that over a wide range of green density levels, the green strength does not vary significantly. This indicates that the green strengths of the materials are more a function of the lubricant system than iron particle interaction. It should also be noted that the green strengths of the two mixes containing 0.6% graphite addition are slightly lower than those of the premixes containing 0.4% graphite. This effect has been observed in other warm compacted premix compositions where higher graphite level appears to have a negative influence on green strength.

Transverse Rupture Strength

Data from transverse rupture strength testing are listed in Tables IV and V for the materials sintered at 2050°F (1120°C) and 2300°F (1260°C), respectively. The sintered density levels of the five materials are shown in Figures 4 (20500F {1120°C}) and 5 (2300°F {1260°C}). The sintered density results generally follow the green density results. When compacted at 50 tsi (690 MPa), the D85 material exhibited the highest sintered density, 7.44 g/cm^3 and 7.48 g/cm^3 at 2050°F (1120°C) and 2300°F (1260°C), respectively.

Test values for dimensional change from die size (Figure 6 for 2050°F {1120°C} and Figure 7 for 2300°F {1260°C}) show that a wide range of results were observed for the five materials and two sintering temperatures. At 2050°F (1120°C), the nickel containing materials (D10, D85 and D15) exhibit the least amount of growth with the growth increasing as the amount of prealloy content decreases. Material D20, the prealloy with no addition of nickel, indicates the highest amount of growth. Interestingly, the amount of shrinkage appears only as a function of density at 2300°F (1260°C), being virtually the same for the three nickel bearing materials. Not unexpectedly, the Mn and Cr material (D41) exhibits significantly higher growth than the other four materials despite the slightly higher sintering temperature.

Transverse rupture strength values are shown in Figures 8 and 9 for 2050°F (1120°C) and 2300°F (1260°C) sintering temperature, respectively. The results show the strength values increasing with both density and the alloy content of the premixes. All of the materials showed a significant amount of deflection during the test. This degree of deflection suggests that the results from tensile testing are a better indication of comparative strength between the various materials transverse rupture strength testing.

As-Sintered Tensile Properties

The results of tensile testing of the as-sintered materials are listed in Tables Vi and VII for the various materials sintered at 2050°F (1120°C) and 2300°F (1260°C), respectively. Figures 10-12 show the 0.2% offset yield strength, ultimate tensile strength and tensile elongation for the materials sintered at 2050°F (1120°c). Similarly, Figures 13-15 illustrate the same properties for the materials sintered at 2300°F (1260°C).

When sintered at 2050°F (1120°C), the strengths of the various materials follow both the alloy content of the materials and the sintered density for a given material. Material D15 compacted at 50 tsi (690 MPa), exhibits the highest values for yield (77,900 psi {537.1 MPa}) and ultimate strength (105,400 psi {726.7 MPa}) while maintaining an elongation of 1.29%. The high sintered density (7.44g/cra$^{s)}$ obtained at 50 tsi (690 MPa) by the 0.85w/o molybdenum prealloy (D85) results in both high yield and tensile strengths (65,300 psi {450.2 MPa} and 92,300 psi {636.4 MPa}) as well as an excellent ductility of over 2.8% elongation. The one surprising result involves material D20. This material exhibits a lower elongation at 50 tsi (690 MPa) than at 40 tsi (515 MPa). The reduction in elongation at the higher density level has been observed in other warm compacted materials[9] especially when sintered at lower sintering temperatures. This phenomenon is generally associated with a more rapid rise in yield strength with density relative to the increase in ultimate strength thus leading to a reduction in elongation. Material D20 indicates a much higher rise in yield strength as density increases than the other materials, and thus slightly lower elongation than expected at the highest density. It is unclear what the exact cause of this phenomenon is at this time. The other three materials all show significant increases in ductility with higher density when sintered at 2050°F (1120°C), with material D10 exceeding 3.5% elongation at the highest density.

The trends demonstrated at the 2300°F (1260°C) sintering temperature are much the same as with the lower sintering temperature, with strength being a function of both alloy content and sintered density. The D41 material exhibits by far the highest strength levels, with yield strength of 107,300 psi (739.8 MPa) and an ultimate tensile strength of 139,400 psi (961:1 MPa) when compacted at 50 tsi (690 MPa). In addition to these very high strength levels, this material shows an excellent elongation value of over 2%. The other four materials sintered at 2300°F (1260°C) exhibit higher strength and elongation values than previously noted with a 2050°F (1120°C) sinter. The elongation values in particular are improved dramatically at the higher sintering temperature.

Impact Properties

Impact properties were developed for the molybdenum-prealloyed materials (D85, D15, and D41). The results are listed in Table VIII and are shown in Figure 16 for the materials sintered at 2050°F (1120°C) and 2300°F (1260°C).

The impact properties are primarily a function of the sintered density and the sintering temperature for the three materials in this test. The results attest to the unique combination of strength and ductility found in the molybdenum-prealloyed materials. Even material D41, which obtained very high as-sintered strength, shows excellent impact properties.

Heat Treated Tensile Properties

Heat-treated tensile properties were developed for material D85 sintered at 2300°F (1260°C) and material 1341 sintered at 2350°F (1290°C). The heat treatment cycle included 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. The test specimen were rough machined prior to heat treatment and finish ground to a gauge diameter of 0.2 inches (5 mm) with threaded ends. The results are listed in Table IX and are shown in Figures 17-19 for yield, tensile and elongation, respectively. Once again, the strength values for both materials indicate a strong dependence on sintered density. Both materials indicate similar strength levels for a given density despite quite different chemistries. Even at these high strength levels, the materials exhibit high ductility for a heat-treated P/M material.

CONCLUSIONS

A variety of premix compositions were evaluated utilizing the ANCORDENSE technology. The following can be concluded from the results:

1. Each of the five tested premixes exhibited excellent flow characteristics at a typical operating temperature of 290°F (145°C).

2. Four of the five mixes obtained green densities of over 7.29 g/cm3 at 50 tsi (690 MPa). The fifth mix (D20) indicated a green density of 7.28 g/cm^3 at 50 tsi (690 MPa), an excellent result for a prealloy with lower compressibility. Material D85, highly compressible molybdenum prealloy, exhibited the highest green density of 7.37 g/cm^3 at 50 tsi (690 MPa), or 98.4% of pore free density.

3. Green strength values in excess of 3200 psi (22 MPa) were measured for all the compositions. The green strength of warm compacted materials appears to be a function of graphite content, with lower graphite contents yielding the highest green strengths.
4. When compacted at 50 tsi (690 MPa) and sintered at 2050°F (1120°C), material D85 reached a sintered density of over 7.4 g/cm^3 and materials D10 and D15 reached over 7.3 g/cm^{s}. When a sintering temperature of 2300°F (1260°C) was utilized, sintered density results of 7.45, 7.41, 7.39, 7.30 and 7.27 g/cm^{s} were achieved by materials D85, D15, D10, D20 and D41, respectively.
5. Yield strength, ultimate strength, elongation and impact properties all indicate a strong dependence on sintered density.
6. High temperature sintering and heat treatment of material D85 compacted at 50 tsi (690 MPa) resulted in excellent strength values (190,200 psi {1311 MPa} yield and 223,200 psi {1539 MPa} ultimate) while maintaining elongation values of over 2%.

ACKNOWLEDGMENTS

The authors would like to thank P. Muetz, C. Oliver, J. Pechulis and M. Pechulis for their work in support of this paper. We would like to thank J. DiLeonardo and V. Kuzmicz for preparing the premixes. We would also like to recognize S. Tworzydlo for helping make this presentation presentable.

