ENHANCING GREEN STRENGTH OF P/M MATERIALS

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ABSTRACT

Lubricants are admixed into P/M mixes to ease compaction and facilitate the ejection of parts from the die. However, the use of conventional internal lubricants usually lowers the strength of green compacts. The development of new lubricating systems that improve the strength of parts in the green state is an interesting avenue to develop new P/M applications. Indeed, the use of these systems opens the possibility of machining green parts and could also prevent part failure during manipulation before sintering.

This paper presents a comparative evaluation of green and sintered properties achieved with FC-0205 mixes containing either a new high green strength binder-lubricant system or a conventional EBS wax pressed at temperatures in the range of 45°C and 65°C to green densities of 6.8, 7.0 and 7.2 g/cm^3. This comparison was performed from TR bars and gear shape specimens pressed on laboratory and production presses. Results showed that mixes containing the new lubricating system exhibit either similar or better compressibility and lubrication behavior than mixes admixed with the conventional EBS wax. Moreover, the green strength of as-compacted TR bars produced with the new lubricating system was more than twice as high as that of specimens containing the conventional EBS wax lubricant. These properties can be explained by the presence of polar groups along the molecular chains of polymers included in the new HD system, which gives to the lubricating film a good affinity for both powder particles and die cavity. The sintered properties of specimens pressed with the new HD system were similar to those obtained with conventional EBS wax lubricant.

INTRODUCTION

The development of new techniques that either enable machining of parts prior to sintering or prevent green part failure could be of great advantage to the P/M industry. Indeed, with the advent of high performance materials that exhibit high apparent hardness and strength after sintering, green machining becomes a very attractive process to improve tool life, increase productivity and promote competitiveness. A recent study clearly demonstrated that typical stresses applied on tooling, when drilling F-0005 parts in a green state, is reduced by a factor of 8 to 10 as compared to drilling executed after sintering [1]; this may be even more pronounced for parts made from sinter hardening materials.

It is common practice to use lubricant during the compaction of ferrous powders to reduce the friction between the powder and die walls. Indeed, the admixed lubricant improves the uniformity of densification throughout the part and also ensures a good transfer of the compacting force during the compaction stage. In particular, the use of lubricants lowers the force required to remove the compact from the die, thus minimizing die wear and yielding parts with good surface finish. However, conventional lubricants such as synthetic waxes and metallic stearates significantly reduce the strength of green compacts.

The development of new polymeric lubricating systems is a promising avenue to prevent part failure and/or enable machining of parts prior to sintering. Only a few studies have explored the feasibility of using such enhancing green strength lubricating systems to meet these requirements [2-6]. The increase in green strength is explained by the higher intrinsic mechanical properties of these polymeric lubricants as compared to standard lubricants such as synthetic waxes and metallic stearates. Additionally, unlike conventional lubricants, which tend to be distributed at the surface of metallic powders during the mixing step, polymeric lubricants most often have a lower deformability and remain as discrete particles. This can potentially favor the formation of interlocking or microwelding between the metal powder particles during the compaction step, and, therefore, increase the green strength [7].

This paper first describes some characteristics that should be considered in the selection of polymeric lubricants. Then, the compaction and lubrication behaviors of mixes containing either a new polymeric lubricating system or a conventional EBS wax are discussed. This comparative study also covers green and sintered properties of parts pressed on either a lab press or a production press.

EXPERIMENTAL PROCEDURE

The green properties of FC-0205 mixes containing either a new binder-lubricant system (HD system) or a conventional EBS wax were compared using a laboratory hydraulic press as well as a production mechanical press. These mixes were prepared with ATOMET 1001, 2.0 wt% copper, 0.60 wt% graphite as well as 0.75 wt% of the respective lubricating system. The compressibility and lubrication behavior during compaction and ejection of two F-0008 mixes containing polymers having different polar characters (P-1 and P-2) were also determined using a full instrumented laboratory press, known as the Powder Testing Centre in order to help in the development of the new high green strength binder-lubricant system.

Hydraulic Press

Standard transverse rupture (TR) bars were prepared to compare green and ejection properties of the FC-0208 mixes containing either the new HD system or the conventional EBS wax. The compressibility and ejection characteristics were determined with 0.64 cm (¼ in) and 1.27 cm (½ in) thick TR bars respectively, while the green strength was evaluated with both sizes TR bar. The die set was heated to reach either 45^{o}C, 55^{o}C or 65^{o}C in order to simulate the frictional heat generated during typical production conditions. Cold powder mixes were fed into the die cavity when thermal equilibrium was reached. The mixes were then pressed to reach green densities of 6.8, 7.0 and 7.2 g/cm^3. Details of the compacting conditions are listed in Table 1.

The ejection properties of the two mixes were evaluated using an automatic data acquisition system. The force required to eject the TR bars was measured throughout the ejection step. By dividing the load by the area of the compact in contact with die walls, it was possible to determine the stripping and sliding pressures needed to eject the specimens. The stripping pressure corresponds to the shearing stress required to initiate the ejection, while the sliding pressure represents the mean stress needed to move parts to the die entrance. Green properties were evaluated according to the MPIF standard test procedure Nº15.

