HIGH DENSITY PROCESSING OF HIGH PERFORMANCE FERROUS MATERIALS

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Abstract

Density is an predominant factor in the performance of powder metallurgy components. Methods such as double press/double sinter, copper infiltration and powder forging have been employed to provide higher densities than traditional single press and sinter operations; however, their widespread use is constrained by cost and geometry considerations.

A commercially proven method for obtaining single compaction/single sinter densities in the 7.25 to 7.55 g/cm³ range by means of the patented ANCORDENSE™ technology is introduced. Conventional compaction pressures and sintering temperatures, typically not exceeding 50 tsi or 2300°F, respectively, are utilized. Resulting properties for several high performance materials are presented.

Test results indicate that the ANCORDENSE process is a cost effective method of providing high density parts with outstanding physical properties. The process is shown to be applicable to a wide variety of high performance materials. Additionally, significant improvements in green strength and ejection forces are realized.

Introduction

The effect of density on a wide variety of powder metallurgy part properties has been thoroughly documented [1,2]. Generally, as density is increased almost all properties, including strength [3,4] and magnetic performance [5], are improved. Traditionally, density has been increased by raising compaction pressures, elevating sintering temperatures, sintering enhancement additions, double press/double sinter processing, copper infiltrating and powder forging. Figure 1 indicates the density ranges that may be expected from these various powder metallurgy processes and Table I lists strengths and weaknesses associated with these processes. As illustrated in Figure 2, increasing the density of powder metallurgy components significantly increases part cost.

Table	1:	Strengths	and	Weaknesses	of	Various	Powder	Metallurgy	
Processes									

Process	Strengths	Weaknesses	
Conventional	Inexpensive	Limited Density	
Compaction		Capability	
	Wide Range of Part		
	Shapes Possible		
Double Press/Double	High Density	Expensive	
Sinter			
		Limited Part Shapes	
		Double Processing	
Copper Infiltration	Improved Performance	Expensive	
		Double Processing	
Powder Forging	Near to Full Density	Very Expensive	
		Complicated	
		Processing	

In addition to those processes listed in Figure 1, performance may be further improved by the use of sinter hardening alloy systems coupled with accelerated cooling. These materials obtain enhanced properties while eliminating a costly heat treating step. While these materials are capable of obtaining high hardness values in the as sintered state, the increased hardness makes secondary machining operations difficult if not impossible. The usage of sinter hardening systems can be greatly enhanced if the problems encountered in machining are addressed.

The inherent flexibility of the powder metallurgy process has led to significant growth during the last 10 years [6]. For the powder metallurgy market to realize full potential and compete with wrought products, a cost effective, flexible and robust manufacturing method for obtaining high densities is needed. The ANCORDENSE process is designed to meet these challenges.



Figure 1. Typical Density Ranges for Several Common Powder Metallurgy Processes



Figure 2. The Relative Cost and Density Ranges for Various Powder Metallurgy Processes

The ANCORDENSE process is a patent protected technology [2] that provides density increases in the range of 0.1 to 0.25 g/cm^3 over conventional processing performed at equivalent compaction pressures. The process also provides increased green strength and reduced die ejection forces.

The ANCORDENSE process can easily be adapted to utilize existing powder metallurgy processes and equipment and is applicable to all Ancorsteel® [3] high performance material systems.

Pore Free Density

Before discussing the ANCORDENSE process in detail, it would be useful to review the concept of pore free density. Pore free density is defined as the density of a green compact in which all interparticle porosity has been completely eliminated. Obtaining a totally pore free compact is essentially impossible in conventional compaction. A key factor in limiting maximum green density is the elastic nature of the materials involved. The elasticity results in green expansion after the compaction load is removed and the part is ejected from the die. Despite the fact that it is impossible to obtain pore free density, the concept provides an important benchmark to evaluate the effectiveness of the compaction process.

The pore free density of a premix can be calculated from the specific density and percentage of each of the constituents present in the premix. The density of several common ferrous powder metallurgy base materials and additives were measured using pycnometry and the results are listed in Table II. The density of ferrous base materials is approximately 7.85 g/cm³. The addition of materials with a specific density higher than the base iron (copper and nickel) slightly increases the pore free density than the base iron (lubricants and graphite) lowers the pore free density. Figure 3 indicates the effect of several common premix additives on pore free density. This figure clearly illustrates that increasing the percentage of lubricant or graphite in the premix will lower the pore free density.

