Double Press Double Sinter Alternatives for High Density Applications

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Abstract

The objective of this study was to determine the capabilities of a single press operation using a heated die and specialized lubricant to match the properties of a double pressed double sintered (DPDS) component. Parameters studied included the amount of lubricant, the compacting temperature and the compaction pressure. Mechanical properties of the single press operation with the developmental lubricant were compared to DPDS. Surface finish of the parts compacted to high densities on a 150 ton hydraulic press (with the special lubricant) were also compared on a VVT Stator. The porosity of the single press/developmental lubricant versus the DPDS method was examined using metallography which was used to explain differences in mechanical properties between the two methods.

Introduction

The largest driving force in strength improvement for a PM part is increasing part density. Much research activity is dedicated to improving the compressibility of powders and blends so as to increase the achievable density upon compaction. The double press-double sinter (DPDS) method arose from the need to reach ever higher densities while working with the physical constraints of compacting metal powder and has been an established practice for many years. Parts are first compacted with admixed lubricant to a height slightly taller than the final specification. These parts are de-lubricated in a furnace and pressed once more. Finally, parts are sintered and undergo any necessary finishing operations.

A key drawback of the DPDS method is the cost associated with additional steps relative to a traditionally pressed part (single press-single sinter). These costs arise from the need for more equipment and the extended time to manufacture the parts. Concerning equipment, an additional die is needed to repress the parts and if parts are to be produced continuously the process requires an additional press and sintering furnace. The study was designed to determine whether a single press method with an experimentally developed lubricant in combination with warm compaction can compete with the DPDS. An additional goal of the lubricant would be to provide adequate lubricity with lower levels of lubricant so that the density of the compacted part would be as high as possible. The lubricant adjustments created an increased pore-free density and with warm compaction aided the densification.

DPDS is a multiple step process with two compaction steps. Initial compaction of a DPDS part is identical to that of a traditionally pressed part. The pressure used is determined by the final requirements and how much density yield is expected in the second press. Since the die used for the second press does not require multiple stages and creates significantly less material movement there is less opportunity for tool breakage during the second press, and greater pressure can be applied in this compacting step relative to the first. The second press requires a die cavity slightly larger than the first to accommodate the part's green expansion and any dimensional change that may occur during the first sinter. Lubricant must be applied to the walls of the die to allow for part ejection after compaction.

Between compaction steps it is essential to remove the internal lubricant that would otherwise hinder the second compaction step. This must be done in a furnace that achieves lubricant burn-off but does not drive the graphite into solution. If the graphite dissolves into the iron the resulting part will not be malleable for the second compaction step, hindering densification. This study did not examine the effect of varying temperatures of the sintering between compaction steps, but this is known to vary the final part density. The temperature used was based on past experience with the material system used.

Development of Lubricant

Key to achieving a density in a single compaction step is to choose a lubricant that would have enough lubricity to allow for low levels of lubricant to be utilized in the premix. It has been well documented that if the levels of lubricant can be reduced the green density of the compact can be increased due to the increase in pore free density [1]. Pore free density is defined as the density of a green compact if all the porosity could be removed and is dependent on the density and the percentage of each premix addition. It has been determined industrially, that at about 98% of the theoretical density, parts that are compacted start to experience lamination cracks. It is widely accepted that 98% of the pore free density is the maximum achievable density in a PM part. Figure 1 shows the effect of level of lubricant on pore free density of a mix as a function of graphite and lubricant content of the mix. Both the lubricant and graphite have lower densities (~1.0 and 2.3 g/cm^3) than the iron and therefore lower the overall density of the mixture. Figure 1 shows that at 0.60 w/o graphite a premix will have a pore free density (98%) of around 7.21 g/cm^3 with 0.75 w/o lubricant and a density of 7.38 g cm^3 with a lubricant content of 0.40%.