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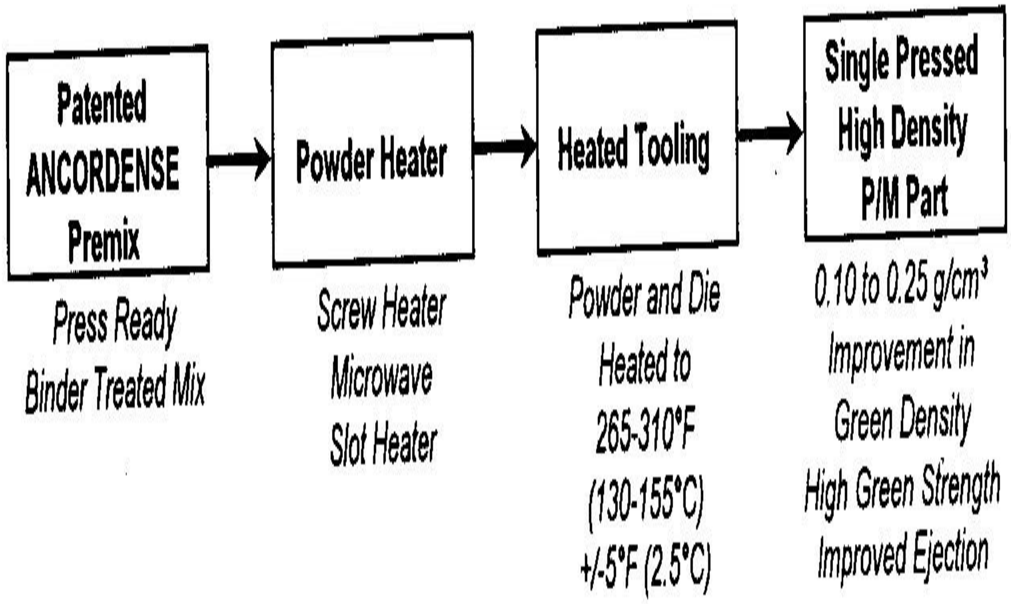


Table I: Premix Compositions Used in Tests

	Nominal Prealloy Content		Nominal Premix Additions				
Material	Mo (w/o)	Ni (w/o)	Ni (w/o)	Cr as Fe-Cr (w/o)	Mn as Fe-Mn (w/o)	Graphite (w/o)	Lubricant (w/o)
D10	--	--	--	--	--	0.6	0.6
D85	0.85	--	2.0	--	--	0.4	0.6
D20	0.58	0.47	--	--	--	0.6	0.6
D15	1.50	--	2.0	--	--	0.4	0.6
D41	0.85	--	1.0	0.75	0.90	0.4	0.6

Table III: Green Properties of the Tested Materials

Material	Compaction Pressure	Green Density (g/cm³)	Green Strength (psi)	Green Strength (MPa)	Green Exp. (%)	Pore Free Density (g/cm³)	% Pore Free Density
D10	30 tsi/415 MPa	7.14	3935	27.1	0.21	7.473	95.5
	40 tsi/550 MPa	7.26	3707	25.6	0.32		97.1
	50 tsi/690 MPa	7.29	3521	24.3	0.35		97.6
D85	30 tsi/415 MPa	7.12	3926	27.1	0.20	7.487	95.1
	40 tsi/550 MPa	7.31	4265	29.4	0.27		97.6
	50 tsi/690 MPa	7.37	3950	27.2	0.34		98.4
D20	30 tsi/415 MPa	6.95	3252	22.4	0.18	7.437	93.5
	40 tsi/550 MPa	7.19	3828	26.4	0.24		96.7
	50 tsi/690 MPa	7.28	3787	26.1	0.30		97.9
D15	30 tsi/415 MPa	7.04	3671	25.3	0.18	7.487	94.0
	40 tsi/550 MPa	7.27	4512	31.1	0.22		97.1
	50 tsi/690 MPa	7.35	4348	30.0	0.29		98.2
D41	30 tsi/415 MPa	7.09	3768	26.0	0.22	7.463	95.0
	40 tsi/550 MPa	7.26	4395	30.3	0.29		97.3
	50 tsi/690 MPa	7.33	4399	30.3	0.34		98.2

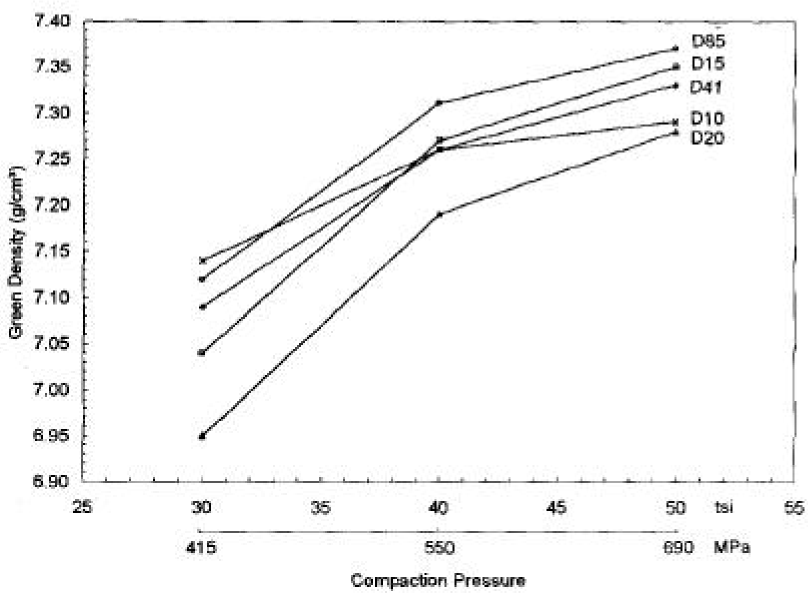
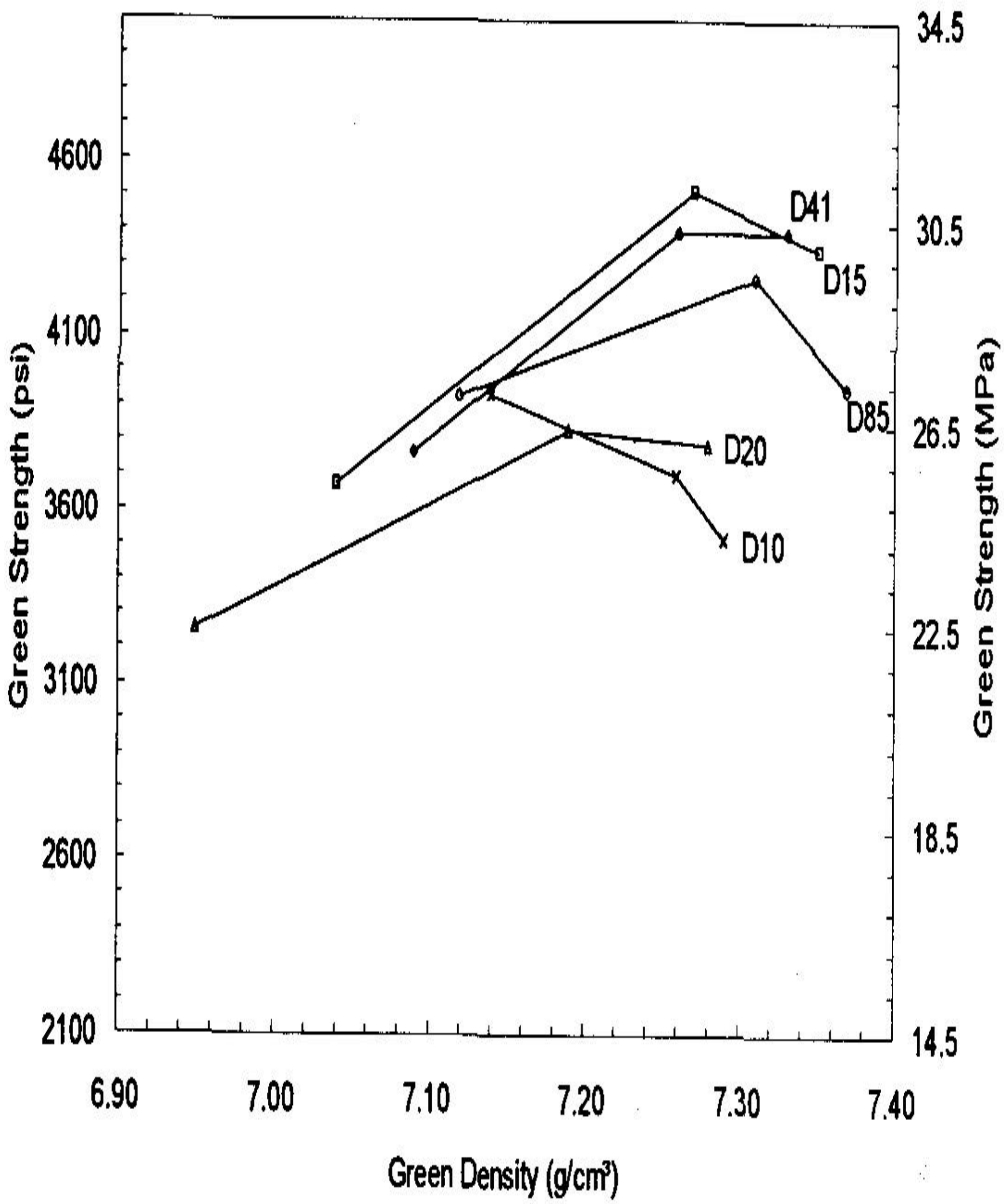


