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Lubricants for High-density applications by pressing at moderate temperatures

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ABSTRACT

The nature of the lubricant and the conditions of compaction, in particular, the temperature and the pressure, can affect significantly the densification of PM powder mixes. While the effect of pressure on densification is straightforward, the effect of temperature is more complex. Depending on the nature of the lubricant, the increase of the compaction temperature can significantly affect the level of friction at die walls as well as the lubricant distribution in the compact, and therefore affect the compressibility of powder mixes.

In this paper, the compressibility and lubrication behavior of PM powder mixes containing conventional and new lubricants for high-density applications is reviewed. The effect of temperature on PM powder mixes containing the new lubricants is particularly highlighted.

INTRODUCTION

The quest for parts with higher density processed by inexpensive shaping processes is a constant focus for the PM industry. The most cost effective compaction processes available to the PM industry to produce high green density components are the die wall lubrication process, warm compaction, and cold compaction using special lubricants.

The warm pressing process¹, which consists in pressing a preheated powder mix in a heated die (most often between 100°C and 180°C), enables the fabrication of parts with high density and green strength by increasing the ductility of the ferrous powder particles. The gain in density achieved by warm compaction versus conventional cold compaction generally ranges between 0.12 to 0.30 g/cm³. The main drawbacks of this technology are the need for specific presses and toolings to heat both the powder mix and the die. The need for specific internal lubricants to provide adequate lubrication at die walls during both the compaction and ejection steps add to the complexity of this process. The die wall lubrication technique² is also a promising route to promote green densities when high compacting pressures are used. The benefits of this technique consist in the possibility to significantly reduce the internal lubricant level in the powder mix, while maintaining good lubrication at die walls during the compaction and the ejection of parts. This technique is not widely used on a mass production scale because of concerns regarding its reliability. Recent improvements seem to have addressed most of these concerns³.

Cold compaction using conventional lubricants such as metallic stearates or amide-based waxes does not generally yield high green density parts. Recently, more effective lubricants have been developed for conventional compaction at room temperature.

Hammond⁴, for example, describes a lubricant system, solid at ambient conditions, but upon application of pressure, it transforms to a liquid phase. It is claimed that this lubricant can be used at reduced concentration due to the efficiency of lubrication from the transformation from a solid to a liquid. Because less lubricant is used, the green density is increased. Compaction processes where a non-heated powder is compacted in a heated die have also been developped⁵. The temperatures involved are around 60°C and thus presses with specific toolings are not required. In addition, the flowability of the powder mixes is less of a concern than in the warm compaction process where the powder mix is heated.

In this paper, the compressibility and lubrication behavior of PM powder mixes containing conventional and new lubricants for high-density applications is reviewed. The effect of the compacting temperature on PM powder mixes containing the new lubricants is particularly highlighted.

BACKGROUND

Several factors affect the green density of PM parts. Among these factors are the friction at die walls, the powder intrinsic compressibility and the springback after ejection⁶. The friction at die walls can be described by the slide coefficient, η^7 . For a cylindrical part of diameter D, compacted in a single action press, the slide coefficient at the end of the compaction can be calculated using the following equation:

$$\eta = \left(\frac{Pt}{Pa}\right)^{\left[\frac{4F}{SH}\right]},\tag{1}$$

where P_a is the pressure applied to the pressing punch, P_t is the pressure transmitted to the stationary punch and H is the height of the cylinder. The numerical value of η varies from 0 to 1; the higher the slide coefficient, the lower the friction at the die walls and the more uniform is the density through the compact.

The powder intrinsic compressibility is a measure of the densification of a powder mix in the absence of friction at die walls, i.e., the pressure transmitted to the powder compact is equal to the applied pressure. It can be evaluated by the relation between the average IN die density (i.e. the density of the part under pressure) and the average pressure seen by the compact. It has been shown⁸ that, for a cylindrical compact of diameter D, the average pressure or net pressure, P_{NET} can be evaluated by the following relationship:

$$P_{NET} = Pa * \eta^{\left(\frac{H}{2D}\right)} = (Pa * Pt)^{1/2}$$
 (2)