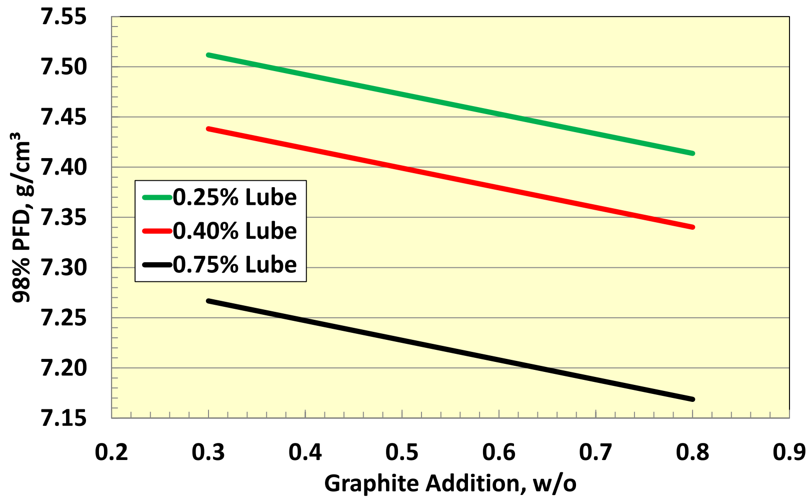

Figure 1: Effect of lubricant percentage at various graphite levels on the pore free density. (Courtesy of Fran Hanjeko).

In general, when the lubricant level is decreased the green density increases, but the lower lubricant levels can increase the ejection forces necessary to eject the part from the die. Typical measurements of ejection forces are the initial ejection pressure (strip) and pressure applied as the bar is exiting the die (slide) [2]. Figure 2 shows the effect of reducing the amount of lubricant on the ejection characteristics (strip and slide) in a FLN2-4405 premix. At higher levels of lubricant (0.75 w/o) the strip and slide pressures are relatively low. At 0.40 w/o lubricant, both pressures increase and in fact at the 0.40 w/o level the pressure to keep the part ejecting from the die (slide) is higher than the initial pressure to start the bar ejecting (strip pressure).

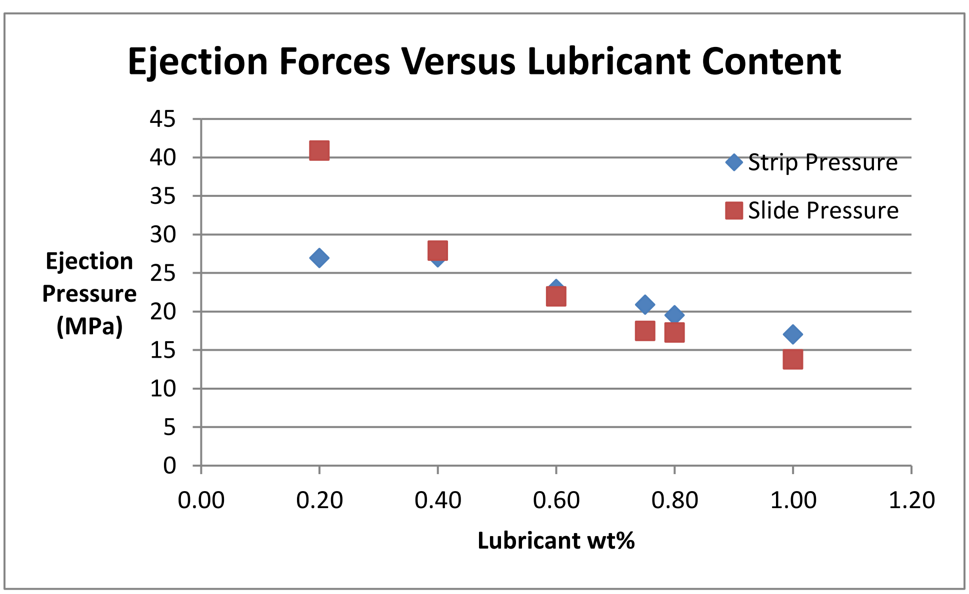

Figure 2: Effect of lubricant content on ejection forces.

It is desirable to have the strip and slide pressures as low as possible. Therefore the goal is to have a lubricant that can be used at low levels (to maximize green density) but achieve close to the same ejection characteristics as standard lubricant levels (for example 0.75 w/o ethylene bis-stearamide (EBS)).

Another technique used to increase the green density of compacted parts is to utilize warm compaction. Warm compaction uses heating of either the powder or the die and in some cases both. Due to the difficulties in maintaining proper powder flow from the hopper to the die cavity of the press when utilizing powder heating, this study utilized only warm die compaction. This is normally performed at temperatures ranging from 63 ºC to 107 ºC, where the dies are heated using either heated bands around the outside of the die or heater cartridges built into the die body. In the case of warm die compaction, the lubricant (and any of the components there in) must not break down in the temperature range described. The lubricant must maintain lubricity at the increased die temperatures and not have a large liquid component that causes loose powder from the die cavity to adhere to the surface of the part. This can cause clearance problems between the tool and the punch and also lead to poor surface quality of the compacted part.

