Key Advantages of High Performance Lubricants for the Manufacturing of Powder Metallurgy Parts

Vincent Paris and Sylvain St-Laurent Rio Tinto Metal Powders Sorel-Tracy, Quebec, Canada J3R 4R4

Yannig Thomas National Research Council Canada (NRC) Boucherville, Quebec, Canada J4B 6Y4

Abstract

In powder metallurgy, the usage of an admixed lubricant to aid the compaction and to ease the ejection of the green parts is most of the time unavoidable. Over the past years, many lubricant formulations which were either developed by metal powder producers or independent lubricant manufacturers were introduced to the market. All the while, some common powder metallurgy lubricants are still widely used in the industry, mostly due to the fact that their behaviour is well known and are also cost competitive. Nevertheless, the actual offering of high performance lubricants offers many advantages over the traditional lubricants. This paper offers a review of the major advantages of high performance lubricants over traditional formulations.

The lubricants were evaluated in FC-0208 and FD-0208 steel powder compositions. Apparent density, flow rate, compressibility, ejection behaviour, surface finish and sintered properties were evaluated. It is demonstrated that high performance lubricants, despite their slightly higher cost compared to conventional lubricants, can offer net benefits to parts manufacturers. The lubricants were tested at equal concentrations (0.7% w/w) in order to offer a fair comparison and ease the process of selecting a given lubricant when designing new parts.

Although great care was taken to insure all lubricants were tested equally, it is understood that some lubricants may perform better than described herein in other compaction conditions or with different powder formulations.

Introduction

In order to manufacture high performance, value-added parts, it is often necessary to increase the green density, have intricate die shapes or use multi-level compaction tooling [1]. The benefits of the powder metallurgy processing route are becoming more obvious as the part complexity and mechanical properties are increasing. In turn, parts manufacturers require a metal powder feedstock with increased performance. The use of high performance lubricants serves just this purpose [2]. While no specific definition of the term high performance lubricant exists, it is generally accepted that lubricants providing superior ejection performance at similar loading levels, good surface finish and clean sintering while contributing to high mechanical properties would fall in this category. Other characteristics of a high performance lubricant could include a better die-fill behaviour, strong part-to-part weight stability and increased green density or the ability to lower the amount of lubricant added to the powder mix without degradation of the ejection performance.

In the past five years, several innovative lubricants have come to the market. Some of those developments were introduced by powder suppliers whilst other are being proposed by additive suppliers. In 2009, Höganäs AB introduced the Intralube which has been steadily gaining popularity [3] [4]. Hoeganaes Corporation also launched Ancormax 225, a lubricant designed for warm compaction, in 2013. Ancorlub W and Ancorlub X are other lubricants developed by this powder supplier [5]. Apex Advanced Technologies is a manufacturer of lubricants aimed at the powder metallurgy business and also has an offering suitable for high performance applications [6]. H.L. Blachford, a supplier of amongst others, zinc stearate and various lubricants, also offers Caplube L.

Although many options are currently available to parts manufacturers willing to embark on the endeavour of producing value added parts, it is somewhat difficult to compare the options offered by either the metal powder suppliers or the standalone additives suppliers on a fair basis. The task of identifying any benefit of using a high performance lubricant over traditional lubricants is consequently difficult.

This paper presents an in-depth evaluation of commercially available known high performance lubricants while also pointing out the advantages of such lubricants over more conventional ones. The lubricants were compared in two formulations using different types of raw powders: a water-atomized steel powder and a diffusion-bonded powder. To emphasize the advantages of the high performance lubricants presented, the results are compared to the well-known, broadly used Acrawax C.

Experimental Procedure

A total of ten, 22 kg (50 lb) samples were prepared with five different lubricants. Each lubricant was tested with two different base powders. The FC-0208 formulation [7] was prepared using ATOMET 1001, a water-atomized steel powder manufactured by Rio Tinto Metal Powders. ATOMET DB46, a diffusion-bonded grade from the same manufacturer, was used for the FD-0208 formulation. The samples

made from ATOMET 1001 were admixed with elemental copper (AcuPowder 165) and natural graphite manufactured by Timcal (PG-25). Both lubricants Apex Superlube were delivered pre-admixed with graphite. The Apex Superlube 50/50 is a mixture of 50% by weight of graphite (grade PM9) and 50% by weight of Superlube 1000b. Due to this characteristic of the Apex Superlube, twice as much lubricant (1.4% w/w vs 0.7% w/w) was added to mixes 4 and 9, which accounts for 0.7% w/w of lubricant and 0.7% w/w of graphite. The same principle applies for the Apex Superlube 67/28, which is a combination of 67% w/w of lubricant (Superlube 2.0) and 28% w/w graphite with the remainder a flowing agent of undisclosed nature. It must be kept in mind that due to the admixed graphite to the Apex Superlube 50/50, sample #9 has 0.1% extra graphite compared to the other DB46 based blends.