The green density of PM parts is usually described in terms of the percentage of the pore free density. The pore free density is the density of a powder compact containing no porosity. In practice, the maximum green density attainable is 98% of the pore free density. The pore free density depends on the powder mix composition. Alloying additives such as copper and nickel increase the pore free density of iron based powder compacts while graphite and lubricant lowers it. The lubricant, with a density of around $1g/cm^3$, is the additive that has the largest impact on the pore free density of PM compacts. For example, the effect of the concentration of an admixed lubricant, having a density of 1 g/cm^3 , on a FC0205 powder mix containing 2 (wt%) of copper and 0.6 (wt%) of graphite is shown on Figure 1. A way of increasing the pore free density and the green

density is to reduce the lubricant concentration in the powder mix. However, this can prove to be difficult from a practical point of view. Indeed, lowering the lubricant content can dramatically increase the friction at the die walls and impede the compaction.

Another strategy for increasing the green density of PM parts is to use alternative lubricants and processing conditions that favor the expulsion of the lubricant from the green compacts during compaction. For a given lubricant concentration, this should increase the pore free density and as a result the green density. It was shown⁹ that, the compressibility of stearate/steel mixes could be improved by increasing the compacting temperature. However, this was not attributed to the higher amount of lubricant expelled from the green compact at higher temperature, but rather to the enhanced plastic deformation of the metallic particles during compaction. Moreover, the green density improvement was limited because of the significant increase of the friction at die walls as the compacting temperature was raised. For certain stearate/steel mixes, this even gave rise to stick and slip during ejection. The friction at die walls increased as much as 15% when the compacting temperature was raised from room temperature to the softening points of the lubricants. This was attributed to the low viscosity of the lubricant for these processing conditions. It was confirmed, in a study on lithium stearate/steel mixes¹⁰, that the higher density measured at high temperature was not due to an expulsion of the lubricant from the compact. Study of the lubricant distribution in the compacts showed that the lubricant had a tendency to flow towards the die surface, but with the processing conditions used, limited lubricant was expelled from the compact.

It must be emphasized that in both studies, the compacting pressure was limited to 620 MPa (45 tsi) due to insufficient lubrication on the die walls that prevented the application

of higher pressures. This could explain the small quantity of lubricant that was expelled from the green compacts. Lubricant systems with an adequate rheological behavior at moderate compacting temperature, i.e., 50 to 100°C, allowing their expulsion from green compacts while maintaining good lubrication at die walls at pressures higher than 760 MPa (55 tsi) have been developed. As a result, PM parts with improved green density can be obtained.

EXPERIMENTAL PROCEDURE

The compaction and ejection characteristics of FC0205 and FC0208 powder mixes made with the ATOMET 1001 atomized steel powder produced by Quebec Metal Powder Ltd were characterized at different compaction temperatures. These two mixes contain 2 wt.% of Cu (MD 165) and respectively 0.6 and 0.8 wt.% of natural graphite (South Western 1651). These powder mixes were admixed with the conventional ethylene bisstearamide (EBS) lubricant and with two proprietary lubricants, Lube A and Lube B, developed for high-density applications. Lube A and Lube B have similar chemical structure but have respectively softening points of 60 and 80°C.

The compaction and ejection characteristics of the FC0208 powder mix were evaluated on an instrumented laboratory single action press, known as the Powder Testing Center or PTC¹¹. Cylindrical specimens with heights of 15 mm and aspect ratios of 6.3 (4.5 times the aspect ratio of rectangular bar (TRS bar, 1.25 inches-long, 0.5 inch-wide and 0.25 inch thick) were pressed in a WC-Co tool having a diameter of 9.525 mm at a compacting speed of 1 mm/sec. The PTC press permits continuous recording of the moving punch displacement, the forces applied to the moving punch and transmitted to

the stationary punch, and the IN-die density, all along the compaction and ejection processes. This allows the determination of the intrinsic compressibility, the slide coefficient, and the ejection forces. The stripping pressure, which corresponds to the force needed to start the ejection process divided by the friction area (contact surface between the compact and the die wall), and the ejection unit energy were estimated from the ejection curve in order to compare the lubricating performance of lubricants. The ejection unit energy is evaluated from the calculation of the area under the ejection curve (force vs. displacement) divided by the displacement and the friction area.