A final performance aspect of the lubricant is its ability to perform at higher compaction tonnages in order to reach high densities. Thus the lubricant must perform at compaction pressures ranging from of 690 to 827 MPa. In this case, the lubricant must perform well under high stresses and must still be able to flow to the die wall for maximum lubrication.

The objective of this study was to investigate a lubricant (for premixes) that could be used with warm compaction (63 ºC to 107 ºC) at low levels with high compaction tonnages, to achieve high green density. The sintered and mechanical properties of test specimens made by this method were then compared to parts (of the same premix composition) that were double pressed and double sintered as a benchmark for density and mechanical properties.

Experimental Procedure

All experiments were performed using an FLN2-4405 composition made with Hoeganaes' Ancorsteel® 85 HP iron powder, 0.6 w/o 3203 Asbury Graphite and 2 w/o Inco 123 Nickel. Some experiments were repeated using other compositions for purposes of comparison. Parts were compacted with lubricant contents of 0.75 w/o Acrawax C (Ethylene Bis-Stearamide or EBS) from Lonza or 0.40 w/o of Hoeganaes' proprietary AncorLube®. Initially mechanical properties were compared between DPDS parts pressed with 0.75 w/o EBS to single press single sinter (SPSS) parts containing 0.40 w/o AncorLube. Parts using EBS were compacted at room temperature while parts using AncorLube were compacted in a heated die at 107°C. Mixes were compacted into transverse rupture strength (TRS), unnotched charpy impact, and conventional flat tensile bars in accordance with MPIF standards 41, 59, and 10, respectively [3]. For parts undergoing DPDS, the pre-sintering was performed at a temperature of 870°C for 20 minutes under an atmosphere of 10 v/o hydrogen and 90 v/o nitrogen. Repressing was achieved by grinding bars until they could fit back in the original die. Grinding for the flat surfaced TRS and charpy impact bars was performed using a planar grinding wheel; the dogbone tensile bars were ground using a Dremel. In order to eject the repressed bars they were coated with die wall lubricant prior to insertion into the die. This experiment used MolyKote Z powder as the die wall lubricant. Initial compaction was done at a variety of pressures while all repressing used a compaction pressure of 827 MPa (60 tsi). Repressed bars and single pressed bars were sintered under identical conditions; 20 minutes at 1120°C under an atmosphere of 10 v/o hydrogen and 90 v/o nitrogen.

The lubricant of choice for the single press operation was tested for its ability to operate under the desired conditions of high temperature and pressure. The lubricity was evaluated by measuring the force required for ejection of green strength bars.

Results & Discussion

The lubricant, AncorLube, chosen for this study was previously described [2,4]. In past studies, the development of the lubricant for use in premixes as regard to premix flow properties and compaction performance was described. Past work utilized typical levels of lubricants found in premixes (0.60 to 0.75 w/o) and compaction characteristics were described at temperatures ranging from room temperature to 63°C. In order to compete with DPDS, the lubricant had to be investigated at lower lubricant content and higher compaction pressures and temperatures. A baseline performance for ejection characteristics (strip and slide) was established utilizing 0.75 w/o EBS in an FLN2-4405 mix compacted at room temperature. EBS is commonly used at this level and temperature in the manufacturing of numerous PM parts. It was reasoned if the AncorLube, at various levels and temperatures, could perform as well or better than the performance of the EBS (compacted at room temperature, 25°C), than it would be suitable for use on a production compacting press.

The results in Table I show AncorLube, even though used in lower percentages and higher temperatures than the EBS (0.40 versus 0.75 w/o), has strip and slide pressures that are comparable or lower than that of the EBS. This indicates that the lubricant has the necessary lubricity to be used at the lower percentages. The lower amount of lubricant also leads to a higher green density for the mix.

Table I: Green and Ejection properties of FLN2-4405 mixes.

Lubricant	Compacted 827 MPa Compaction Temperature	Green Density (g/cm^3)	Green Strength (MPa)	Strip (MPa)	Slide (MPa)
Acrawax C (0.75%)	25°C	7.19	18	21	17
AncorLube (0.40%)	60°C	7.34	23	18	17
AncorLube (0.40%)	77°C	7.36	25	18	17
AncorLube (0.40%)	107°C	7.38	25	17	17

Figure 3 shows the green density and green strength of test specimens compacted over a range of compaction pressures at a temperature of 107 °C. The green density measured at 827 MPa is 7.41 g/cm^3 versus pore free density of 7.54 g/cm^3. The green strength of the lubricant is excellent achieving over 25 MPa at a compaction tonnage of 827 MPa. This is approximately 38% higher than the EBS compacted at room temperature.