In previous powder metallurgical systems, approaching the pore free density was rather difficult. With the new ANCORDENSE process, achieving near pore free density is now possible. The new lubricant system designed for ANCORDENSE is specifically engineered to operate successfully at lower lubricant levels than have been customary which servers to increase the green density that can be realized. Additionally, to take full advantage of the ANCORDENSE process, graphite additions can be minimized by utilizing a careful Systems approach to alloy selection to insure that the best combination of density and performance is achieved.

Material	Specific Density (g/cm ³)
Ancorsteel 1000B	7.841
Ancorsteel 4600V	7.844
Distaloy 4800A	7.896
-100 mesh Cu	8.047
Inco Ni 123	8.846
Graphite	2.295
Acrawax	1.003
Zn Stearate	1.139
ANCORDENSE Lube	1.030

Table II: The Specific Density of Several Common Powder Metallurgy Base Materials and Additives as Measured by Pycnometry

The ANCORDENSE Process

The ANCORDENSE process is shown schematically in Figure 4. The process utilizes traditional powder metallurgy compaction

equipment but requires that the powder and the die be heated to a temperature in the range of 265°F to 310°F. It is recommended that the process temperature be controlled to maximum variation of +/-5°F. The ANCORDENSE process is a derivative of the ANCORBOND® [4] technology [7] and a highly engineered lubricant system to optimize performance within this temperature range.

With the ANCORDENSE process (Figure 4), the press ready powder is transferred to a storage hopper on the press and then fed into a heating device prior to transfer into the shuttle. Various heating systems can be used to heat the powder uniformly to the specified temperature range. The heated powder is then fed into a storage container that provides powder to the shuttle. Following heating, the powder is fed into the die and compaction continues in the same manner as for a traditional powder metallurgy part. The part can then be sintered in a customary fashion to provide a high density/high performance powder metallurgy part.

Typically, both the container and shuttle are heated and insulated to prevent heat loss and to insure uniform temperature of the powder. The die is also heated to the specified temperature range. The die can be heated using resistance type heaters with a thermocouple embedded within the die to provide temperature control. It is important that all tool members be at a uniform temperature to avoid a tool collision resulting from thermal mismatch. Therefore, the top punch assembly and large core rode assemblies should also be heated. Bottom punches are typically not directly heated as the punches remain in contact with the die and are heated by conduction from the die. Care must be taken to insure that heat transfer from the die into the press frame is minimized to prevent any possible press damage. If needed, simple insulation techniques can be utilized to limit the heat transfer from the die to the platens.



Figure 3: Individual Effect of Common Premix Additions of Pore Free Density



Figure 4: The ANCORDENSE Process

Several considerations should be made in terms of tool design. Because the die operates at temperatures above 265°F, care should be taken in utilizing a shrink fit carbide insert die. Adequate shrink must be applied to the die to insure that the insert remains under sufficient compressive load. This is not a problem with steel dies. Additionally, the ANCORDENSE process provides much higher densities than were before possible. Obtaining these higher densities increases the load on the die and it is suggested that the die be sufficiently rigid to handle the increased loads.

Property Enhancements

High Performance Structural Materials

In order to evaluate the technology, two premixes composed of Distaloy 4800A with 0.6 wt.% graphite were prepared. One sample contained 0.6 wt.% Acrawax while the second sample contained 0.6wt. % of the new ANCORDENSE lubricant. The materials were compacted into standard test specimens using production compaction equipment. The Acrawax containing premix was compacted at ambient conditions while the ANCORDENSE premix was compacted at 290°F. Green and sintered properties were then analyzed according to standard test methods. Green and sintered (2050°F for 30 minutes at temperature in 25% $N_2/75\%$ H₂ atmosphere) densities were measured on TRS test specimens and are presented in Figure 5. The data indicates that a density increase of from 0.15 to 0.25 g/cm³ is realized in both the green and sintered state for a given compaction pressure.

