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Design of High Efficiency Lubricants for Challenging PM Applications

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ABSTRACT

As powder metallurgy usage broadens, the pressure is on to deliver challenging parts with higher densities or more complex shapes. This, in turn, requires more sophisticated powder mixes and high performance lubricants. In the case of high-density parts, the lubricant content must be decreased in order to ease compaction and reach higher densities. This requirement calls for lubricants that can provide the same level of lubrication at much lower concentrations than the traditional lubricants. With more complex parts, including tall or multi-level parts, the sliding distance before parts are released from the die might increase, calling for a lubricant that offers superior lubrication. This paper exposes the development work that has been conducted on new lubricant formulations. It focuses mainly on the compaction and ejection performance of various lubricating systems designed around a high potential lubricant as evaluated with an instrumented industrial press at high shear rates. The results were compared to commonly used lubricants in the PM industry.

INTRODUCTION

The growth of the PM market is linked to a large extent to the production of more and more complex parts with improved sintered properties and higher densities in a cost competitive way. As an example, it is reported that the density of synchroniser hubs has increased from 6.9 g/cm³ in 2000 to 7.1 g/cm³ in 2010 [1]. However, compaction of parts with complex geometry, high aspect ratio and surface area in contact with the die or higher densities is more challenging and requires high performance powder mixes. On the other hand, parts manufacturers also require better surface finishes together with a reduced dependency on metal stearates or zinc-based lubricants. Many factors affect the compaction behaviour of pre-mixes but lubricant is by far one of the most critical ones. This explains why so much effort is devoted to the development of new lubricants with improved lubricating properties as compared to conventional ones (EBS waxes, Zn stearate and Kenolube).

The role of lubricants in powder metallurgy is complex. The lubricant must reduce the internal friction between the particles and at the die wall during compaction so that most of the energy is available to press the part rather than lost as thermal energy. It also plays a significant role during the ejection cycle, where performing lubricants will ease ejection therefore contributing to smoother surface finishes. The lubrication at the die wall is directly proportional to the amount of lubricant admixed in the powder. It is therefore not difficult to achieve better ejection characteristics by simply increasing the lubricant content. However, due to their very low specific gravity, the internal lubricant has also a strong effect on the maximum density that can be reached during compaction, which corresponds practically to about 98% of the theoretical pore free density of the mix. On the other hand, lubricant burn-off in the pre-heat zone of the sintering furnace is also affected by the density reached and the quantity of lubricant in the part. Sintered properties can also be negatively affected by high levels of lubricant. For these reasons, it is therefore critical to limit as much as possible the quantity of lubricant that is added to powder mixes. What is lost in quantity must therefore be compensated by a much higher efficiency, in order to maintain the compaction and ejection properties. It is not uncommon to add as little as 0.3 to 0.4% by weight of lubricant when densities in the vicinity of 7.35 g/cm^3 are targeted.

The properties of some experimental lubricants, characterised by lower softening point than EBS wax or Zn stearate, were described in several papers presented in the last decade [2, 3]. It has been shown that significant density gains can be achieved with such lubricants when low compacting temperatures – about 60 to 70°C – are used. These lubricants also provided very good lubrication during compaction and ejection, often comparable and sometimes even better than ultrafine EBS wax. The density gain obtained with these lubricants is somewhat linked to the migration of the lubricant out of the part and to the die walls during compaction. This most likely also explains the excellent lubricating behaviour obtained, even at relatively low lubricant concentrations. The migration of lubricant can also occur even after the parts are ejected from the die in industrial presses when higher compaction rates are applied. As such, it was shown that the amount of lubricant which is expelled out of the part may be as much as 25% of the initial lubricant content in the mix. Although providing excellent ejection properties, this excess lubricant can also have significant drawbacks under mass production conditions. As an example, residual powder can stick to the lubricant film and later sinter to the part, creating a surface defect. Therefore, composite lubricants with smaller amount of these new effective lubricants were developed and characterised. The properties of some of these composite lubricants were presented last year at the EPMA 2010 World Congress [4]. The excellent densification and ejection performance of these lubricants was confirmed on a lab-scale press. A full-scale industrial test on small, eccentric gears also provided excellent results. However, very little information on the ejection behaviour was obtained from this industrial test. The faster compaction rates used in the industry may affect the behaviour of the lubricant which would not be detected on slower lab-scale presses. It is therefore necessary to obtain data on the ejection behaviour on a large press to fully characterise and validate the effectiveness of such lubricants. This paper reports the most recent findings of a R&D program designed to characterise the behaviour and performance of several lubricating systems based on Lube HD-C with an instrumented mechanical press for densities ranging between 6.8 and 7.35 g/cm³. In addition to the compaction and ejection behaviour, the flowability, burn-off and sintered characteristics of these mixes are also presented.