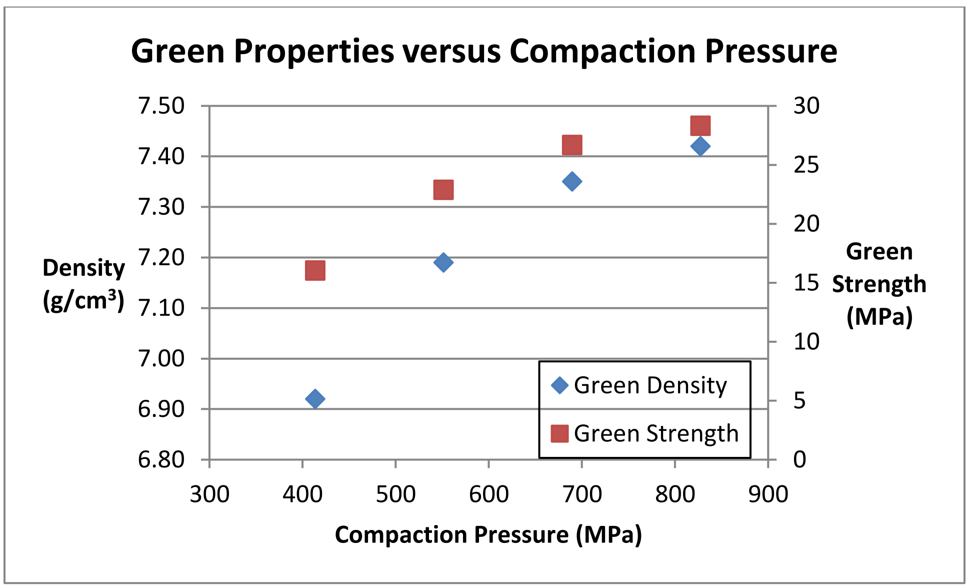

Figure 3: Green Density and Strength versus Compaction Pressure.

One of the key components to the lubricant system is the surface finish and performance on an actual part. The small test specimens produced for measuring properties do not signify that the lubricant will be able to make a part with a surface finish and density that are acceptable on a production scale. The geometries of the test bars are relatively simple and because of the limited number of specimens produced the chance for lubricant breakdown or powder adherence (which can lead to poor surface finish) are minimal. In order to test the lubricant performance VVT Stator rings were pressed on a 140 tonne hydraulic press. The stator had a diameter of 73 mm and a height of 16.7 mm weighed approximately 156 grams. The parts were compacted to a density of approximately 7.28 g/cm^3 and were FC-0208 composition. Over 500 parts were compacted. The surface quality of the compacted parts is shown in Figure 4.

(a)

(b) **(c)**

Figure 4: (a) VVT Stator and (b) Surface quality of standard lubricant system versus; (c) AncorLube with 0.40 w/o and warm compacted.

While the parts using the standard lubricant system exhibited acceptable surface quality they still exhibited some striations on the surface. The striations on the surfaces of the parts pressed from AncorLube were less visible. Although not part of this study, the weight scatter was normal for a premix and the surface quality was consistent throughout the compaction cycle when utilizing the AncorLube at 0.40 w/o.

Double versus Single Press Compaction with AncorLube

Since data presented showed that the AncorLube could be utilized for high tonnage-warm compaction with low lubricant levels, a premix of FLN2-4405 using AncorLube was compared to the same mix using DPDS. The double pressing used a standard lubricant, 0.75w/o EBS; whereas the single pressed bars used 0.40 w/o AncorLube compacted with a heated die (107 ^{o}C.) at 827 MPa. The mechanical properties of the single press-single sinter (SPSS) with AncorLube are compared to those of the DPDS with EBS in Table 2. The SPSS with AncorLube achieved higher density and the same hardness as the DPDS, but the tensile and impact properties were lower.

The data shows that even in instances where the SPSS achieved a higher density that DPDS there are performance gains using a DPDS approach. This indicates there is an aspect of the DPDS process, most likely not related to the lubricant, which provides strength benefits beyond the increase in density.

Table 2: Comparison of mechanical properties for different compaction methods.