Production Mechanical Press

A total of 900 gears were compacted from each FC-0205 mix on a Gasbarre 150 ton mechanical press to a green density of 7.0 g/cm^3. The gear had an outer diameter of 5.08 cm (2.0 in) as well as an overall height (OAH) of 2.41 cm (0.95 in) with a hub section 0.89 cm (0.35 in) high. The outer diameter and wall thickness of the hub section were 2.03 cm (0.80 in) and 0.38 cm (0.15 inch) respectively. An illustration of the gear is shown in Figure 1. For this part, the frictional area between the gear and the die walls is significantly higher than that of standard 0.64 cm (¼ in) thick TR specimens, i.e. 39.0 cm^2 versus 5.7 cm^2.

The mechanical press was set to produce 7.5 gears per minute. Prior to the production run, die and punches were heated to the temperature obtained during typical production conditions, i.e. 55°C. The temperature of parts was measured with a contact probe immediately at the exit of the die cavity at a frequency of once every 50 parts. The tonnage and ejection curves were recorded during the production run by using an automated data acquisition system. Details of the experiments are given in Table 1.

Figure 1. Illustration of the Gear with a Hub.

TABLE 1
Compacting Temperature and Green Density for TR bars and Gears Pressed from HD and EBS Lubricating Systems

Type of Part	Lubricating System	Compacting Temperature (°C)	Green Density (g/cm^3)
TR bar	HD	45 / 55 / 65	6.8 / 7.0 / 7.2
	EBS	45 / 55 / 65	6.8 / 7.0 / 7.2
Gear	HD	55	7.0
	EBS	55	7.0

Powder Testing Centre

The evaluation using the Powder Testing Center (model PTC-03DT / KZK Powder Technologies Corp.) was performed by compacting 8.0 mm high cylindrical specimens at either a constant IN-die density of 7.25 g/cm^3 or at an applied pressure of 620 MPa at 45°C. This instrumented press enabled continuous recording of the punch displacements, the forces applied to the moving punch and transmitted to the stationary punch and the IN-die density all along the compaction and ejection stages. The measure of the displacement of the mobile (lower) punch was converted to calculate the average density of the part by assuming a rigid behavior of the D2 steel die of 9.525 mm diameter.

The lubricant behavior during compaction was evaluated by determining the slide coefficient. This parameter is directly related to the loss of pressure between the applied pressure and the transmitted pressure on the compact due in particular to friction between metal powder particles and also between the surface of the compact and the die walls [8]. Its numerical value varies between 0 and 1. Values of slide coefficient reported in this study correspond to the values obtained at the end of compaction cycle for a constant IN-die density of 7.25 g/cm^3 at 45°C. In these conditions, the greater the slide coefficient, the lower the level of friction between powder particles and between the surface of the part and the walls of the die.

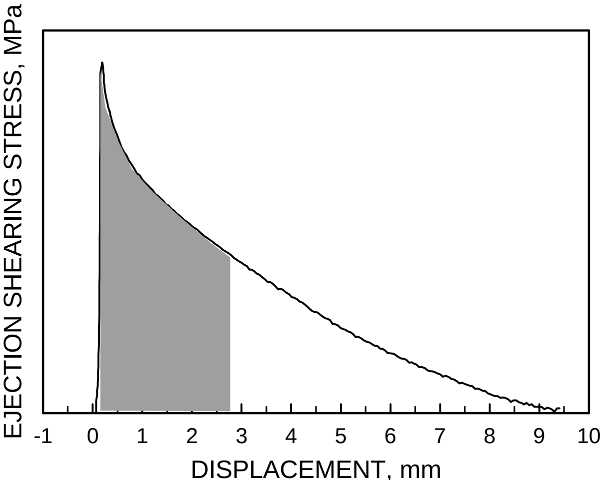

Figure 2. Illustration of typical ejection

The ejection performance was determined for specimens pressed at a compacting pressure of 620 MPa at 45°C. The measurement of the force acting on the compact during its ejection as a function of the displacement inside the die enabled the evaluation of the ejection shearing stress, by dividing the force by the cylindrical area of the compact in contact with the die walls, as shown in Figure 2. The initial quick increase of the shearing stress, called stripping pressure, is necessary to overcome the static friction coefficient, while the subsequent reduction of the ejection force is attributed to the lower dynamic friction coefficient and the asperity reduction at the surface of the compact resulting from friction wear. An interesting parameter extracted from the ejection curve, the unit ejection energy, was evaluated from the calculation of the area under the ejection curve (ejection shearing stress vs. displacement) divided by the displacement of the part to the die entrance (2.54 mm).

RESULTS AND DISCUSSION

Selection of Enhanced Green Strength Lubricating Systems

Polymeric lubricants were recently considered as potential candidates to enhance the green strength of compacted parts. This is mainly because of their higher intrinsic mechanical properties as compared to conventional lubricants. Indeed, polymers differ from most conventional lubricants since they are composed of macromolecular chains, which consist of a large number of repeating units. In addition, this type of compound has lower deformability than conventional lubricants and thus a lower tendency to form a continuous lubricating film at the surface of powder particles during mixing or compaction, which could potentially favor the formation of stronger bonds between the steel powder particles. As already reported, the green strength may be controlled by the formation of solid contacts or microwelding between the powder particles [7].