Green strength results are presented in Figure 6. The plot is made on a density basis rather than comparing compaction pressures to indicate the clear superiority of the ANCORDENSE process. Distaloy 4800A possesses an inherently high green strength, even utilizing conventional lubricants and compaction techniques; however, the ANCORDENSE process still produces a green strength enhancement of over 50% to approximately 4000 psi. In all systems tested to date, the ANCORDENSE process has maintained a green strength level of about 3000 to 5000 psi, independent of the Ancorsteel alloy system. This advantage may allow automated handling of delicate parts while minimizing the possibility of green part damage.

Green expansion and dimensional change characteristics observed in the ANCORDENSE and conventional processes are shown in Figure 7. The graph indicates very little difference in dimensional change characteristics between the two processes up to densities of around 7.2 g/cm³. Above 7.2 g/cm³, ANCORDENSE processed samples exhibited greater green and sintered dimensional change. The greater dimensional change, observed at density levels only obtained with the ANCORDENSE process, is the result of the greater spring back of the compact as it approaches pore free density. As the overall density of the compact nears the pore free density, there is more efficient transfer of compaction energy through the compact and the density distribution within the compact becomes more uniform. The more uniform green density should lead to a more uniform sintered dimensional change within all sections of the part. Because of the high green densities achieved, the tooling design is critical due to of the high stresses experienced within the die.



Figure 5: Green and Sintered Density for ANCORDENSE and Conventionally Compacted Premixes of Distaloy 4800A+0.6% Lubricant (Sintered at 2050°F for 30 minutes in a 75% H₂/25% N₂ atmosphere)



Figure 6: Green Strength of Distaloy 4800+0.6% Graphite+0.6% Lubricant Compacted Conventionally and with the ANCORDENSE Process



Figure 7: Dimensional Change From Die Size for Distaloy 4800A+0.6% Graphite+0.6% Lubricant Compacted Conventionally and with the ANCORDENSE Process (Sintered at 2050°F for 30 minutes in a 75% $H_2/25\%$ N₂ atmosphere)



Figure 8: Transverse Rupture Strength for ANCORDENSE and Conventionally Processed Premixes of Distaloy 4800+0.6% Graphite +0.6% Lubricant (Sintered at 2050°F for 30 minutes in a 75% $H_2/25\%$ N₂ atmosphere)

Transverse rupture strength results obtained are shown in Figure 8. As expected, the increase in density as a result of the ANCORDENSE process resulted in significantly increased strength. Experience to date has indicated that at a minimum, equivalent strengths are obtained from the ANCORDENSE process and in a number of cases the strength is higher than normally processed materials at the same sintered density.

Ejection loads were measured for the two processes during compaction of a 0.4"XO.4"X2.5" test specimen in a fully instrumented mechanical press. The data for specimens compacted at 50 tsi are presented in Figure 9. The highest point on each curve represents the peak force needed to initiate movement of the part from the die, while the remaining points are the forces measured while the part was sliding from the die cavity. The ANCORDENSE process exhibited significantly lower ejection forces relative to an Acrawax containing premix. Considering that the ANCORDENSE sample had a green density improvement of over 0.1 g/cm³ over that of the conventionally prepared sample, the lower ejection forces are even more significant. The lower ejection forces should promote better die life and allow compaction of more complex geometries. However, if the ejection forces obtained during conventional processing with Acrawax prove to be acceptable, the possibility exists to run the ANCORDENSE process at a reduced lubricant level. As each reduction in lubricant level of 0.1 wt.% allows the pore free density to increase about 0.05 g/cm³, additional increases in green density are possible.