The lubricant loss during compaction and after ejection was also measured on the FC0208 powder mix. TRS bars, 0.25-inch thick, were compacted at 65°C to green densities of 6.8 g/cm³, 7 g/cm³ and 7.3 g/cm³ (760 MPa), using a floating die and a 100-ton hydraulic press. The specimen were slightly polished to eliminate the lubricant on the sample surface and then sintered at 1120°C for 30 minutes in dissociated ammonia to remove the lubricant from the compacts. Weights before and after sintering were measured.

The behavior of the FC0205 powder mix was evaluated on an industrial 150 tons. Gasbarre mechanical press. Straight gears (15 teeth), one-inch thick, with an aspect ratio of 4.7, were pressed in a CPM15V tool die preheated at a temperature of 70°C. The temperature of the parts at steady state was around 80°C. The compacting pressure was varied from 415 to 830 MPa (30 to 60 tsi). Compressibility curves were drawn and ejection forces were recorded.

RESULTS AND DISCUSSION

Effect of temperature on compressibility and ejection forces

Figure 2 shows the effect of temperature on the green density of two FC0208 powder mixes, compacted on the PTC press, containing respectively 0.75 (wt%) of EBS and Lube A. At room temperature, the green density of the two powder mixes is relatively similar. However, for compacting temperatures near and above the softening point of Lube A (60°C), the green density of the mix containing Lube A experiences a big jump while that of the other mix remains almost constant. At a compacting pressure of 760 MPa (55 tsi) and a temperature of 65°C, the green density of the mix containing Lube A is approximately 0.2 g/cm³ higher than that of the mix containing the EBS lubricant and about 99.1% of the pore free density. As shown in Figure 3 to 5, this increase in green density can be related, in part, to the increase of the intrinsic compressibility and the decrease of the springback after ejection when the compacting temperature is near or above the softening point of Lube A. This significant increase in the intrinsic compressibility can be explained by the improvement of the internal lubrication and by the expulsion, from the green compacts, of a portion of the lubricant. In the compacting temperature range used in this study, the effect of temperature on the ductility of the metallic powders should have a limited impact. Another factor explaining this green density increase, is the behavior of the slide coefficient with the compacting temperature (Figure 6 and 7). The slide coefficient of the mix containing Lube A is maintained or even improved for a compacting temperature near or above the softening point of Lube A. This is a major difference with the warm compaction of stearate/steel mixes near the melting points of the stearate lubricants, where a sharp decline of the slide coefficient is observed. The increase in green density with temperature observed in this study can be explained, both by the improvement of the intrinsic compactability and the decrease of the springback after ejection, and by the ability of the lubricant, Lube A, to maintain a good lubrication at die walls during compaction.

Figure 8 and 9 show the variation of the stripping pressure and ejection energy, of the two powder mixes containing the EBS and the Lube A lubricants, with the compacting temperature. Again, in sharp contrast with the warm compaction of stearate/steel mixes⁹, the ejection performances of the mix containing Lube A are maintained even though the compacting temperature is near the softening point of Lube A. This property allows high aspect ratio parts, having high densities, to be compacted without damaging tool dies.

Effect of temperature on lubricant loss during compaction

Figure 10 shows that no lubricant was expelled from the powder compacts pressed with the mix containing the EBS lubricant. The same behavior was recorded for the mix containing the Lube A lubricant for densities below 6.8 g/cm³. However, for higher densities, the lubricant loss increased rapidly with the green density. For a compacting pressure of 760 MPa (or a green density of 7.3 g/cm³), more than 25% of the initial quantity of lubricant was expelled from the green compact. This represents an increase of the pore free density of approximately 0.1 g/cm³ or 1.2%. A green density of 7.3 g/cm³ represents 99.1% of the initial pore free density, or 98% after the lubricant loss is taken into account. Expulsion of the lubricant from the compact decreases the amount of lubricant present in the porosity. This postpones the inhibition of the compaction at

higher densities. Therefore, higher green densities can be reached. The variation of the pore free density as a function of the compacting temperature may also have an impact.