Mix	Base Powder	Copper (w/w %)	Graphite (w/w %)	Lubricant (w/w %)
1				0.7% Kenolube P11
2	_		0.8% Timcal PG-25	0.7% Acrawax C
3	ATOMET 1001	2.0% AcuPowder 165		0.7% Caplube L
4	_		0.1% Timcal PG-25	1.4% Apex Superlube 50/50
5	_		0.51% Timcal PG-25	1.04% Apex Superlube 67/28
6				0.7% Kenolube P11
7	_		0.6% Timcal PG-25	0.7% Acrawax C
8	ATOMET DB46	-		0.7% Caplube L
9	_		-	1.4% Apex Superlube 50/50
10	_		0.31% Timcal PG-25	1.04% Apex Superlube 67/28

 Table 1. Detailed composition of the powder samples prepared for this experiment.

The apparent density and Hall flow rate were evaluated according to MPIF Standard Test Methods 4 and 3, respectively [8]. Sintered properties were measured on MPIF Standard 41 TRS bars [8], pressed at a fixed compaction pressure of 827 MPa (60 tsi). The sintering was carried out in the Rio Tinto Metal Powders quality control furnace under a 90% $N_2 / 10\%$ H₂ atmosphere. The maximum sintering temperature was 1120°C (2048°F) with a holding time of a minimum of 25 minutes.

The ejection behaviour was evaluated at the National Research Council Canada (NRC) in Boucherville, QC, Canada, using a 150 mt Gasbarre mechanical press. The top and bottom punches of the press are equipped with strain gauges in order to monitor the compaction and ejection forces applied on the parts. The compacts are rings with a diameter of 25.4 mm (1.0 in), 12.7 mm (0.5 in) high and a core pin

measuring 14.2 mm (0.56 in) across. The compaction was performed at a stroke rate of five parts per minute. The warm die compaction technique was used. The tungsten carbide (WC-Co) die was heated to 60°C (140°F) and the powder was kept at room temperature. Each powder sample was tested at four different target compaction pressures: 482 MPa (35 tsi), 620 MPa (45 tsi), 717 MPa (52 tsi) and 827 MPa (60 tsi).

For each sample compacted, ejection curves which record the ejection force as a function of the elapsed time are produced by the press software. These curves are then analysed using an Excel-based software and four data points corresponding to important moments of the ejection process are extracted. Figure 1 shows a schematic of a typical ejection curve and demonstrates what data points are collected. The extracted data points are the stripping, sliding, out-of-die and maximum force, the latter being most of the time equal to the stripping force. The forces are converted to shear stresses through formula 1, where F is the force extracted from the ejection curve, h is the part height, d_o the outer diameter of the part and d_i the core pin diameter.

$$\tau = \frac{F}{\pi h(d_o + d_i)} \tag{1}$$



Figure 1. Representation of an ejection curve and the data extracted from it to describe the ejection behaviour.

The sintered aspect was evaluated on cylindrical slugs compacted at a green density of 7.1 g/cm³ and sintered in a Lindberg furnace. The sintering was carried out in a 90% N_2 / 10% H_2 atmosphere with a maximum heating rate of 70°C/min. The maximum sintering temperature was 1120°C.

Results and Discussion

Physical Characterisation

To manufacture complex or value added powder metallurgy parts, high apparent densities and fast flow rates are generally favoured. This is explained by many design factors such as shape complexity, thin walled parts or even density distribution. For example, tall parts require a long die fill. This, in turn, leads to long sliding distances while the part leaves the die. This long sliding, in theory, requires more lubricant than a short sliding distance would. Consequently, the higher the apparent density, the shorter

the sliding distance along the die. As an example, consider the theoretical parts detailed in Table 2. This example demonstrates that a 15% increase in the apparent density of the powder contributes to a 25% reduction in the sliding distance. In the same order of idea, tall die fills require a fast flow rate in order to maintain a high production rate. Furthermore, a faster flow rate promotes a better fill of the intricate sections within the die of thin-walled parts which leads to a more consistent density distribution in the finished parts. A high performance lubricant should therefore contribute to high apparent densities and fast flow rates.

Table 2. Theoretical sliding distances (in mm) for two parts of different heights, compacted using two different powder mixes at a green density of 7.2 g/cm^3

Green Part Height	Apparen (g/	nt Density ^(cm³)
(11111)	2.80	3.20
20	31.4	25
40	62.8	50

The Hall flow rate and apparent density are reported in Table 3. The fastest flow rates are obtained with both Kenolube and Caplube L. On a FC-0208 formulation with admixed copper, the flow rates range from 27.9 seconds per 50 g to a complete no flow. Acrawax C has the worst flow rate of all samples – besides Apex Superlube 50/50, which recorded no flow – and Apex Superlube 67/28 has a comparable flow rate to Acrawax C. All FC-0208 samples except the Apex ones had high apparent densities, above 3.0 g/cm³. The samples with the highest apparent densities are the Kenolube and Caplube L samples. The same tendency is observed for the FD-0208 formulations. Once again, the Caplube L and Kenolube samples exhibit the fastest flow rates with the highest apparent densities.