However, it is worth mentioning that not all polymers exhibit adequate shear resistance as well as lubricating properties during compaction and ejection. Indeed, when formulating a polymeric lubricating system, the selection of polymers have to take into account several characteristics such as the nature, structure, molecular weigth, softening temperature and/or particle size to achieve a compromise between the compressibility and lubricating properties of the powder mixes, while maintaining a good surface finish of parts. For example, under specific conditions and for a given polymeric lubricants, an increase of the molecular weigth may have a beneficial effect on the lubricating performance during ejection, but too high a molecular weigth may also deteriorate the compressibility of mixes due to the lower deformability or the higher shear resistance of the polymer [3].

The ability of some polymers to have adequate lubricating properties during either compaction or ejection may be attributed to the regular arrangement of macromolecular chains and their ability to slide over one another when submitted to a shear stress as well as their affinity with metallic substrates. Indeed, it is believed that polymeric lubricants should possess adequate wetting, adsorbing and adhering properties with the metallic powders and die cavity to form a lubricating film and reduce the shearing stress during compaction and ejection of parts. The adsorbtion characteristics of polymeric lubricants with metallic surfaces rely both on the nature of the polymers and on the roughness and active sites on the metallic surfaces. In particular, polymers such as saturated polyolefins will adsorb to metallic surfaces only by weak Van der Waals forces, and the polymeric film formed can be readily swept from these surfaces [9]. Most other polymers (polyether, polyester, polyamide, etc.) contain functional groups, which are either electron donor group (basic group) or proton donor group (acidic group). These groups may be part of the macromolecular chains or be present as end-groups of the chains. These “intrinsic” or end-groups are able to interact either by acid-base or Van der Waals interactions with the acidic and basic sites (Fe_2O_3, $Fe(OH)_3$, etc.) located at the surface of metallic substrates. The effect of the end-groups is even more pronounced for low molecular weight polymers, as it is the case with polymeric lubricants. Typical acid groups in polymers include hydroxyl, carboxyl, amine or amide groups, while basic groups include the nitrogen or oxygen atoms located in amine, carbonyl or hydroxyl groups. Depending on the strength of the affinity of the polymeric functional groups to the active metallic sites, the solid film formed between metallic surfaces will more or less strongly adhere to these surfaces. For example, instead of pure homopolymer of polymethacrylate containing only ester groups, the use of copolymer with a small content of chain unit containing strongly adsorbing carboxylic groups were shown to significantly enhance the level of adsorption of polymethacrylate on iron powder [10].

Figures 3 and 4 show typical examples of the effect on both green and ejection properties of two polymers with a similar intrinsic molecular structure, but containing some functional groups having different polarities and affinities with the iron based surfaces (P-1 and P-2). Both polymers also have a similar molecular weight (M_w) of 5000 g/mol and a polydispersity (M_w/M_n) of about 4.0. This evaluation was performed using a Powder Testing Center (PTC) for more accuracy. Steel powder particles were coated with the lubricating system to avoid any effect of the particle size.

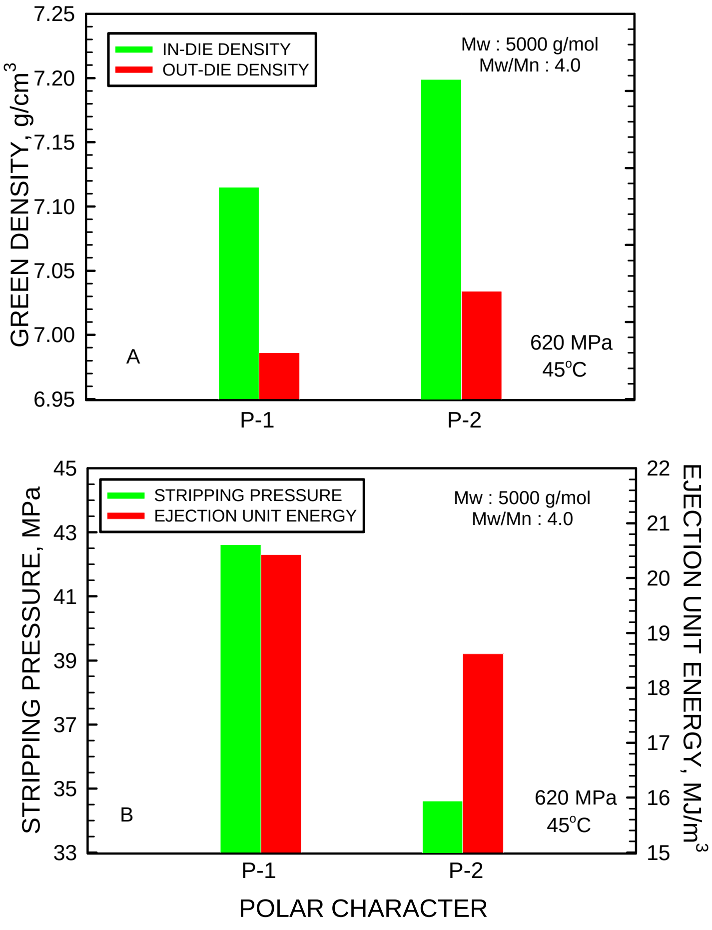

Figure 3. Effect of the Polar Character of the Functional Groups of a Polymeric Type Lubricant on Green Density, Stripping Pressure and Ejection Unit Energy. (P-1 < P-2)