Figure 2: Compressibility Curves



Green Strength (psi)
4600
4100
3600
3100
2600
2100
Green Strength (MPa)
34.5
30.5
26.5
22.5
18.5
14.5
6.90
7.00
7.10
7.20
7.30
7.40
Green Density (g/cm³)
D41
D15
D85
D20
D10

Table IV: Transverse Rupture Results for the Materials Sintered at 2050°F (1120°C)

Material	Compaction Pressure	Sintered Density (g/cm³)	TRS (10^3 psi)	TRS (MPa)	Dimensional Change From Die (%)	HRB
D10	30 tsi/415 MPa	7.11	124.2	856	+0.08	71
	40 tsi/550 MPa	7.28	148.1	1021	+0.11	75
	50 tsi/690 MPa	7.33	158.7	1094	+0.12	77
D85	30 tsi/415 MPa	7.10	154.6	1066	-0.02	81
	40 tsi/550 MPa	7.30	186.1	1283	+0.03	86
	50 tsi/690 MPa	7.42	210.2	1449	+0.05	89
D20	30 tsi/415 MPa	6.89	107.6	742	+0.08	69
	40 tsi/550 MPa	7.15	138.6	956	+0.17	76
	50 tsi/690 MPa	7.25	149.3	1029	+0.36	80
D15	30 tsi/415 MPa	6.97	157.2	1084	-0.07	82
	40 tsi/550 MPa	7.21	192.2	1325	-0.02	87
	50 tsi/690 MPa	7.36	218.1	1508	0.00	92

Table V: Transverse Rupture Results for the Materials Sintered at 2300°F (1260°C)

Material	Compaction Pressure	Sintered Density (g/cm³)	TRS (10^3 psi)	TRS (MPa)	Dimensional Change From Die (%)	HRB
D10	30 tsi/415 MPa	7.17	128.2	884	-0.25	69
	40 tsi/550 MPa	7.33	147.5	1017	-0.15	73
	50 tsi/690 MPa	7.39	156.1	1076	-0.13	76
D85	30 tsi/415 MPa	7.14	152.2	1049	-0.25	79
	40 tsi/550 MPa	7.34	176.7	1218	-0.15	85
	50 tsi/690 MPa	7.45	199.2	1373	-0.11	87
D20	30 tsi/415 MPa	6.94	115.5	796	-0.11	70
	40 tsi/550 MPa	7.20	147.5	1017	0.00	78
	50 tsi/690 MPa	7.30	160.0	1103	+0.02	81
D15	30 tsi/415 MPa	7.04	161.2	1111	-0.32	83
	40 tsi/550 MPa	7.28	196.7	1356	-0.20	89
	50 tsi/690 MPa	7.41	221.1	1525	-0.14	93
D41	30 tsi/415 MPa	6.99	199.3	1374	+0.24	91
	40 tsi/550 MPa	7.18	237.4	1637	+0.37	96
	50 tsi/690 MPa	7.27	252.7	1742	+0.43	98

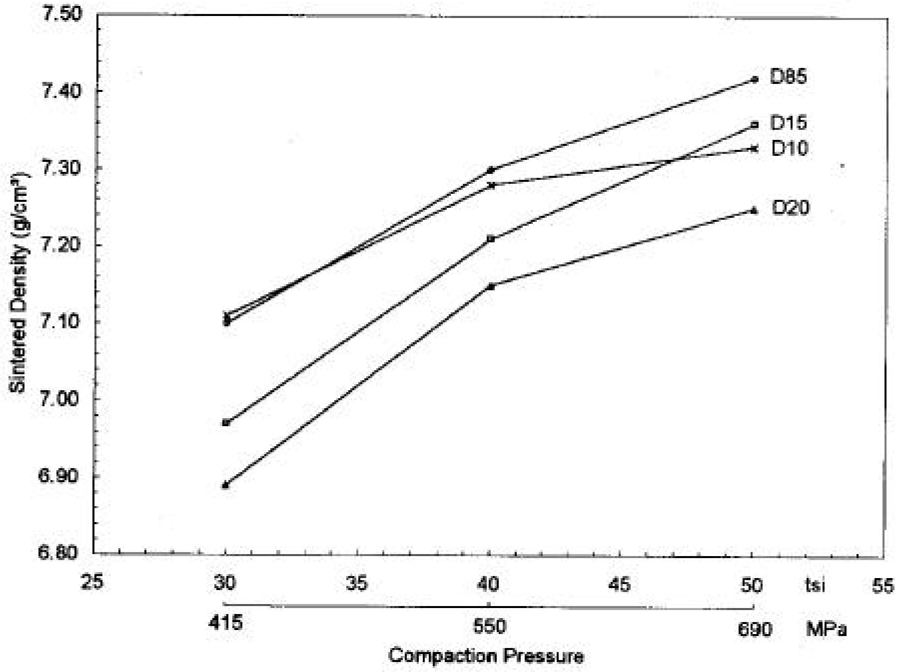


Figure 4: Density Levels for Materials Sintered at 2050°F (1120°C)