		TRS Test				Tensile Test					Impact Test		
		Density	Hardness (HRA)	TRS (MPa)	% Δ	Density	.2% YS (Mpa)	UTS (Mpa)	% Δ	Elong. %	Density	Impact (J)	% Δ
FLN2-4405	DPDS	7.36	59.4	1634	8	7.32	538	807	21	2.62	7.35	34	25
	SPSS	7.49	59	1510		7.40	503	641		1.34	7.41	25.5	

An examination of porosity from different samples gives some insight into a possible cause. Figure 5 shows micrographs from the FLN2-4405 premix processed with SPSS and DPDS. The SPSS bars have a greater number of large pores, which would contribute to the lower strength despite slightly increased density.

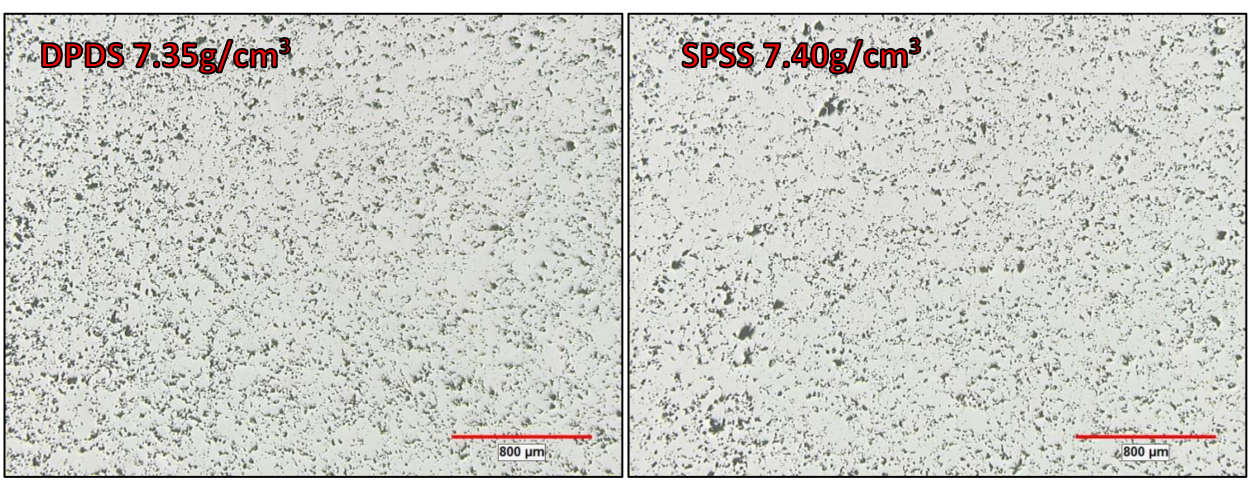

Figure 5: Cross-sectional micrographs of impact bars composition FLN2-4405.

It is well known that larger pores can lead to a decrease in mechanical properties [4]. In order to rule out the role of the lubricant as the cause of the variation in mechanical properties shown in Table 2, a second set of impact specimens were made with AncorLube and these specimens were processed by the DPDS and another set of SPSS specimens were made with EBS. Results shown in Figure 6 show that the impact energies of the DPDS made with both AncorLube and EBS are similar and likewise for the single press and single sinter. The porosity was examined in these samples as well (Figure 7) and the porosity with the AncorLube samples using DPDS was finer than that of the AncorLube made by SPSS. This would indicate that the DPDS