It can be seen that the polarity of the functional groups of a polymer strongly affects the compressibility of a mix (Figure 3a). In particular, the IN-die and OUT-die green densities noticeably increase with the polymeric lubricant having the highest polar character (P-2). The improvement in compressibility is mainly explained by the better intrinsic compressibility due to the lower friction between the particles leading to a better rearrangement during compaction as well as the lower level of friction at the die walls. Indeed, the slide coefficient significantly increases with the P-2 polymeric lubricant when compacting TR bars at a constant IN-die density of 7.25 g/cm^3 (0.663 P-2 vs. 0.625 P-1). The higher the slide coefficient, the lower the level of friction between the steel powder particles and at the die walls during compaction and the higher the net pressure transmitted through the specimens. The lower level of friction between the powder particles and at the die walls may be explained by the better ability of the P-2 polymer to form an adhering lubricating film at the surface of these metallic substrates.

The lubrication behavior during the ejection step is also significantly enhanced when using a polymeric lubricant having a better affinity with the metallic powder and die cavity surfaces. Indeed, it is seen in Figure 3b that the stripping pressure and the energy required to move the specimen to the upper edge of the die cavity are both significantly reduced with the polymer having the highest polar character (P-2).

Finally, it should be emphasized that the formation of strong interaction between the polymer and steel powder particles also leads to a significant improvement in green strength, as shown in Figure 4. It is seen in this Figure that the green strength is increased with the polymer having the highest polar character (P-2), although the applied pressure required to reach the desired green density is significantly reduced. Hence, it is strongly believed that the use of polymers having strong polar groups and a sufficiently high molecular weight will favor the formation of a thin and strong film that adheres to the iron particles and strengthens the green compact.

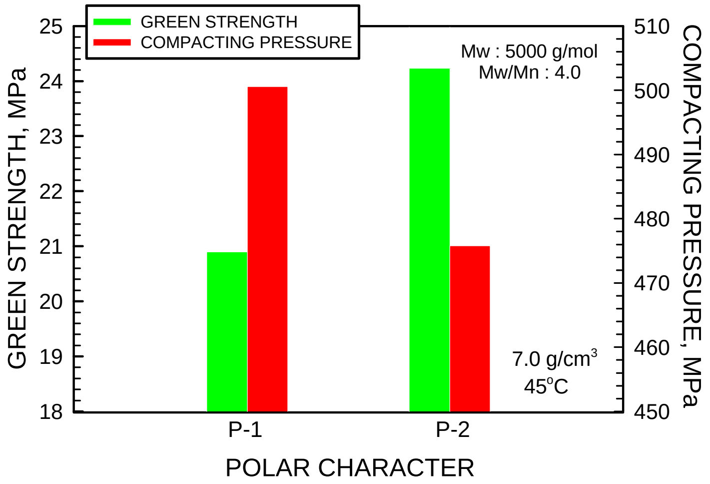

Figure 4. Effect of the Polar Character of the Functional Groups of a Polymeric Type Lubricant on Green Properties. (P-1 < P-2)

Therefore, all these results strongly suggest that it is essential to take into account several intrinsic characteristics such as the molecular weight, softening temperature as well as the acid-base properties of polymers in order to develop polymeric lubricating systems and achieve optimum green properties and ejection performance. The selection of polymeric lubricants contained in the new HD system evaluated in this paper was based on these considerations as well as on their ability to be used in conditions that simulate most production applications in terms of compacting temperature and green density.

Compaction and Lubrication Behavior

Figures 5a, 5b and 5c present the compressibility of mixes containing either the new HD system or the EBS lubricating system measured on 0.64 cm thick TR bars compacted on a lab press at 45°C, 55°C and 65°C. The compacting pressure needed to press these mixes on a production press to a green density of 7.0 g/cm^3 at 55°C is also given in Figure 5b.

It is seen that the compressibility of mixes made with the new HD lubricating system is either similar or significantly better compared to that of the EBS containing mix at any compacting temperature from 45°C to 65°C. For example, the compacting pressure required to reach 7.0 g/cm^3 is equivalent regardless of the lubricating system for TR specimens pressed at these compacting temperatures and also for gears pressed on a production press at 55°C. Additionally, it is shown that the use of the new HD lubricating system significantly improves the compressibility of mixes when the green density increases from 7.0 to 7.2 g/cm^3 at any compacting temperature from 45°C to 65°C. For instance, when pressing TR specimens, the compacting pressure required to reach 7.2 g/cm^3 with mixes containing the HD system is 115 MPa lower compared to the conventional EBS wax containing mix at 45°C and also about 155 MPa lower at 55°C and 65°C.