EXPERIMENTAL PROCEDURES

The FD-2000 formulation from MPIF standard 35 was used for this work. ATOMET DB46, a diffusion-alloyed steel powder produced by Rio Tinto Metal Powders and containing 0.5%wt Mo, 1.75%wt Ni and 1.5%wt Cu was admixed with 0.3%wt natural graphite and various lubricant systems as described in Table 1. Mixes containing 0.7wt%Kenolube P11 and 0.7%wt ultrafine EBS wax were also produced as reference. Kenolube was selected since it is recognised as one of the most efficient lubricants in terms of ejection performance.

The compaction and ejection behaviour of mixes was first evaluated by pressing cylindrical slugs in an instrumented press model PTC-03DT. Cylindrical slugs 10 mm high were pressed at a compaction rate of 1 mm/s and 830 MPa. The slide coefficient during compaction and ejection stripping and sliding pressures were measured as described in by St-Laurent *et al*. [5].

The compaction and ejection behaviour of mixes under more standard compacting conditions were determined with a fully instrumented Gasbarre 150 tons mechanical industrial press at the NRC – Institute of Industrial Materials, Boucherville, Canada. Rings about 12.7 mm high were pressed in a 25.4 mm die at a stroke rate of 5 parts/min, which corresponds to a compaction rate about 50 times higher than that used with the PTC. The core pin diameter was 14.2 mm. Two different set of dies were used for these tests: WC-Co and Vancron 40. Vancron 40 is a Tool steel containing Cr-Mo-W-V and doped with nitrogen. Punches were made out of D2 steel. Compaction was done without heating the die and by heating the die at 60 or 80°C.

Mix Name	Lubricant system
Ref-K	*0.7% Kenolube P-11*
Ref-W	*0.7% Acrawax C atomized (EBS)*
HD-2/0.55	*0.55% Lube HD-C*
HD-2/0.45	*0.45% Lube HD-C*
HD-2/0.35	*0.35% Lube HD-C*
HD-3/0.55	*0.27% Lube HD-C + 0.28% Lube WP*
Mod Wax	*0.18% Lube HD-C + 0.52% EBS*

Table 1. Description of mixes prepared for this study

The burn-off characteristics of each lubricant were evaluated by Thermogravimetric analysis (TGA) with a Setaram apparatus model TGA-96. Tests were performed at 2°C/min in a re-purified argon atmosphere between 20 and 550°C (650°C for wax). The flow rate and apparent density was measured with a Hall flowmeter according to MPIF Standard 35.

Sintered properties were also evaluated. Standard TRS specimens were pressed at 7.2 g/cm³ and 80°C and sintered at 1140°C for 25 minutes in a 90% N_2 -10% H_2 atmosphere.

RESULTS AND DISCUSSION

1. Compaction and Ejection Performance

The excellent densification and lubrication behaviour of lube HD-C when pressed at temperature of 70°C or above and at relatively low compacting rate was already presented by St-Laurent et al. [4]. Table 2 shows the results obtained on the PTC (WC-Co die, 60°C and 830 MPa) for two references and mix HD-2/0.55. Mix HD-2 gave slightly higher green density than the two reference mixes. The lubrication was comparable to that of Kenolube and significantly better than that of mix with EBS wax even if the level of lubricant in mix HD-2 was 0.15% lower.