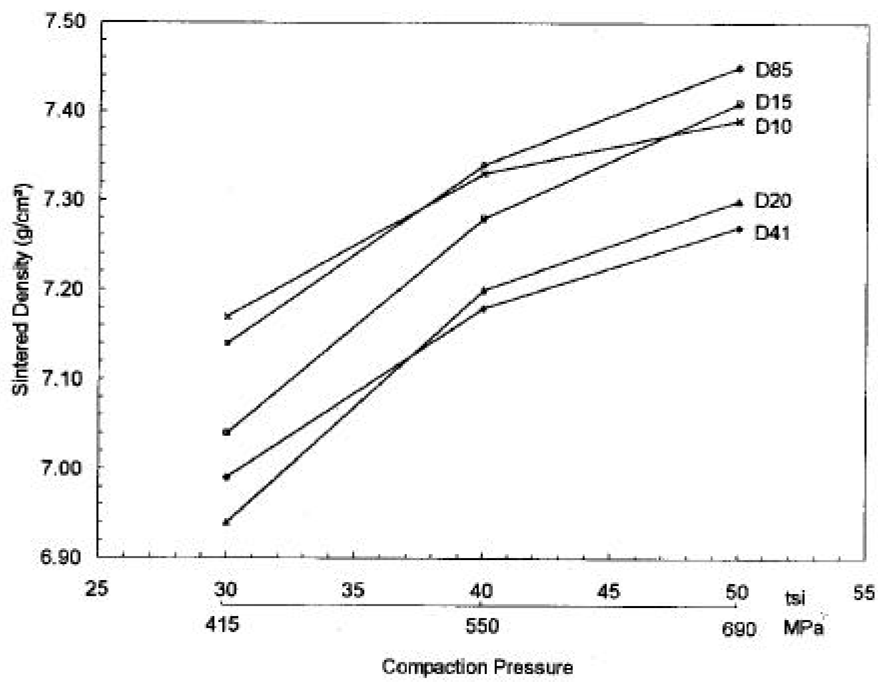


Figure 5: Density Levels for Materials Sintered at 2300°F (1260°C) [D41–2350°F{1290°C}]

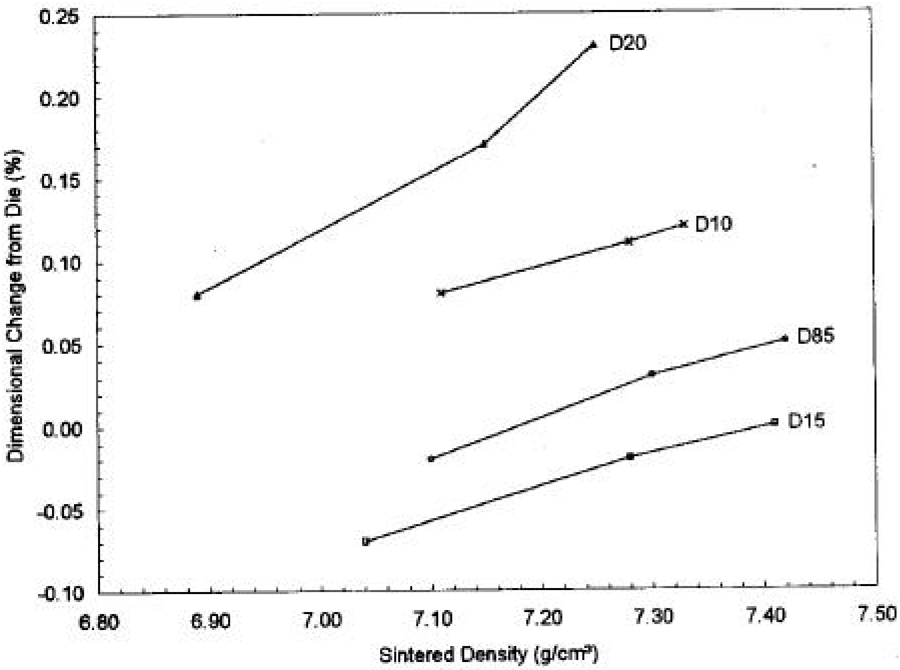


Figure 6: Dimensional Change from Die Size–2050°F (1120°C) Sinter

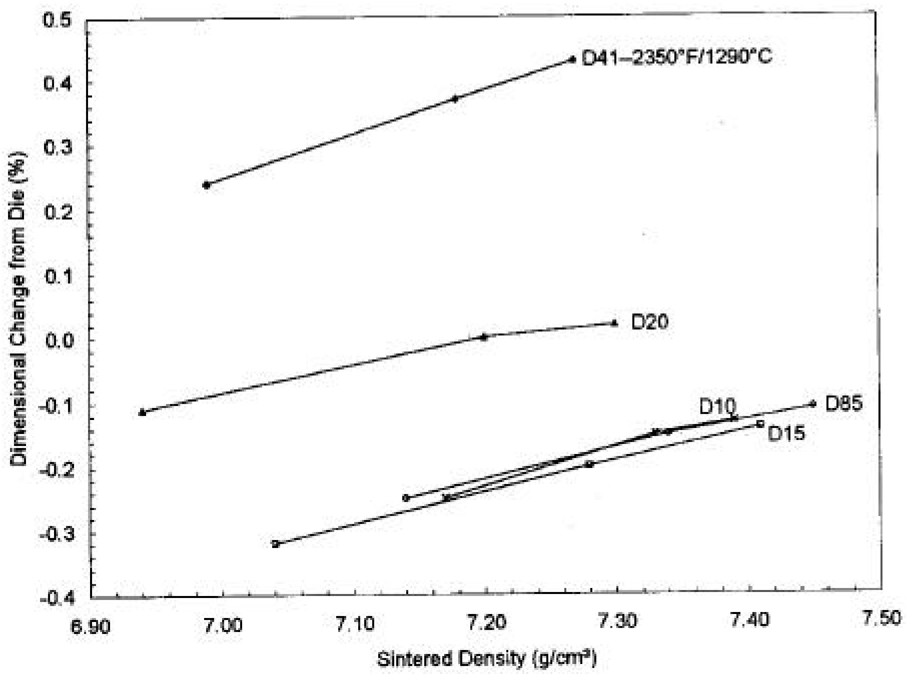


Figure 7: Dimensional Change from Die Size–2300°F (1260°C) Sinter

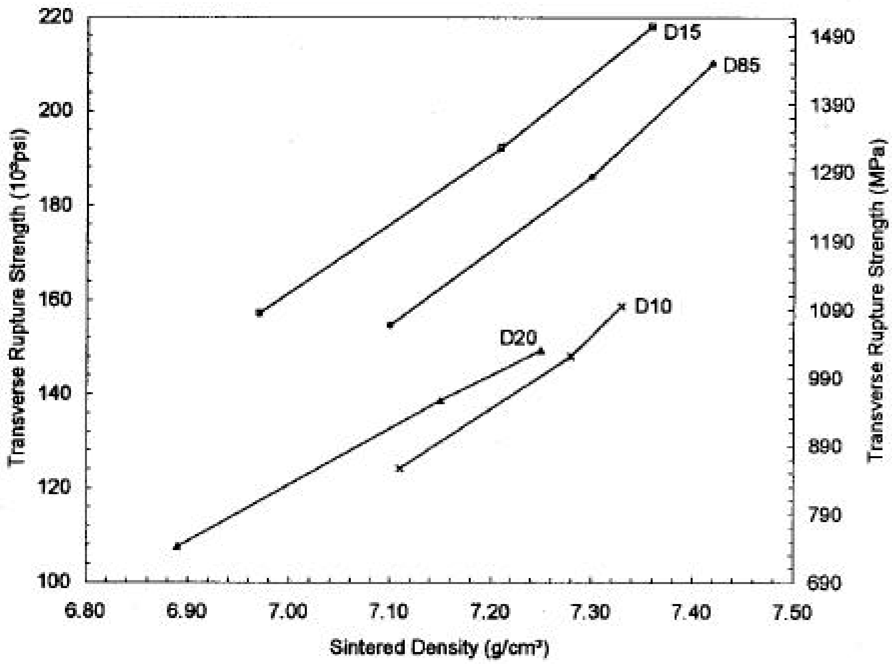


Figure 8: Transverse Rupture Strengths for Materials Sintered At 2050°F (1120°C)