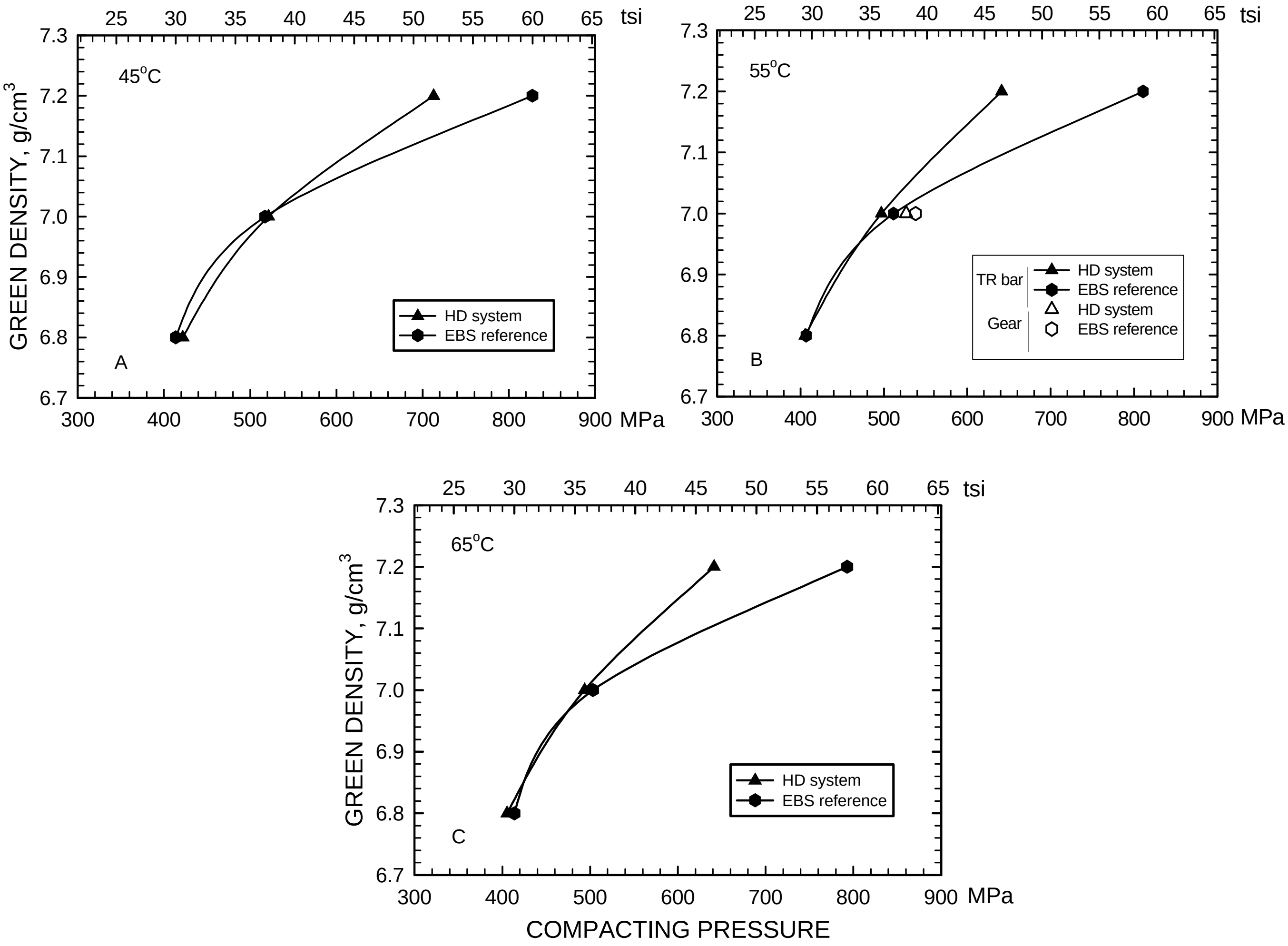

Figure 5. Compressibility of Mixes Containing HD and EBS Lubricating Systems on Lab and Production Presses at Various Compacting Temperatures.

With regards to the ejection performance, it is seen in Figures 6 and 7 that the HD system exhibits excellent lubricating properties. Indeed, the stripping pressure, which determines the shearing stress to start the ejection, is equivalent when using the new HD system at 45°C and also significantly lower at compacting temperatures of 55°C and 65°C (Figures 6a, 6b and 6c). For example, it is seen that the stripping pressure of 1.27 cm thick TR bars made from the new HD system is 35 and 25 % lower than with the EBS mix for specimens pressed to green densities of 6.8 and 7.0 g/cm^3 respectively at either 55°C or 65°C. With regards to the production of gears on a production press, the stripping pressure was reduced by 25 % when using the new HD system at 7.0 g/cm^3 and 55°C. The sliding pressure is equivalent for both the HD and EBS lubricating systems regardless of the compacting temperature, as shown Figures 7a, 7b and 7c.

Therefore, the powder mix containing the new HD lubricating system exhibits good compressibility and excellent lubricating performance on a lab press and also when compacting complex parts on a production press. This can be attributed to the good shear resistance during compaction along with superior lubricating properties of the various organic components in the new polymeric HD system compared to the conventional EBS wax.