Magnetic Materials

Samples of Ancorsteel 45P were prepared and compacted conventionally with 0.6% Acrawax and prepared and compacted utilizing the ANCORDENSE process. Both materials were sintered at 2300°F in 100% H_2 for 30 minutes. Physical properties were determined on the ANCORDENSE material and are listed in Table III. These data make the advantages of higher f density obvious The extremely high ductility values that result from high density processing are most noteworthy

Table III Physical Properties of ANCORDENSE Processed Ancorsteel 45P

Compaction Pressure (tsi)	Green Density (g/cm ³)	Sintered Density (g/cm ³)	TRS (psi)	0.2% Yield Strength (psi x 10 ³)	Ultimate Tensile Strength (psi x 10 ³)	Elong. (%)	Hard. (HRb)
30	7.06	7.25	NB*	33.6	52.3	16.4	53
40	7.28	7.46	NB*	36.1	57.3	20.4	61
50	7.39	7.57	NB*	39.0	61.7	25.2	67

*NB = No Break

Toroids were also compacted and sintered at 2300°F for 30 minutes in a hydrogen atmosphere Magnetic hystersis curves were generated at drive fields of 15 and 30 Oe. The data indicates that the large improvements in sintered density obtained with the ANCORDENSE enhance magnetic performance significantly. Additionally, the ANCORDENSE process yields slightly improved magnetic properties (Figure 10) for a given density when compared with conventional processing. The large increase in density realized from the ANCORDENSE process also results in an increase in maximum induction (B_{max}, Figure 11) indicating an increase in saturation induction. This improvement is especially useful in applications where powder metallurgy parts are utilized as flux carriers for a permanent magnet. In many of these cases, the permanent magnet easily saturates the powder metallurgy part and the increase in density allows smaller components to be utilized.



Figure 9: Ejection Stresses for ANCORDENSE and Conventionally Processed Premixes of Distaloy 4800+0.6% Graphite+0.6% lubricant (0.4"X0.4"X2.5" bar)



Figure 10: Magnetic Properties for ANCORDENSE and Conventionally Processed Ancorsteel 45P (Sintered at 2300°F for 30 minutes in a 100% H₂ atmosphere)



Figure 11: The effect of Density of Ancorsteel 45P on Maximum Induction Measured at 30 Oe(Sintered at 2300°F for 30 minutes in a 100% H_2 atmosphere)



Figure 12: Green Strength for Several Premix Compositions Compacted Utilizing ANCORDENSE and Conventional Techniques

GREEN STRENGTH AND MACHINING

The importance of green strength to powder metallurgy parts is obvious. The need to move green parts from the press to sintering furnaces provides significant opportunity for part chipping and cracking. Automated handling of green parts, although cost effective and efficient, represents another opportunity for inducing green cracks. In the worst case, green damage may go undetected until sintering and other secondary operations are completed, thus adding significantly to part production costs.

As mentioned previously, machining of high performance powder metallurgy alloys presents significant challenges. Sinter hardened systems can provide excellent physical properties and apparent hardness in excess of Rockwell Rc 20 but the increased hardness makes secondary machining operations difficult if not impossible.

The ANCORDENSE process has been shown to provide extremely high green strength. It is interesting to note that improved green

strength can be obtained even at lower compaction pressures and density levels (Figure 6). Figure 12 indicates the level of improvement that may be expected from several additional compositions. This improved strength should help minimize green scrap problems encountered with some parts.

In order to investigate the possibility of green machining, blanks (3.5" long by 0.45") were prepared from Distaloy 4800A with 0.5% graphite and 0.6% lubricant utilizing the ANCORDENSE process. These blanks were then machined in the green state utilizing standard machine tools. In all cases, the machining was performed with no lubricant. Rather than generating traditional machining swarf, the operation resulted in powder being removed from the bar. Clamping of the work piece was done at significantly lower pressures to prevent cracking in the green part. Tool loads were not measured but are expected to be only a small fraction of what might be expected in machining a sintered part.

The possibilities of drilling and tapping a hole by green machining were evaluated. A hole was drilled in the blank perpendicular to the pressing direction on a drill press. Threads were then tapped in the hole with a hand tap. The bar was then sintered at 2050°F for 30 minutes in a 75% $H_2/25$ % N_2 atmosphere for 30 minutes at temperature. A bolt was then easily threaded into the hole without any further operations. The machined part is pictured in Figure 13a.

A second part was chucked in a metal lathe and turned round in the center utilizing a standard parting tool. The resulting surface finish of the machined section was excellent (Figure 13b). The major difficulty encountered in this trial was occurrence of chipping at the corner of the bar in the transition region between the round center and the square ends. It was discovered that utilizing higher work piece rotational speeds (>1000 RPM) eliminated the chipping.