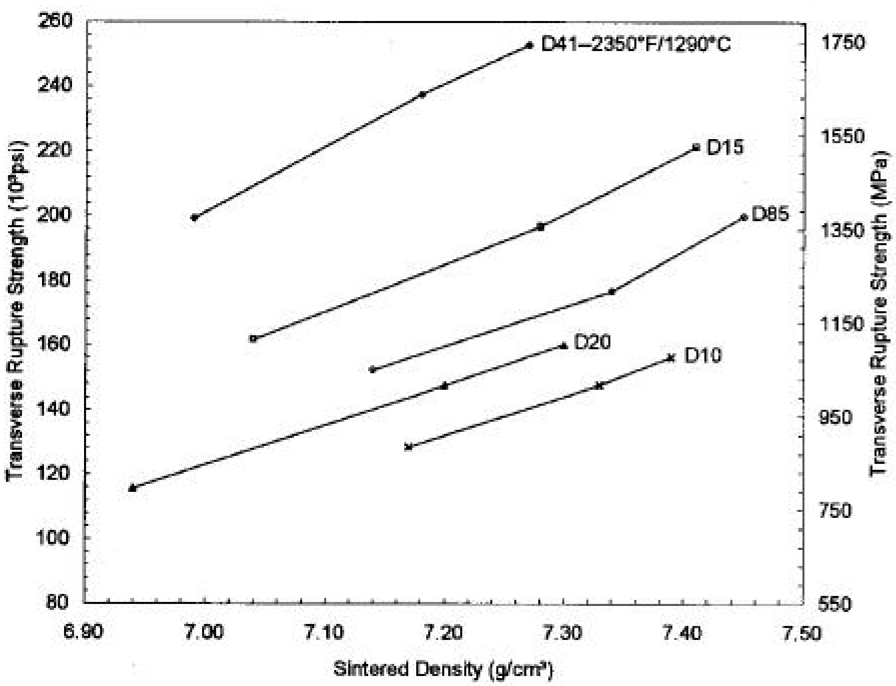


Figure 9: Transverse Rupture Strengths for Materials Sintered At 2300°F (1260°C)

Table VII: As Sintered Tensile Properties of the Materials Sintered at 2050°F (1120°C)

Material	Sintered Density (g/cm³)	0.2% Offset Yield Strength (10^3 psi)	0.2% Offset Yield Strength *(MPa)*	Ultimate Tensile Strength (10^3 psi)	Ultimate Tensile Strength *(MPa)*	Elg. (%)
D10	7.15	35.6	*246*	57.7	*398*	2.7
	7.32	39.1	*270*	65.4	*451*	3.2
	7.37	40.0	*267*	68.7	*457*	3.5
D85	7.17	60.0	*414*	76.5	*527*	1.4
	7.35	63.2	*436*	85.4	*589*	2.1
	7.44	65.3	*450*	92.3	*636*	2.8
D20	6.88	49.4	*341*	59.5	*410*	1.4
	7.12	53.5	*369*	66.0	*455*	1.8
	7.24	60.6	*418*	73.8	*509*	1.7
D15	7.05	68.1	*470*	88.0	*607*	1.1
	7.29	74.0	*510*	97.8	*674*	1.1
	7.40	77.9	*537*	105.4	*727*	1.3

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

Material	Sintered Density (g/cm³)	0.2% Offset Yield Strength (10^3 psi)	0.2% Offset Yield Strength *(MPa)*	Ultimate Tensile Strength (10^3 psi)	Ultimate Tensile Strength *(MPa)*	Elg. (%)
D10	7.22 7.38 7.43	39.2 41.4 44.3	*270* *285* *305*	65.8 74.0 79.8	*454* *510* *550*	4.0 4.5 5.2
D85	7.22 7.41 7.48	59.9 66.9 68.6	*413* *461* *473*	81.8 89.8 96.3	*564* *619* *664*	2.4 2.9 3.9
D20	6.98 7.20 7.31	53.3 56.5 57.9	*368* *390* *399*	66.6 74.9 81.2	*459* *516* *560*	1.6 2.1 2.9
D15	7.09 7.33 7.45	66.7 68.6 73.7	*460* *473* *508*	88.5 101.0 107.5	*610* *696* *741*	2.1 2.6 3.2
D41	7.03 7.24 7.32	88.0 99.9 107.3	*607* *689* *740*	114.3 130.4 139.4	*788* *899* *961*	1.5 1.8 2.0

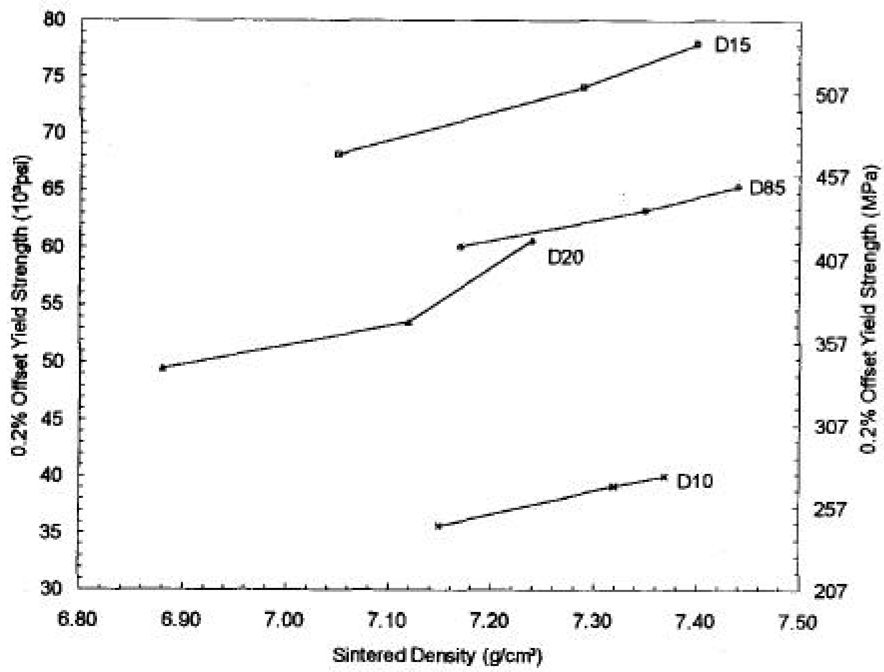


Figure 10: 0.2% Offset Yield Strengths for Materials Sintered at 2050°F (1120°C)

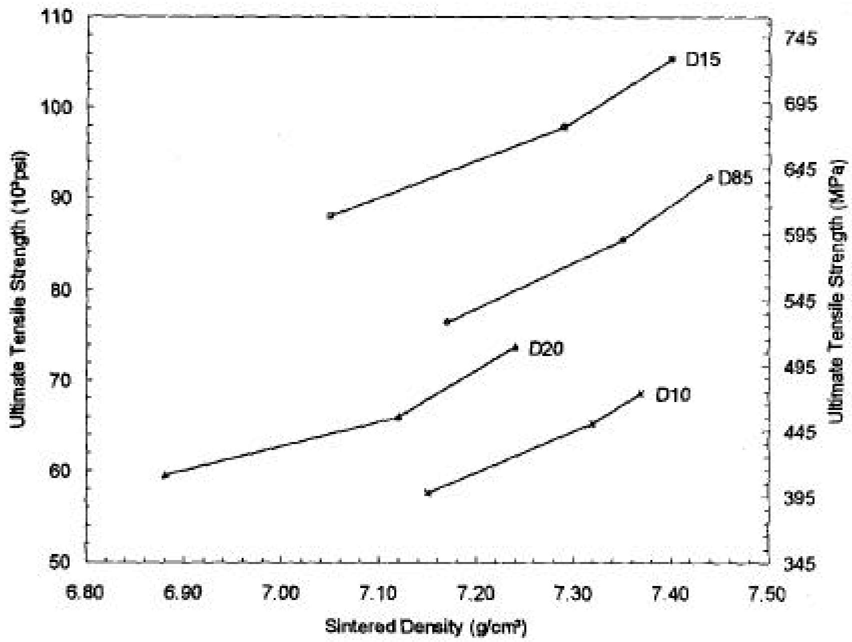


Figure 11: Ultimate Tensile Strengths for Materials Sintered at 2050°F (1120°C)