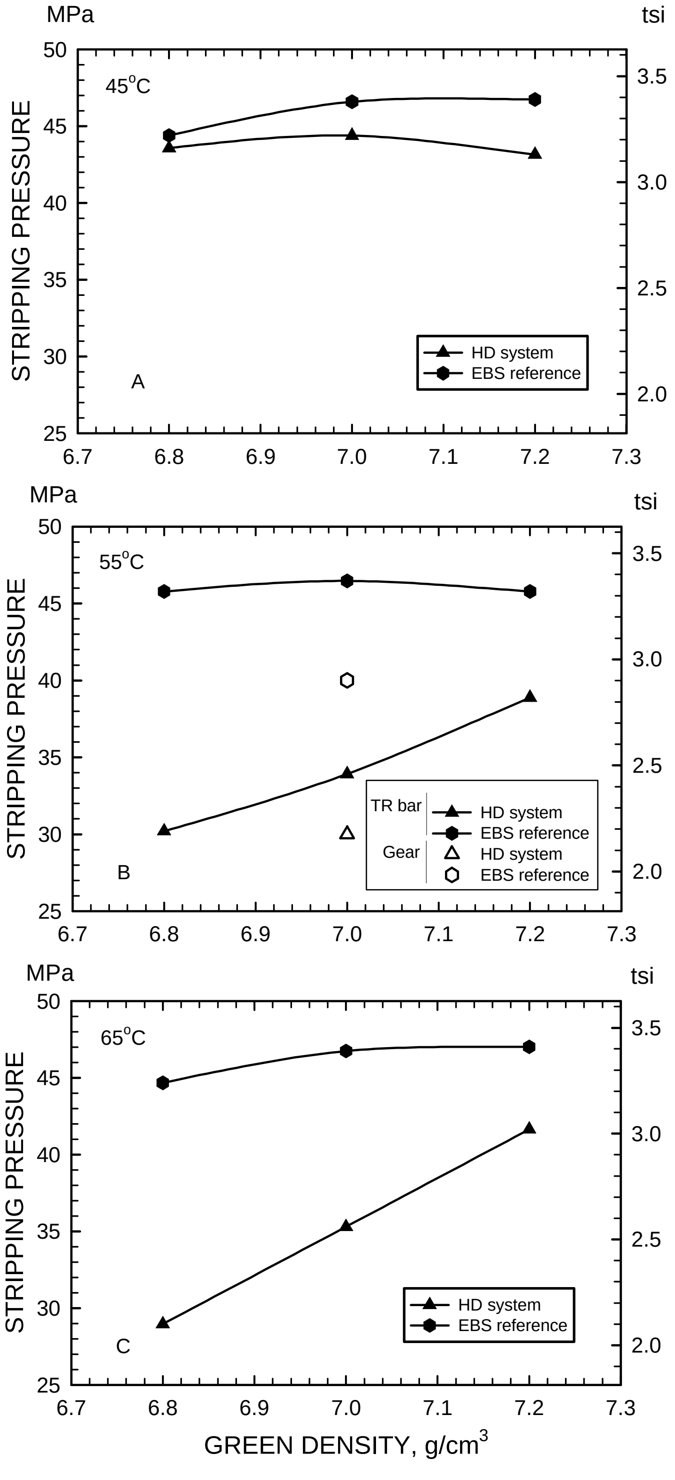

Figure 6. Stripping Pressure Required to Eject Specimens Containing the New HD Lubricating System or Conventional EBS Reference on Lab (TR bar) and Production (Gear) Presses at Various Temperatures.