Finally, a series of grooves were milled into the blank (Figure 13c). The test blank was clamped in a milling machine and 0.2" grooves were cut 0.2" into the surface of the bar in a single pass. The resulting machined surface appeared to be excellent. The only problem encountered was when the end mill broke through the opposite side of the part where some chipping occurred. This problem was easily rectified by starting the groove from both sides of the bar and finishing in the middle.

Lower Compaction Pressures

In order to investigate the effect of the ANCORDENSE process at low compaction pressures, an ANCORDENSE premix was prepared from Ancorsteel 1000B P/F with 2% Cu, 0.5% MnS, 0.7% graphite and 0.75% lubricant. Green strength bars were compacted from 10 tsi to 40 tsi. The green density and green strength results are reported in Figure 14. The results indicate that even at compaction pressures as low as 20 tsi, green densities greater than 6.7 g/cm^3 and green strengths greater than 2500 psi are obtained. These results suggest two possibilities. If the lower densities provide acceptable part performance, significantly larger part areas can be compacted on a given size press. This enables larger parts to be manufactured on existing equipment thus expanding the market opportunities for powder metallurgy. Secondly, compacting much more fragile parts to moderate density may be possible. The combination of utilizing fairly low compaction pressures to obtain acceptable density levels with potentially delicate tooling and the resulting higher part green strength may provide new part opportunities.

Preliminary Part Production Experience

Hoeganaes Corporation, in conjunction with Cincinnati Incorporated and several parts manufacturers, has evaluated the potential of the ANCORDENSE process on a series of actual powder metallurgy components. The parts evaluated ranged in size from about

100 grams to in excess of 1000 grams (Figure 15). Shape complexity varied from simple, single level spur gears to multilevel internal/external tooth configurations and helical gears. In all cases, existing powder metallurgy tooling was modified to incorporate resistance heaters capable of maintaining the tools at 300°F. Production press rates of 10 parts per minute and greater were maintained for these trials.

The results of these actual part trials indicate that the ANCORDENSE process is capable of producing powder metallurgy parts with sintered densities of up to 7.45 g/cm³. All parts tested obtained either an increase in sintered density in the range of 0.1 g/cm³ to 0.25 g/cm³ for a given compaction pressure or a decrease of at least 10 tsi to obtain a given density. The part trials



Figure 13: Examples of Green Machined ANCORDENSE Samples



Pressures (Ancorsteel 1000B P/F+2.0% Cu+0.5% MnS+0.7% Graphite+0.75% Lubricant)



Figure 15: Examples of Powder Metallurgy Parts Produced in Actual Part Trials with ANCORDENSE

also confirmed that the high green strength, more uniform part density and reduced ejection forces observed in laboratory studies were successfully translated into a production environment. Die filling and flow characteristics of the ANCORDENSE powder met expectations for conventionally blended premixes.

Conclusions

This paper introduces the new ANCORDENSE process. The following can be concluded about the ANCORDENSE process:

1)The new ANCORDENSE process can provide densities of up to 7.45 g/cm^3 from a single compaction process. Density and performance levels previously achieved through costly additional processes are now easily obtained by single compaction.

2)Higher density levels can be achieved in parts with complex shapes that are unsuitable for double pressing/double sintering.

3) The ANCORDENSE process provides significantly higher green strength than conventionally pressed parts. The increased green

strength will limit green damage during handling.

4) The increased green strength of the parts presents opportunities to machine green parts. This possibility may be critical in the use of sinter hardening alloys where machining after sintering may prove impractical.

5) Parts made from the ANCORDENSE process show improved part ejection characteristics.

6) Higher densities can be achieved at lower compaction pressures. This may overcome a press capacity limitation.

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NOTES

1. ANCORDENSE is a trademark of the Hoeganaes Corporation

2. The information included in this paper is covered by one or more of the following U.S. Patents: 4090868,4093449, 4503025,4126452, 4190441,4483905, 4676831,4834820, 5060001,5080712, 5108493,5154881, 5198137,5217683, 5225459,5256155 and 5268140. Additional U. S. Patents are pending and various foreign patent coverage exists or is pending.

3. Ancorsteel is a registered trademark of the Hoeganaes Corporation.

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