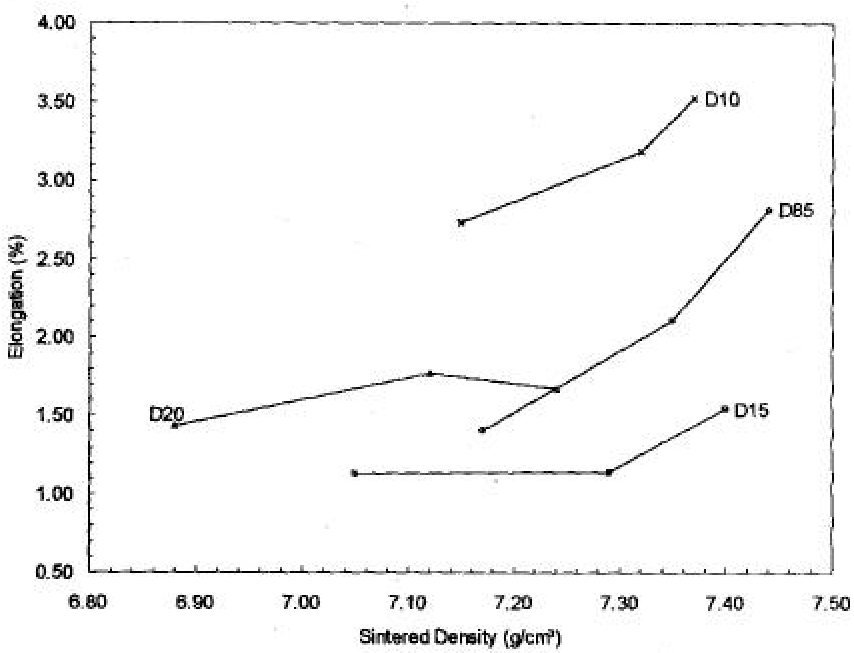


Figure 12: Tensile Elongations for Materials Sintered at 2050°F (1120°C)

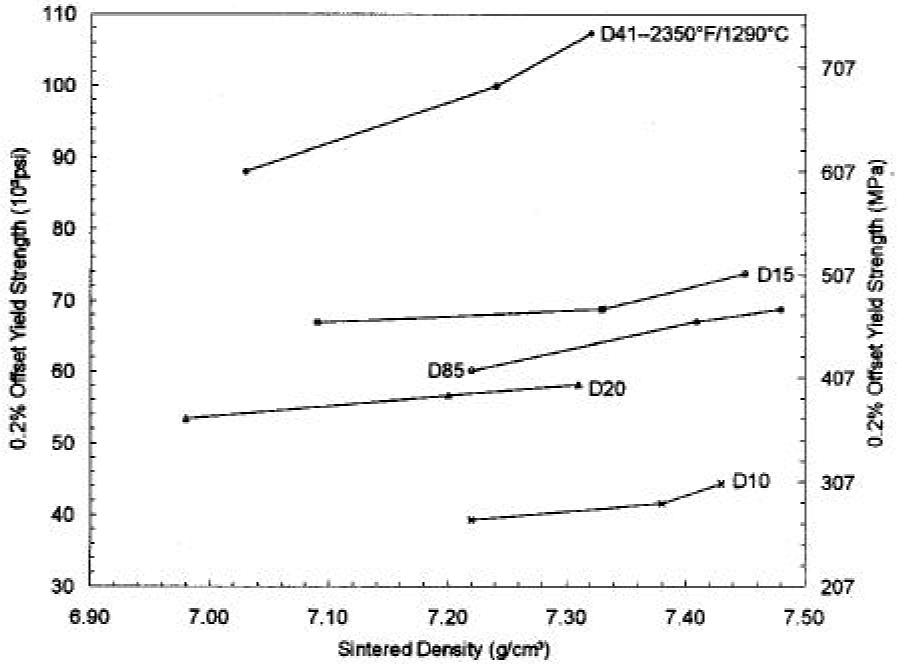


Figure 13: 0.2% Offset Yield Strengths for Materials Sintered at 2300°F (1260°C)

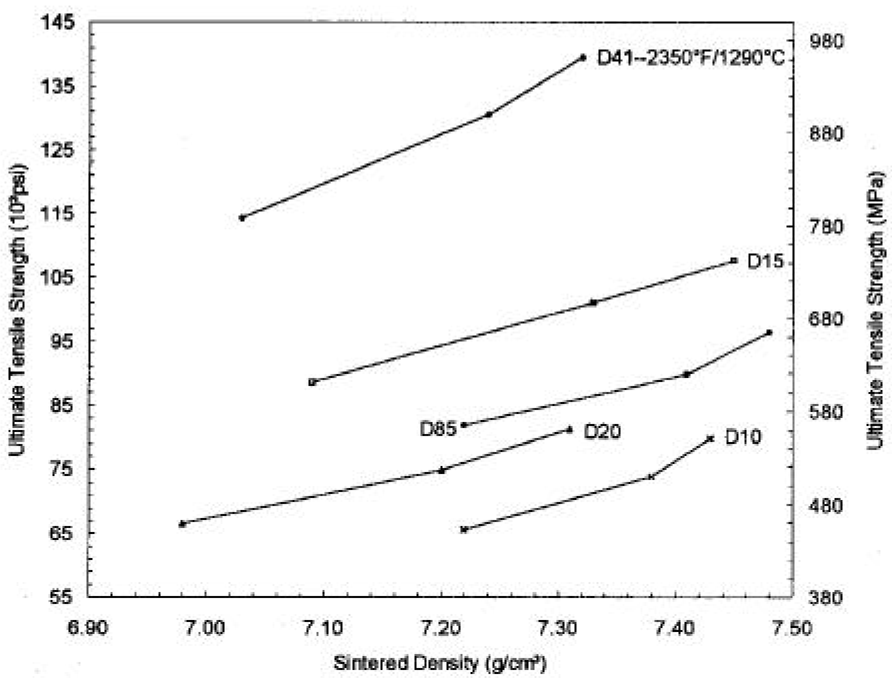


Figure 14: Ultimate Tensile Strengths for Materials Sintered at 2300°F (1260°C)