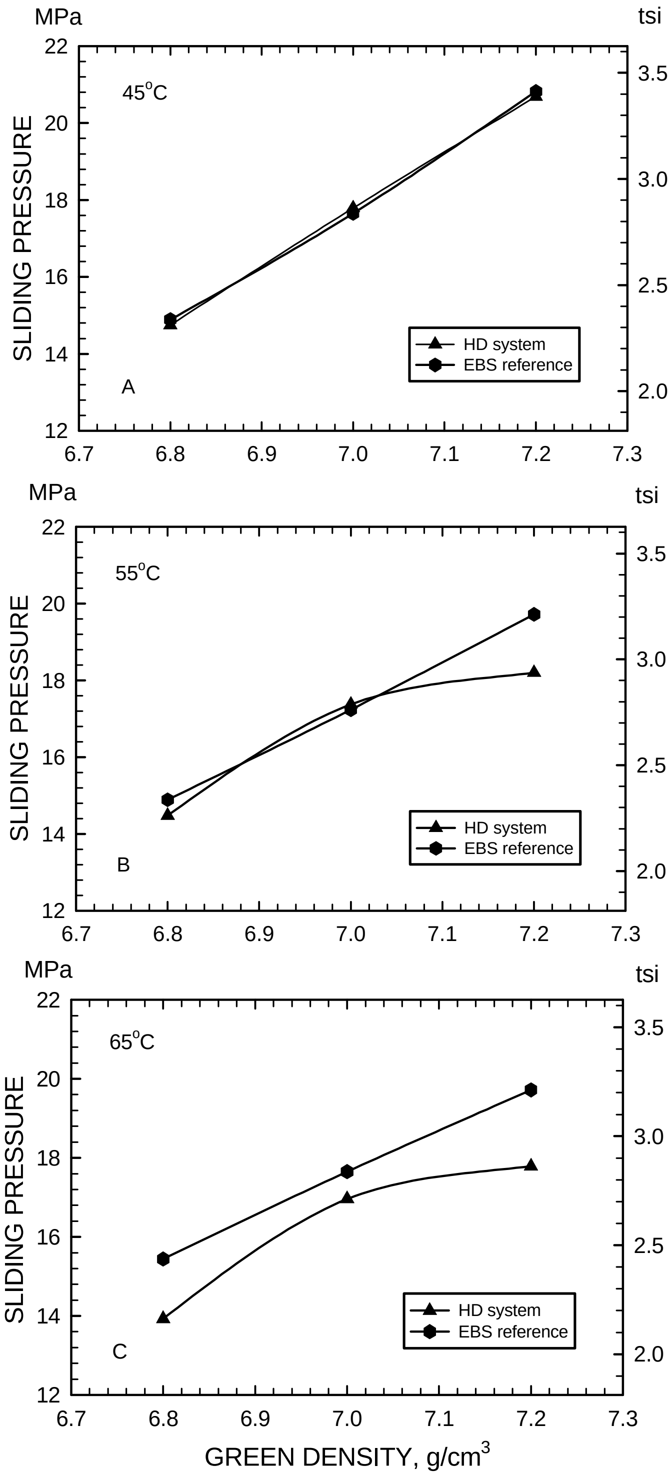

Figure 7. Sliding Pressure Required to Eject Specimens Containing the New HD Lubricating System or Conventional EBS Reference on a Lab Press at Various Temperatures.

Green Strength

Figure 8 compares the green strength of specimens made from the HD and EBS lubricating systems as a function of green density when pressed at 55°C. It is obvious that, whatever the green density, the strength values of 0.64 cm thick bars compacted from the HD system containing mix are significantly higher than those obtained with the EBS containing mix. Indeed, the green strength reaches 28, 35 and 43 MPa at 6.8, 7.0 and 7.2 g/cm^3 respectively, which is more than twice that values achieved with the conventional EBS system. The increase in green strength may be explained by the formation of stronger bonds between the steel powder particles mainly due to the formation of a strong and thin discontinuous film of polymer that has a good affinity with the powder particles.

Green strength also significantly improves by simply increasing the thickness of TR specimens from 0.64 to 1.27 cm (Figure 8). It should be noted that both 0.64 and 1.27 cm thick TR bars are recommended by MPIF for the evaluation of green strength (standard N^{o}15). In this study, the green strengths measured on 1.27 cm thick TR specimens containing the new HD system are 36 and 53 MPa at 6.8 and 7.2 g/cm^3 respectively for specimens pressed at 55°C. This represents an improvement of 23 to 32 % compared to green strengths measured on 0.64 cm thick TR specimens.

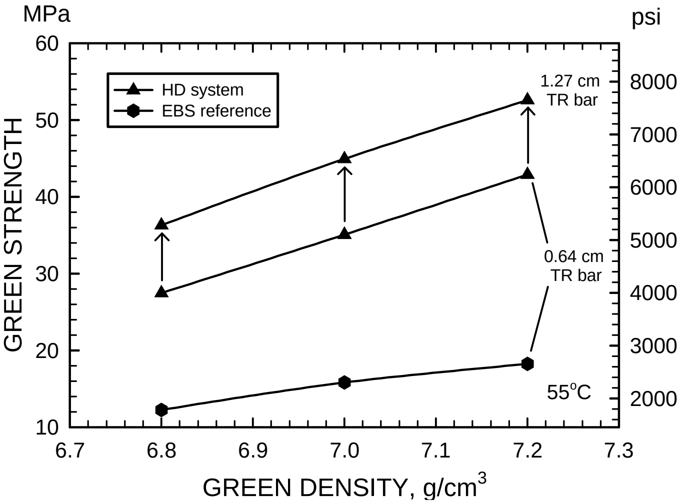

Figure 8. Green Strength of Specimens Pressed from the HD System and the EBS Reference as a Function of Green Density.

Finally, it is worth mentioning that the green strength of specimens containing the new HD system also noticeably improves as the compacting temperature increases from 45°C to 55°C and reaches a steady value above 55°C, as shown in Figure 9. Indeed, the green strength increases by about 40 % by raising the compacting temperature from 45 to 55°C for specimens pressed to 7.0 g/cm^3. However, it is interesting to notice that the green strength achieved at 7.0 g/cm^3 and 45°C is still sufficiently high to either prevent part failure during handling or enable machining operations in the green condition. In fact, this property easily attains

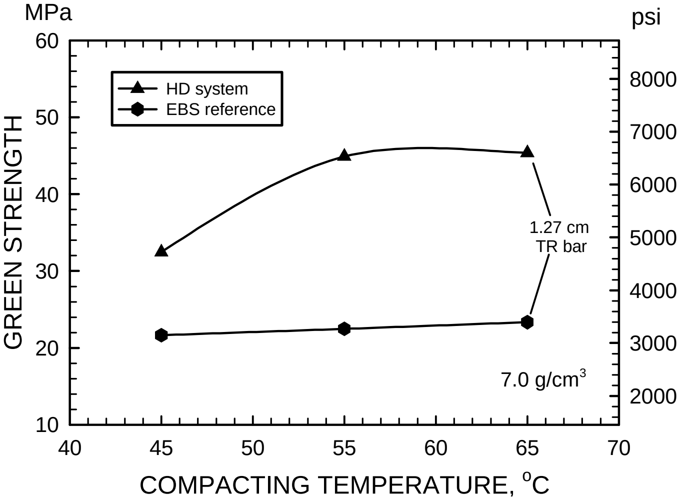

Figure 9. Green Strength of Specimens Pressed from the HD System and the EBS Reference as a Function of the Compacting Temperature.

about 32 MPa with 1.27 cm thick specimens pressed with the new HD containing mix in these compacting conditions.