The mechanisms by which the lubricant is expelled from the green compact are rather complex. If we consider the case where the lubricant is in a liquid or a semi-liquid state, we can assume that the lubricant flows into the porous compact by capillary flow, assisted by pressure. The simplest model describing the kinetics of penetration of a liquid in a porous medium is the Lucas-Washburn model¹², given by the following equation:

$$L^2 = \left(\frac{\sigma D \cos(\theta)}{4\mu}t\right) , \tag{3}$$

where L is the depth of penetration of the liquid, t is the time, σ is the surface tension of the liquid, θ is the contact angle between the solid and the liquid, D is the average pore diameter, and μ is the dynamic viscosity of the liquid.

As the compaction proceeds, the pore volume and pore size decrease. Equation (3) shows that it becomes more and more difficult to move the lubricant through the pores towards the die walls. As the pressure is increased, the lubricant fills the pore volume. Once the pore volume is filled, the lubricant will eventually exits the compact, if it reaches the die walls before the pores are closed. As shown by equation (3), this will depend mainly on the viscosity of the liquid. The viscosity must be sufficiently low to allow the rapid movement of the liquid and at the same time sufficiently high to provide adequate lubrication at the die walls. The lubricant expulsion towards the die walls will carry on as long as the applied pressure is higher than the capillary pressure required to move the liquid lubricant. It was observed experimentally that the expulsion of the lubricant could

carry-on even after ejection of the green parts from the die. After ejection, the pressure is relaxed in the solid phase, but not in the liquid phase. This induces a backward pressure, promoting the expulsion of the lubricant. The total lubricant loss includes the expulsion of the lubricant during compaction and after ejection. Expulsion of the lubricant after ejection can be controlled easily by controlling the viscosity and the concentration of the lubricant.

It must be noted that, in this study, the green density is improved even at low compacting pressures where the porosity should accommodate easily the lubricant. At low compacting pressure, the green density improvement is related to the fact that the good lubrication properties of Lube A enables to reduce friction and therefore enable to increase the density. At higher compaction pressures, the excellent lubrication properties and the properties of the lubricant itself (viscosity) enable to apply sufficient pressure to expulse the lubricant from the compact. Therefore, the increase of intrinsic compressibility while maintaining low friction at die walls explains the increase of density at high compaction pressure.

Validation on a industrial press

Expulsion of the lubricant towards the die walls is a kinetic driven phenomenon. As such, it depends on the compaction rate. The compaction rates of laboratory presses are far from industrial conditions. In order to validate the results observed on the laboratory presses, gears (15 teeth) were pressed on an industrial press at a stroke rate of 10 parts per minute. FC0205 powder mixes containing 0.6 (wt%) of EBS and Lube B were compacted. The pore free density of these mixes is 7.47 g/cm³. Lube B is a lubricant

designed for high-density applications requiring excellent lubrication properties. The compaction temperature was near the softening point of Lube B (80°C) for the mix containing Lube B. It had to be set lower for the mix containing the EBS lubricant (60°C) because of lubrication difficulties. Even at this compacting temperature, the lubrication of this mix was insufficient to press gears with heights higher than half an inch. Figure 11 shows the compressibility of the two powder mixes. At 830 MPa (60 tsi), the green density of the mixes containing Lube B and EBS is respectively 7.3 g/cm³ and 7.23 g/cm³ or 97.7 and 96.6% of the pore free density. It must be emphasized, that the surface of friction of the gear compacted with the Lube B lubricant (10in²) was twice as high as this of the gear made with the EBS lubricant. As shown on Figure 12, the ejection performances of the mix containing the Lube B lubricant were still better than those of the mix containing the EBS lubricant.

CONCLUSIONS

Lubricant systems designed for pressing at moderate temperature (< 100°C) were developed. The compressibility and lubrication behavior of PM powder mixes containing conventional and these newly developed lubricants were shown as a function of the compacting temperature. It was shown that, significantly higher green densities could be obtained with these lubricants. This was attributed to their ability to migrate and exit green compacts at compacting temperatures near their softening point. Capillary flow assisted by pressure was proposed as the possible mechanism explaining the lubricant expulsion during compaction. The amount of lubricant expelled from the green compacts depends on the viscosity and chemical nature of the lubricant. The viscosity must be

sufficiently low and the pressure sufficiently high to allow the lubricant to reach the die walls before the pore closure. At the same time the viscosity of the lubricant must be sufficiently high to provide adequate lubrication at the die walls. When these conditions are fulfilled, high-density parts with high aspect ratios can be compacted with these lubricants. The density gains are of the order of those of the warm compaction process. However, the compacting temperatures involved are significantly lower. This pressing approach is a good alternative to the warm compaction process.