Mix Name	G. Density, g/cm³	Slide Coef.	Stripping P., MPa	Sliding P., MPa
Ref-K	7.29	0.75	24.5	12.5
Ref-W	7.29	0.66	40.2	19.8
HD-2/0.55	7.33	0.80	19.2	14.4

Table 2. Results of tests carried out with the PTC in a WC-Co Die at 830 MPa and 80°C.

As mentioned in the introduction, the aim of this work was the validation of the compaction and lubrication performance of Lube HD-C at higher compacting rate typical of the PM industry. The following paragraphs discuss the results obtained with a mechanical press.

<u>Compaction tests with Vancron 40 Die</u>

Figure 1 shows the compressibility curves of mixes pressed at different temperatures in a Vancron 40 die. At room temperature, mix Ref-K gave the best compressibility, about 0.05 g/cm³ higher than that of mix HD-2/0.55. However, this behaviour was inverted when the die was moderately heated to 60°C. Mixes containing lubricant HD-C gave slightly higher green densities than Ref-K at all pressures used. The density gain obtained with HD-2/0.55 and HD-3/0.55 when the die temperature was increased from room temperature to 60°C was quite significant, varying between 0.12 to 0.20 g/cm³. In comparison, the gain in density was only half that improvement for Kenolube.

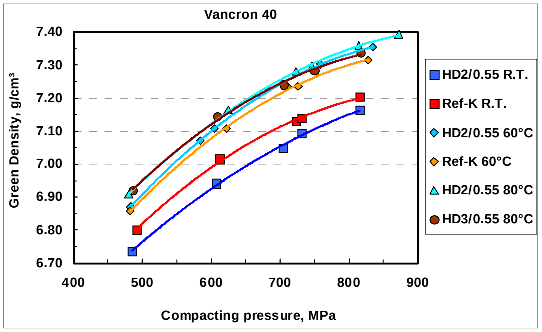

Figure 1. Compressibility curves obtained with a Vancron 40 die.

The maximum ejection pressure as a function of density and temperature is shown in Figure 2. At room temperature, mixes Ref-K and HD-2/0.55 gave very similar ejection values. Also, ejection pressure tends to level off at high compacting pressure for both mixes. Mix Ref-K gave slightly lower ejection values when pressed at 60°C, especially at high compacting pressures. However, a much more significant drop in ejection pressure was obtained with mix HD2/0.55. Even lower ejection values were obtained at 80°C. Mix HD-3/0.55, a composite lubricant (HD-C + WP) gave also very good ejection performance at 80°C. Results in Figures 1 and 2 clearly indicate the benefit of a moderate heating of the

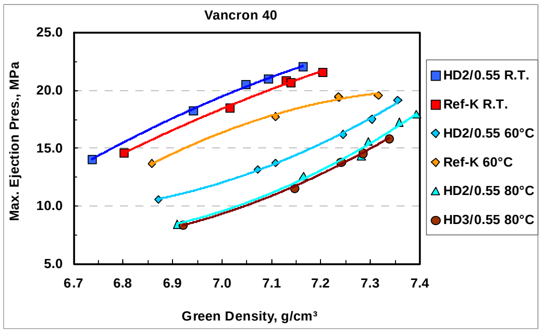

Figure 2. Ejection obtained with a Vancron 40 die.

die on the lubrication performance of lube HD-C.
The effects of reducing the level of lube HD-C on compressibility and ejection at 60°C are illustrated in Figure 3. As expected, no significant difference in density was obtained at relatively low compacting pressure. However, mixes with 0.35 and 0.45%wt Lube HD-C gave slightly higher density at higher pressures, densities up to 7.4 g/cm³ being achieved at ~ 880 MPa. On the other hand, ejection performance was quite decent, ejection values being only slightly higher compared to the mix with 0.55% Lube HD-C. Ejection pressures remained even lower than that achieved with Kenolube for densities varying between 6.8 and 7.3 g/cm³, even if the level of lubricant was significantly lower.