Sintered properties

Sintered properties of specimens pressed with the new HD system containing mix are quite similar to those achieved with specimens pressed from the EBS wax system at 7.0 g/cm^3 and 55^{o}C, as shown in Table 2. The sintered density is 6.88 g/cm^3 for both lubricating systems, the sintered strength reaches about 928 MPa with the new HD system as compared to 907 MPa for the EBS wax reference, while the dimensional change and the apparent hardness remain in the +0.54-0.55 % and 73-74 HRB ranges.

TABLE 2
Sintered Properties of Specimens Compacted to 7.0 g/cm^3 at 55^{o}C with either the New HD Lubricating System or the Conventional EBS wax

LUBRICATING SYSTEM	**PROPERTY**			
	Sintered Density (g/cm^3)	Sintered Strength (MPa)	Dimensional Change (% from die size)	Apparent Hardness (HRB)
HD				
	6.88	928	+0.55	74
EBS wax				
	6.88	907	+0.54	73

CONCLUSIONS

This study was undertaken to evaluate the feasibility of using a new polymeric lubricating system to increase green strength and enable machining operations in the green condition. The polymeric lubricants comprised in this new system were selected based on their ability to strengthen the green compacts and also by taking into consideration their capacity to shear and lubricate parts during compaction and ejection in conditions similar to those found in actual production. The most relevant conclusions that can be drawn are summarized as follows:

- The development of polymeric lubricating systems have to take into account several intrinsic characteristics such as the molecular weight, softening temperature and/or particle size as well as the acid-base properties of the polymers to achieve, for given specific processing conditions, a compromise between the compressibility and lubricating properties of powder mixes.

- Mixes containing the new lubricating HD system exhibited either similar or better compressibility and lubrication behavior than mixes containing conventional EBS wax for TR bars and gear shape parts pressed in various compacting conditions (6.8 to 7.2 g/cm^3 at 45^{o}C to 65^{o}C) on both laboratory and production presses. The improvement of these properties as well as that of green strength can be explained by the presence of polar groups in the polymers included in the new HD system, which gives to the lubricating film a good affinity for both powder particles and die cavity.

- The green strength obtained with the newly developed polymeric lubricating system was significantly enhanced compared to that of conventional EBS wax lubricant. For instance, this property reached 32 and 45 MPa for 0.64 and 1.27 cm thick TR specimens respectively pressed to 7.0 g/cm^3 at a compacting temperature of 55°C. These values are more than twice as high as those achieved with 0.75 wt% EBS wax.

- The sintered properties of specimens pressed with the new HD system were similar to those obtained with the conventional EBS wax lubricant.

REFERENCES

1. M. Gagné and F. Chagnon, “Characterizing the Machinability of Green P/M Parts”, *Adv. Powder Metall. Particulate Mater.*, Vol. 3, Part 12, 1999, pp. 119-129.

2. F. Chagnon, L. Tremblay, S. St-Laurent and M. Gagné, “Improving Green Strength to Enable Green Machining”, *SAE Technical Paper No. 1999-01-0337*, Oct., 1998, pp.71-76.

3. L. Tremblay and Y. Thomas, “Enhanced Green Strength Lubricating Systems for Green Machining Ferrous Materials”, *Adv. Powder Metall. Particulate Mater.*, Vol. 1, Part 2, 1999, pp. 141-156.

4. S.H. Luk and A.B. Davala, “Enhanced Green Strength Material System for Ferrous and Stainless P/M Processing”, *Adv. Powder Metall. Particulate Mater.*, Vol. 5, Part 17, 1996, pp.127-152.

5. L. Tremblay, F. Chagnon, Y. Thomas and M. Gagné, “Green Machining of P/M Parts using Enhanced Green Strength Lubricating Systems”, *SAE Technical Paper No. 2001-01-0399*, March, 2001.

6. Y. Thomas, K.C. Cole, L. Tremblay, “Complexable Polymeric Binder-Lubricant Blends”, to be presented in PM^{2}Tech 2001, New Orleans.

7. M. Gagné, “Behavior of Powder Mix Constituents During Cold and Warm Compaction”, *Adv. Powder Metall. Particulate Mater.*, Vol. 1, Part 3, 1997, pp.19-33.

8. S. Turenne, C. Godère, P.E. Mongeon and Y. Thomas, “Evaluation of Friction Conditions in Powder Compaction for Admixed and Die Wall Lubrication”, *Powder Metall.*, Vol. 42, N^{o}3, 1999, pp. 263-268.

9. John A. Schey, *Tribology in Metalworkin - Friction, Lubrication and Wear*, American Society for Metals, Metal Park, Ohio 44073, 1983, pp. 54-56.

10. I.D. Robb, "Adsorption", in *Comprehensive Polymer Science*, C. Booth & C. Price Ed., Pergamon Press, Vol.2, 1989, pp. 733-754.

11. D. Briggs, D.G. Range, "Surface Properties", in *Comprehensive Polymer Science*, C. Booth & C. Price Ed., Pergamon Press, Vol.2, 1989, pp. 707-732.