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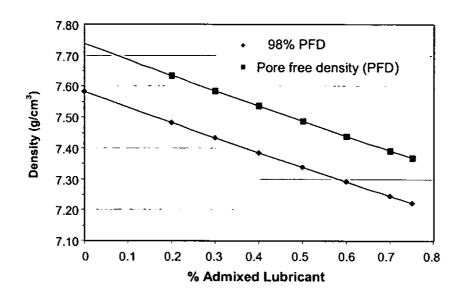


Figure 1: Effect of lubricant content on the pore free density of a FC0208 powder mix.

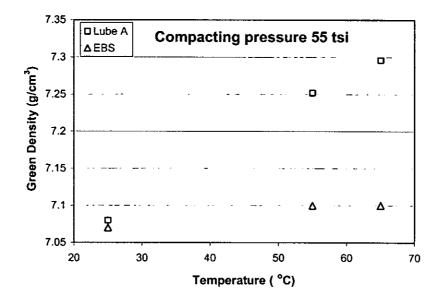


Figure 2: Effect of temperature on the green density of FC0208 powder mixes, compacted at 55 tsi (760 MPa), containing 0.75 (wt%) of EBS and Lube A.

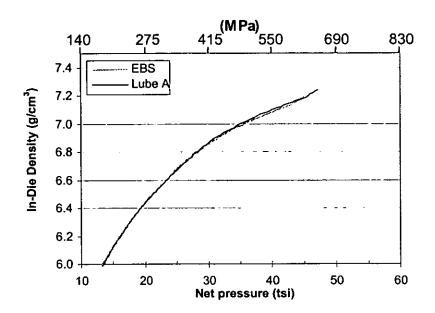


Figure 3: Intrinsic compressibility of FC0208powder mixes, containing 0.75 (wt%) EBS and Lube A, at room temperature.

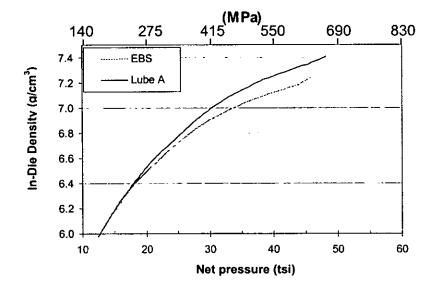


Figure 4: Intrinsic compressibility of FC0208 powder mixes, containing 0.75 (wt%) EBS and Lube A, at 55°C.

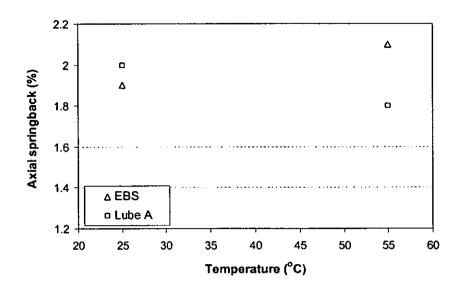


Figure 5: Effect of temperature on the axial springback of FC0208 powder mixes containing 0.75 (wt%) of EBS and Lube A. Note that the axial springback was measured by the following formula (H2-H1/H2) where H1 is the height of the part In-Die (under pressure) and H2 is the height of the part out-die. The measure of the displacement of the mobile (lower) punch of the PTC press enables to evaluate precisely the In-Die height (H1)

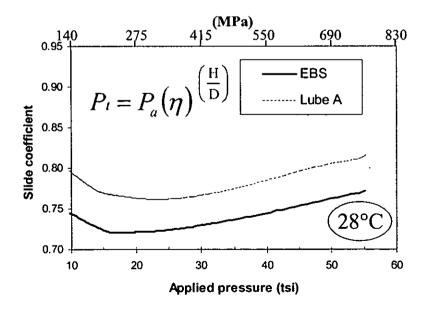


Figure 6: Effect of the applied pressure on the slide coefficient of FC0208powder mixes, with 0.75 (wt%) of EBS and Lube A, compacted at room temperature.