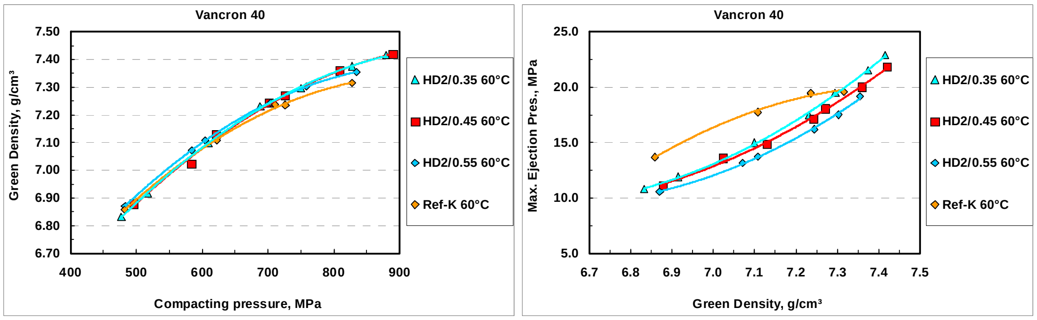

Figure 3. Compressibility and ejection of mixes containing lower levels of lube HD-C.

Compaction tests with WC-Co Die

Figure 4 gives the compressibility curves and maximum ejection pressure as a function of density for mixes pressed at 60°C in a WC-Co die. Note that not all the mixes were tested with the WC-Co because of time constraint. As it was the case with the Vancron 40 die, the compressibility of mix HD-2/0.55 with Lube HD-C was slightly better than that of mixes with Kenolube and EBS wax, especially at compacting pressures higher than 700 MPa. For instance, the gain in density obtained with HD-2 was typically 0.06-0.07 g/cm³ at ~ 800 MPa.

The benefits of using lubricant HD-C are clearly seen in Figure 4. Lubricant HD-C gave better ejection performance at compacting pressure below 800 MPa. Compared to mixes Ref-K and Ref-W, ejection pressures were between 7 and 12% lower. At compacting pressures over 800 MPa, however, the ejection pressure became more important than what was obtained with mix Ref-K. In particular, the ejection energy (not shown here) was 15% higher with mix HD-2/0.55 at higher compacting pressure when a green density of 7.3 g/cm^3 was reached. This result could be due to a smaller quantity of lubricant expelled to the die walls with the WC-Co compared with the Vancron 40 material discussed previously.

The compressibility and ejection of a mix containing a mixture of EBS wax and lubricant HD-C- referred to as Mod Wax, is also shown in Figure 4. Mod Wax was developed with the goal of providing excellent lubrication at relatively low cost. It can be seen that the compressibility of that mix is similar to that of mixes Ref-K and Ref-W. It is noteworthy to mention that this mix also gave very similar compressibility when compaction was performed at room temperature (not shown here). The ejection performance of mix Mod Wax was comparable to

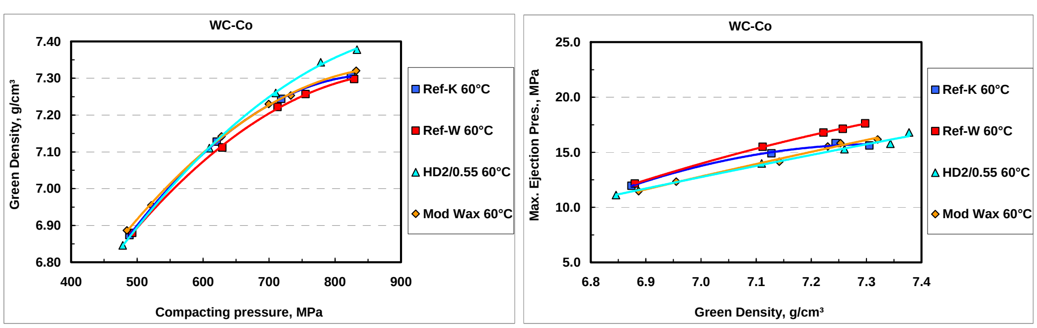

Figure 4. Compressibility curves and ejection of mixes pressed with a WC-Co die at 5 SPM.

that of a mix with 0.55% lubricant HD-C. It also offers a better performance compared to the performance of Ref-K (with 0.7% Kenolube) for densities at or below 7.2 g/cm^3. Similar to the behaviour of mix HD-2/0.55, the ejection pressure and ejection energy are more important when the compacting pressure reaches above 700 MPa. However, this lubricant system is a cost effective solution to Kenolube when green densities of 6.8 to 7.2 g/cm^3 are targeted, without the use of Zn stearate and its associated drawbacks.