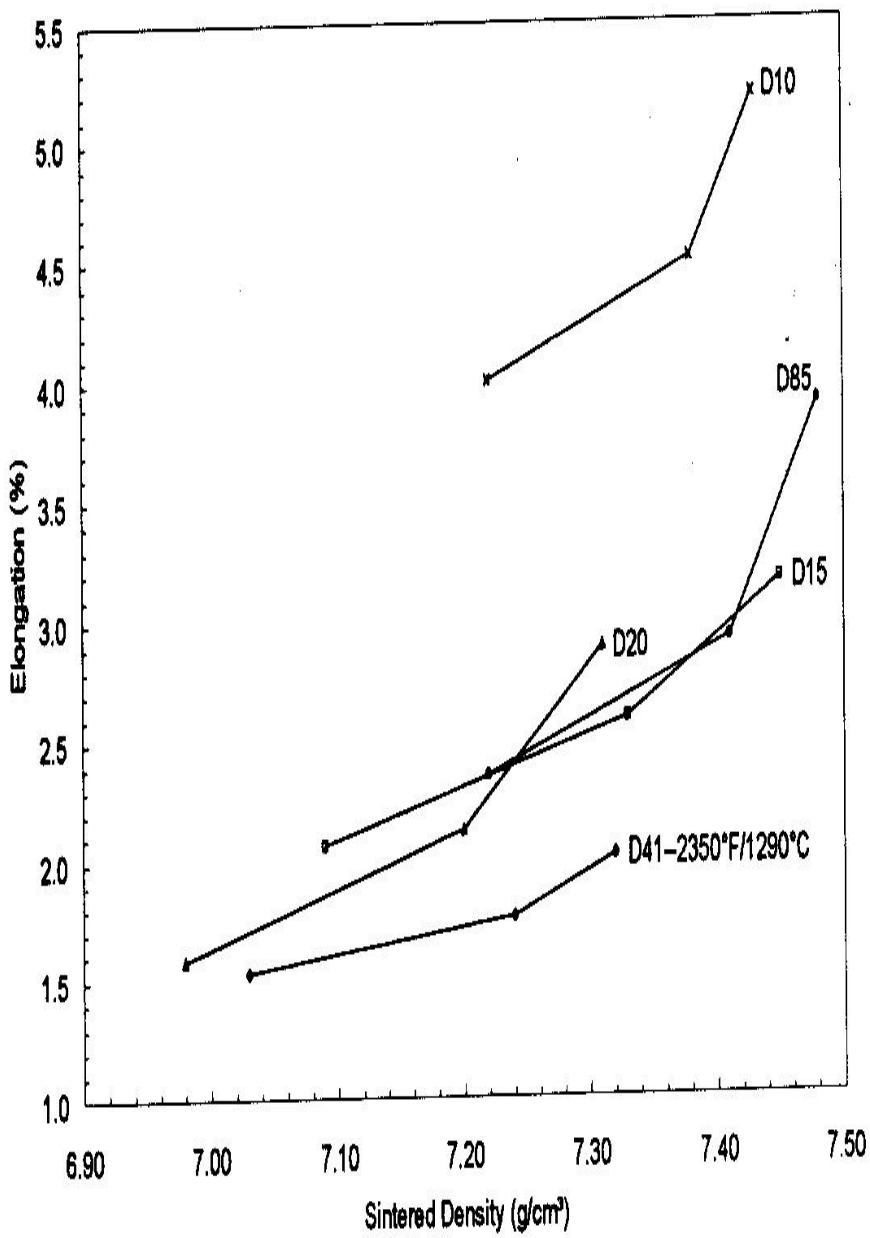


Figure 15: Tensile Elongations for Materials Sintered at 2300°F (1260°C)

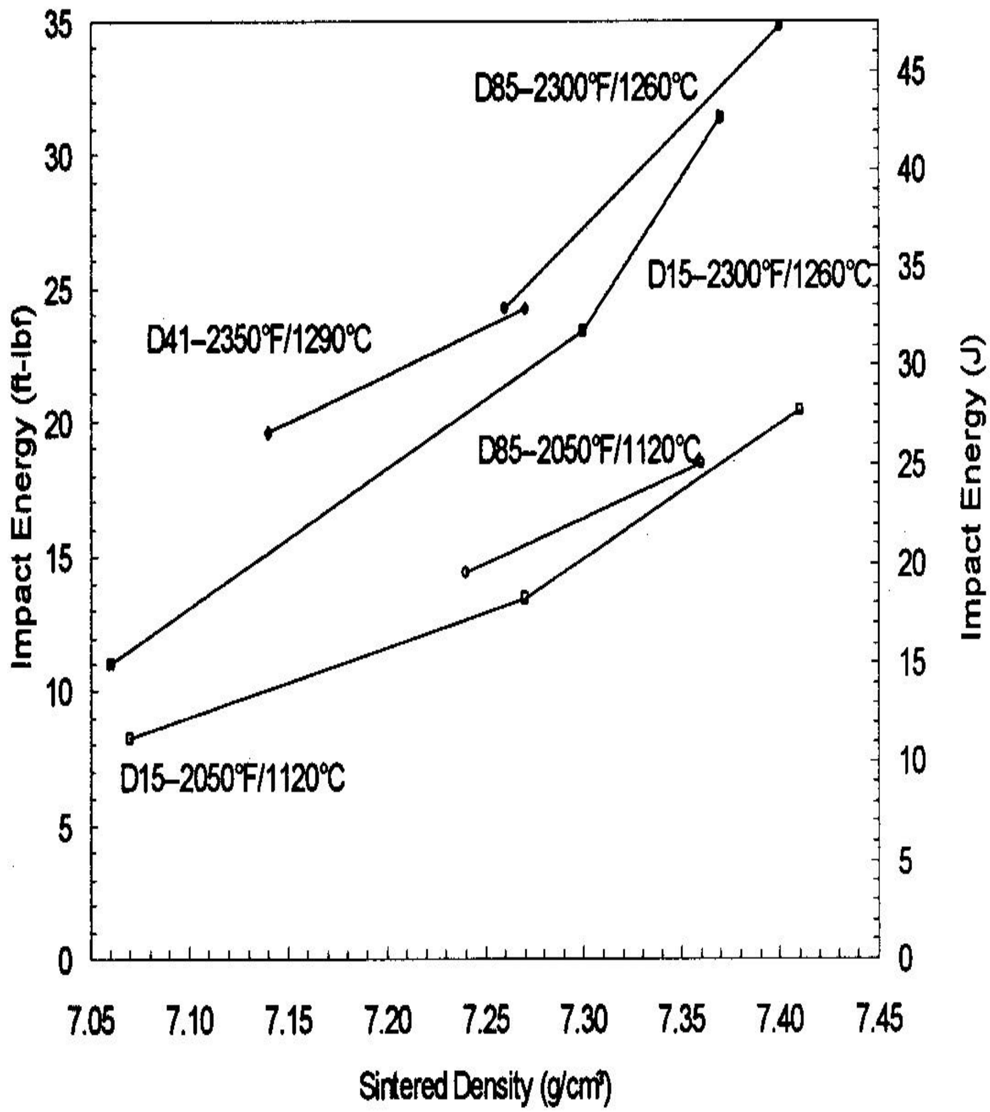


Figure 16: Impact Properties

Table VIII: Impact Properties

Material	Compaction Pressure (tsi/*MPa*)	2050°F(1120°C) Sinter Sintered Density (g/cm³)	2050°F(1120°C) Sinter Impact Energy (ft-lbf)	2050°F(1120°C) Sinter Impact Energy (*J*)	2300°F(1260°C) Sinter Sintered Density (g/cm³)	2300°F(1260°C) Sinter Impact Energy (ft-lbf)	2300°F(1260°C) Sinter Impact Energy (*J*)
D85	35/*483*	7.24	14.4	*19.5*	7.26	24.2	*32.8*
	45/*620*	7.36	18.8	*25.5*	7.40	34.8	*47.2*
D15	30/*415*	7.07	8.2	*11.1*	7.06	11.0	*14.9*
	40/*550*	7.27	13.4	*18.2*	7.30	23.4	*31.7*
	50/*690*	7.41	20.4	*27.7*	7.37	31.4	*42.6*
D41	35/*483*				7.14	19.6	*26.6*
	45/*620*				7.27	24.2	*32.8*