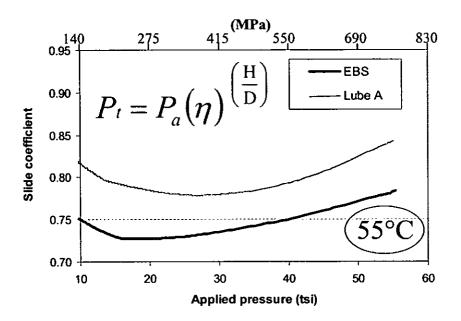


Figure 7: Effect of the applied pressure on the slide coefficient of FC0208 powder mixes, with 0.75 (wt%) of EBS and Lube A, compacted at 55°C.

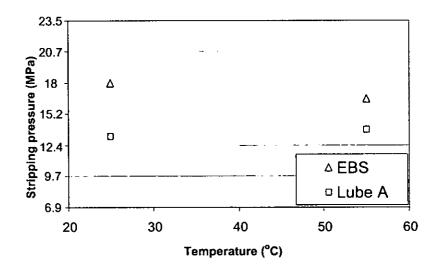


Figure 8: Effect of temperature content on the stripping pressures of FC0208 powder mixes containing 0.75 (wt%) of EBS and Lube A.

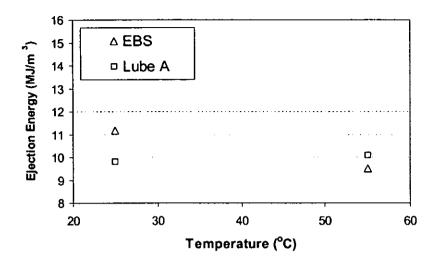


Figure 9: Effect of temperature content on the stripping pressures of FC0208powder mixes containing 0.75 (wt%) of EBS and Lube A.

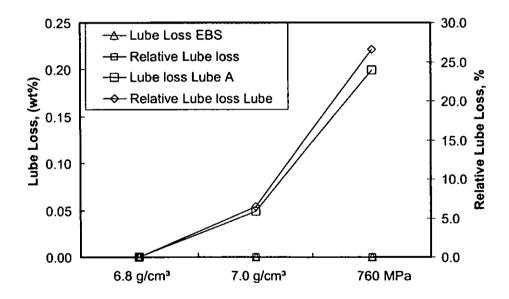


Figure 10: Total lubricant loss during compaction at 65°C and after ejection of FC0208 powder mixes containing 0.75 (wt%) of EBS and Lube A.

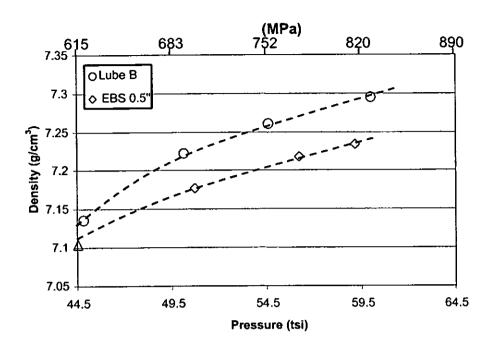


Figure 11: Compressibility of FC0205 powder mixes containing various lubricants. Results for the EBS lubricants correspond to half-inch thick gears compacted at 60°C while these of Lube B refer to one-inch thick gears compacted at 80°C.

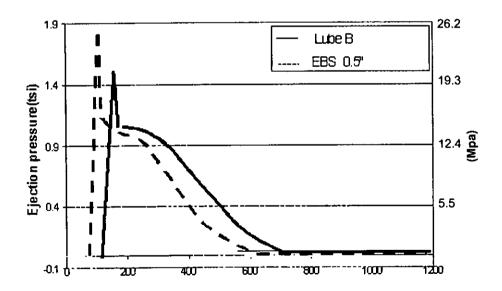


Figure 12: Ejection behaviour of FC0205 powder mixes containing various lubricants. Results for the EBS lubricants correspond to half-inch thick gears while other lubricants refer to one-inch thick gears.