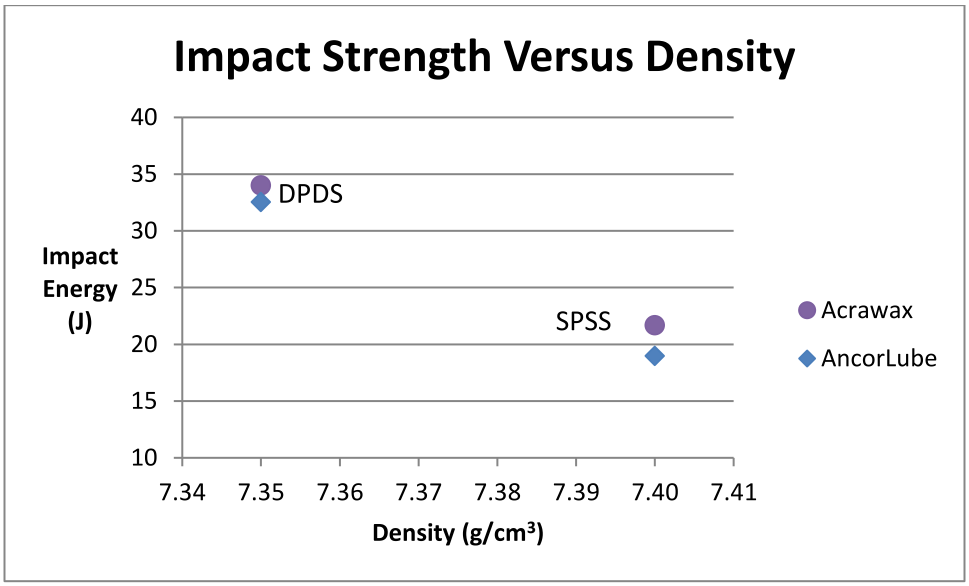

Figure 6: Impact strength versus density for different processing routes.

approach has the additional benefit of reducing the pore size in the final specimen and leads to higher mechanical properties regardless of the lubricant chosen. One possible explanation is that the second press collapses pores eliminating these large voids or forming multiple smaller pores in their place. From this data it is apparent the double press process offers performance benefits beyond density increase.

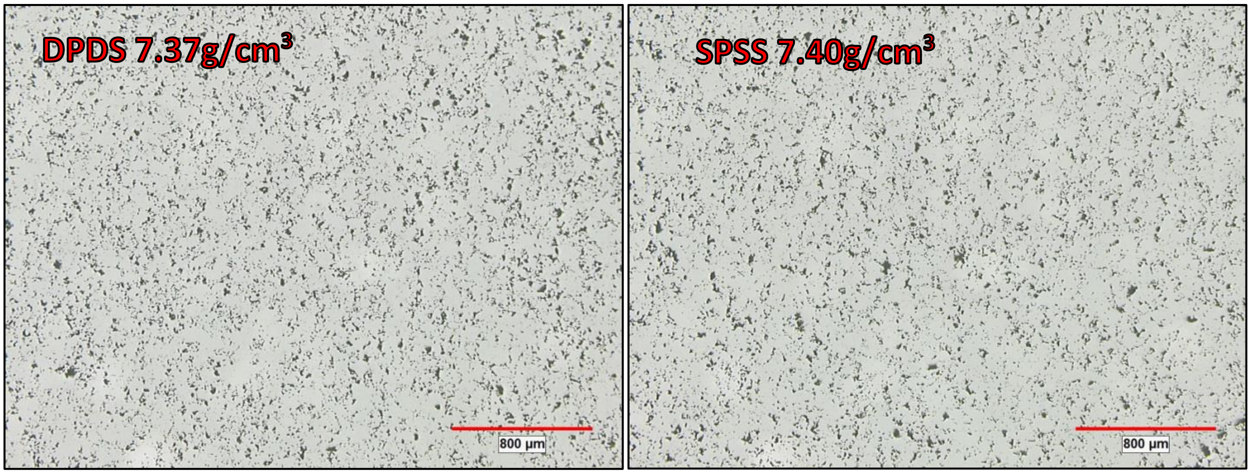

Figure 7: Porosity of impact bars both pressed initially using AncorLube.

Conclusions

- Densities upwards of 7.40 g/cm^{3}are possible in only a single compaction step utilizing specially designed lubricants and processing methods.
- Specially designed lubricants can offer significant performance improvements allowing increased pore free density.
- The DPDS method provides performance benefits over a single press operation that reaches the same density.
- These performance benefits are largely related to porosity and the second compaction step.

REFERENCES

1. H. Rutz, J. Khanuja, S. Kassam "Single Compaction to Achieve High Density in Ferrous P/M Materials in Automotive Applications", *Proceedings of the 1996 World Congress on Powder Metallurgy & Particulate Materials*, Washington, D.C., June 16-21, 1996.
2. K. McQuaig, P. Sokolowski, C. Schade "Development of a Lubricant System for Improved Performance of Premixes", *Proceedings of the 2013 International Conference on Powder Metallurgy and Particulate Materials,* Chicago, Illinois, June 24-27, 2013.
3. "Standard Test Methods for Metal Powders and Powder Metallurgy Products", Metal Powder Industries Federation, Princeton, NJ, 2016.
4. A. Wartenberg, P. Sokolowski, C. Schade "The Effect of Physical Properties of Lubricants on the Performance of Premixes", *Proceedings of the 2016 International Conference on Powder Metallurgy and Particulate Materials,* Boston, Massachusetts, June 5-8, 2016.