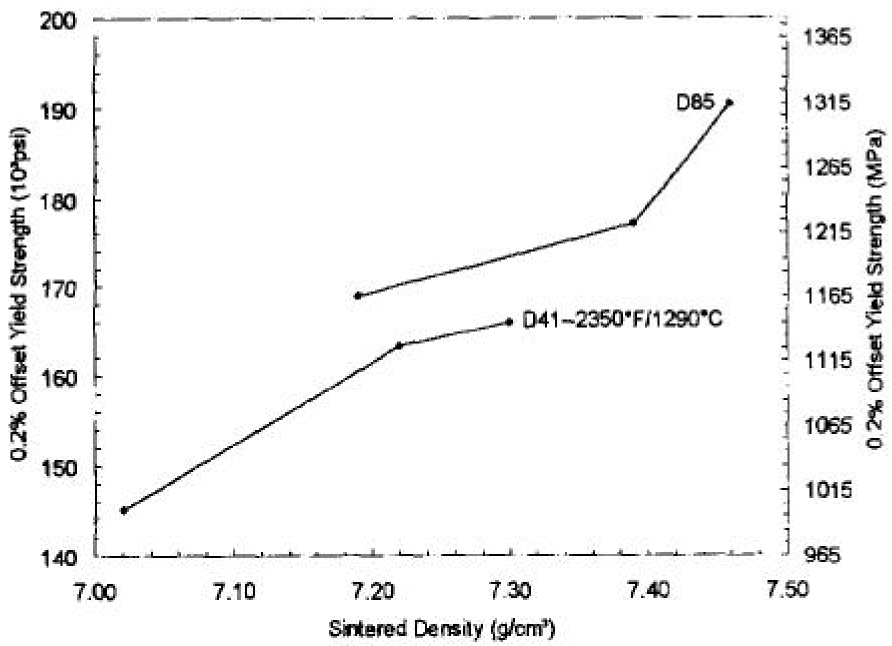


Figure 17: 0.2% Offset Yield Strengths for Heat Treated Materials

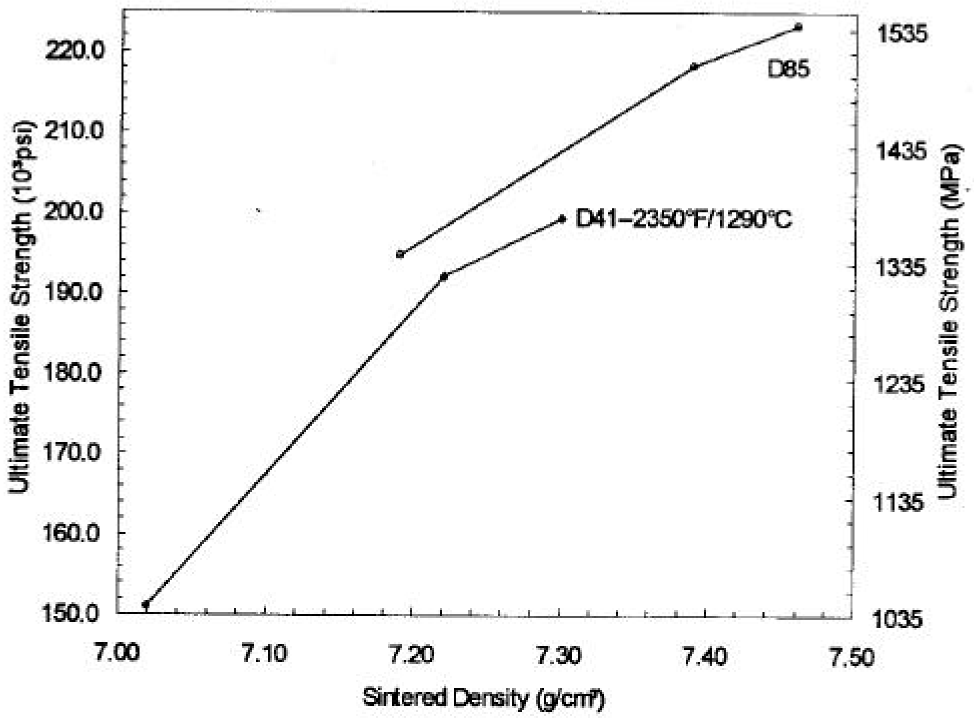


Figure 18: Ultimate Tensile Strength for Heat Treated Materials

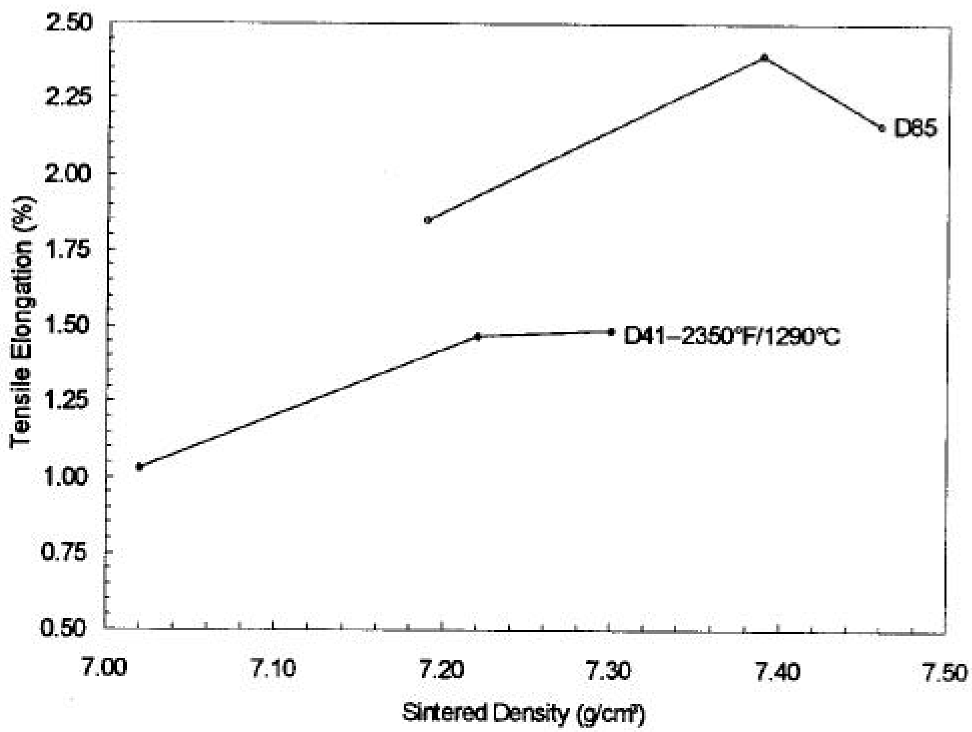


Figure 19: Tensile Elongation for Heat Treated Materials

Table IX: Heat Treated Properties for Materials D85 (2300°F/1260°C) and D41 (2350°F/1290°C)

Material	Sintered Density (g/cm^3)	0.2% Offset Yield Strength (10^3 psi)	0.2% Offset Yield Strength *(MPa)*	Ultimate Tensile Strength (10^3 psi)	Ultimate Tensile Strength *(MPa)*	Elongation (%)
D85	7.19	169.0	*1165.2*	194.7	*1342.4*	1.85
	7.39	177.2	*1221.8*	218.1	*1503.7*	2.39
	7.46	190.2	*1311.4*	223.2	*1538.9*	2.17
D41	7.07	145.1	*1000.4*	151.0	*1041.1*	1.03
	7.22	163.2	*1125.2*	192.0	*1323.8*	1.47
	7.30	166.0	*1144.5*	199.2	*1373.4*	1.43