In order to fully validate the ejection performance of these new lubricants, tests are planned with higher parts and faster compaction rates on the industrial press. These tests were not yet completed and results will be presented in an upcoming publication.

2. Flowability and Burn-off Characteristics of Lubricants

In addition to the lubricating behaviour, several other properties must be considered when designing a lubricant system for PM. One of them is the flowability. Lubricants must promote or maintain good flow properties in order to ensure stable die filling. This in turn helps in reducing the part-to-part weight variation and maintains the density consistent during compaction. The apparent density and flow rate of mixes investigated in this study are reported in Table 3. Mixes with lubricant HD-C gave very good flow rates of about 28 s/50g or below and apparent densities of about 3.20 g/cm^3. Mix HD-3/0.55 containing both lubricants HD-C and WP gave even better flow rates at 24.2 s/50g and apparent density in excess of 3.30 g/cm^3. It has been demonstrated by St-Laurent *et al*. [4] that the excellent flow rates achieved with mixes containing both lubricants HD-C and WP resulted in very good part-to-part consistency during compaction. In all cases, flow rates were better than those obtained with EBS wax. Mix Ref-K also gave a very good flow of ~ 25 s/50g. However, the flow rate of that lubricant is known to be affected by several factors, such as the blending time, blend size as well as the temperature and humidity during blending and compaction [6].

	Ref-K	**Ref-E**	**HD-2/0.55**	**HD-2/0.45**	**HD-2/0.35**	**HD-3/0.55**
A.D., g/cm³	*3.21*	*3.18*	*3.16*	*3.22*	*3.24*	*3.32*
Hall Flow, s/50g	*25.1*	*32.8*	*28.2*	*26.7*	*25.5*	*24.2*

Table 3. Apparent Density and flow rate of Mixes evaluated in that study.

Lubricants must also have very good burn-off characteristics. The ideal lubricant must burn-off entirely and easily and not cause harmful effects on the environment and the sintering furnaces. The burn-off behaviour of lubricants was verified using the TGA. The TGA curves are given in Figure 5. EBS wax (Acrawax C atomised), lube WP used for warm compaction and the new lubricant HD-C totally decompose without residues upon heating. The decomposition of lube HD-C also starts and ends at lower temperatures than the other lubricants. On the other hand, Kenolube and zinc stearate were not completely decomposed in the experiment. This result was expected due to the presence of zinc. As a matter of fact, approximately 90% and 84%, respectively of these lubricants were decomposed, leaving a significant amount of residues. This usually results in stains on the sintered parts.

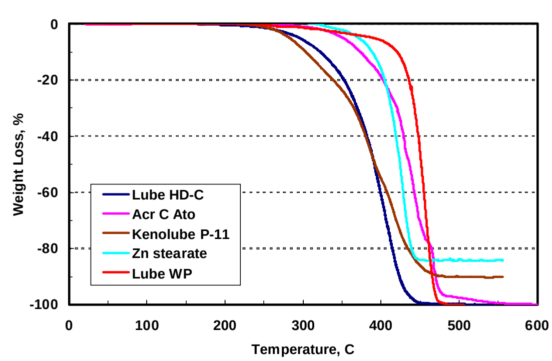

Figure 5. TGA curves obtained for different lubricants.

Table 4 gives the sintered properties of mixes as evaluated at 7.2 g/cm^3. These tests were done in order to confirm that new lubricant had no negative effect on sintered properties. All mixes gave very similar sintered strength and hardness. Dimensional change from green size varied from ~+0.03 to +0.08%. It can be observed that mixes HD-2/0.45 and HD-2/0.35 gave increased densities and sintered strength, likely due to the lower amount of lubricant used.

Mix	C. Pres., MPa	Springback, %	S. Dens. g/cm³	DC vs Green, %	TRS, MPa	App. Hard., HRB
Ref-K	*585*	*0.188*	*7.159*	*0.035*	*1100*	*78*
Ref-W	*581*	*0.164*	*7.161*	*0.040*	*1135*	*78*
HD2/0.55	*552*	*0.164*	*7.15*	*0.080*	*1070*	*77*
HD2/0.45	*552*	*0.164*	*7.153*	*0.077*	*1090*	*76*
HD2/0.35	*558*	*0.164*	*7.175*	*0.064*	*1135*	*76*
HD-3/0.55	*565*	*0.18*	*7.161*	*0.045*	*1140*	*77*

Table 4. Sintered properties of mixes pressed at 7.2 g/cm³ and 70°C.

Conclusions

The compaction and ejection behaviour of lubricating systems based on a new experimental lubricant called Lube HD-C, was evaluated mainly with an industrial press under typical pressing conditions used in the PM industry. In summary, the compressibility and ejection of lubricant HD-C was better than that of Kenolube and EBS wax when pressed at 60°C with two different die materials, the Vancron 40 and the WC-Co at compacting pressure of 700 MPa or below. Gain in density obtained with lubricating systems based on lubricant HD-C is mostly linked to the migration of the lubricant from the compact to the die walls during compaction. At higher compacting pressures, excellent ejection performances were maintained in the tool steel die while the ejection energy slightly increased with the carbide die, which could be due to lower amount of lubricant expelled to the die walls with the WC-Co die. Mixes containing as low as 0.35% Lube HD-C showed very good compressibility and ejection performance with the Vancron 40 die. A composite lubricant HD3 containing lower amount of HD-C lube also performed very well for a die temperature of 80°C. This lubricant also prevented excessive amounts of lubricant to be expelled out of the part. In order to fully validate the ejection performance of these new lubricants, tests are planned with higher parts and faster compaction rates on the industrial press. Results will be presented in another publication in the future.

In addition to the good lubricating behaviour, these new lubricant systems were shown to provide good flow characteristics. Tests done previously confirmed the excellent die filling performance and the very consistent part-to-part weight and density during compaction. This new lubricant also had very clean burn-off characteristics. This would prevent any adverse effect on the environment of the sintering furnaces. Finally, similar sintered strength and hardness were obtained when compared to reference mixes with Kenolube and EBS wax.

References

1 Wimbert, L.; Dollmeier, K.; Kruzhanov, V.; R. Lindenau, R., *Advanced Lubricants – A Complex Challenge for Powder Metallurgy: Density, Lubricity, Environment*, Proceedings of the 2010 PM World Congress in Florence, Italy, EPMA, October 2010.

2 St-Laurent, S.; Azzi, L.; Thomas, Y., *High Performance Iron Powder Mixes for High Density PM Application*, Proceedings of the 2006 Powder Metallurgy World Congress & Exhibition, Busan, Korea, Sept. 24-28, 2006; JPMA, pp. 3.1-3.13.

3 Azzi, L.; Thomas, Y.; St-Laurent, S., *Lubricants for high-density compaction at moderate temperatures*, Int. J. Powder Metall., 43 (6), pp. 36-46 (2007).

4 S. St-Laurent, S.; Wang, O.; Guo, R.; Zhang, K.; Cao, A., Achieving High Density By Single Compaction Of Steel Powder Premixes, Proceedings of the 2010 PM World Congress in Florence, Italy, EPMA, October 2010.

5 St-Laurent, S.; Chagnon, F.; Thomas, Y., *Study of compaction and ejection properties of powder mixes processed by warm compaction*, Adv. Powder Metall. & Particulate Mater., MPIF, pp. 3 79-3 91 (2000).

6 Thomas, Y.; St-Laurent, S.; Pelletier, S.; Gelinas, C., *Effect of Atmospheric humidity and temperature on the flowability of lubricated powder metallurgy mixes*, Adv. Powder Metall. & Particulate Mater., MPIF, USA, pp. 3.14-3.25 (2009).