	-	-	-	
Mix	Base Powder	Lubricant	Apparent Density (g/cm ³)	Hall Flow Rate (s/50g)
1		Kenolube	3.22	28.5
2		Acrawax C	3.06	38.2
3	AT-1001	Caplube L	3.25	27.9
4		Superlube 50/50	2.88	No Flow
5		Superlube 67/28	2.80	35.3
6	DB46	Kenolube	3.26	26.9

Table 3. Apparent density and Hall flow rate results for all powder samples.

7	Acrawax C	3.14	34.6
8	Caplube L	3.24	26.0
9	 Superlube 50/50	2.94	No Flow
10	 Superlube 67/28	2.82	34.1

Compressibility

The benefits of using compressible metal powders are multiple. Probably the most notable advantage of highly compressible powders is the possibility of attaining higher green and sintered density through single pressing operations. Due to the low specific density of lubricants – all of them generally have a density in the vicinity of 1 g/cm³ – they occupy a large volume within the die cavity [5]. This greatly limits the achievable green density of any parts manufactured from lubricated powder mixes. Lubricants have intrinsic properties that will impact the compressibility of metal powders such as their melting point, chemical composition (which in turn impacts the melting point), particle size distribution and shape [9]. Figure 2 demonstrates the impact of the lubricant on the compressibility of the powder. For a FC-0208 material, Kenolube is the most compressible lubricant over the complete compaction pressure range. Acrawax C and Caplube L are comparable although Capluble L appears slightly less compressible than the EBS. The compressibility of Apex Superlube 50/50 is similar to Kenolube at higher compaction pressures, but below 52 tsi, it is less compressible. The Apex Superlube 67/28 is in between Kenolube and its counterpart, Superlube 50/50 up to 717 MPa (52 tsi) where it encounters a crossover point. At compaction pressures above 717 MPa (52 tsi), there is clear degradation in the compressibility from this lubricant.

Two lubricants appear to have better compressibility when admixed in a FD-0208 formulation, Acrawax C and Kenolube. While other lubricants achieved green densities of about 7.20 g/cm³, these two respectively reached 7.24 g/cm³ and 7.23 g/cm³. Both Apex Superlube lubricants have comparable compressibility curves to all the other lubricants up to about 744 MPa (54 tsi), after which they depart from Kenolube and Acrawax C. Caplube L has a similar behaviour to the Apex lubricants but differentiation occurs at a lower compaction pressure, 717 MPa (52 tsi).



Figure 2. Compressibility curves for FC-0208 (A) and FD-0208 (B) powders.

Ejection Behaviour

Perhaps the most important characteristic of a high performance lubricant is its impact on the ejection process itself. Whilst more conventional lubricants – for instance, EBS wax – would suffice to compact many lower density, short parts; as soon as the parts to be compacted are more complicated, the limits of conventional lubricants are soon reached. In order to achieve higher green and sintered densities, a high performance lubricant should be capable of lubricating as well as conventional lubricant at lower loading levels [2]. The flipside of this affirmation – and the basis for this experiment – is that at similar concentration, parts made from a powder mix containing a high performance lubricant should be ejected more easily from a die cavity. The following graphs show the recorded stripping, sliding and out-of-die shear stresses for all samples, at various compaction pressures.

In the case of the FC-0208 formulation, shown in Figure 3, there is a definite difference between almost all the lubricants tested which is mainly visible at high compaction pressures. Acrawax C, without much surprise, has the highest shear stresses at all the ejection periods (stripping, sliding and out-of-die). Caplube L and Kenolube are somewhat similar but at the higher densities, Kenolube appears just slightly inferior to Caplube L. Differences were also observed in the two versions of the Apex Superlube lubricant. The Apex Superlube 50/50 falls in between the Acrawax C and the Caplube L and Kenolube. Apex Superlube 67/28 is much closer to the Kenolube and Caplube L.

Figure 4 shows a sample of an ejection curve from all the FC-0208 samples tested. The curves, obtained from parts which were all compacted at a targeted compaction pressure of 827 MPa (60 tsi), demonstrate the differences between the lubricants. Kenolube and Caplube L appear to have similar ejection behaviour since the curves are almost overlapping. Both Apex lubricants are more difficult to eject than Kenolube and Caplube L.

Presented at the 2014 World congress on Powder Metallurgy & Particulate Materials Powder held in Orlando, USA in May 2014 and published in the Advances in Powder Metallurgy & Particulate Materials-2014 conference proceedings available from the Metal Powder Industries Federation.



Figure 3. Stripping (A), sliding (B) and out-of-die (C) shear stresses for the FC-0208 formulation (AT-1001).



Figure 4. Sample of an ejection curve for each of the lubricants tested with the FC-0208 (AT-1001) formulation. (Curves were manually offset to avoid overlapping)

The behaviour of all the lubricants appears to be different in a FD-0208 formulation. Most lubricants have ejection shear stresses which are higher than with the FC-0208 powders. Figure 5 shows the same

data points as Figure 3 but for the FD-0208 samples. To ease comparison between the two data sets, all scales of the graphs were kept equal. Furthermore, especially at the lower compaction pressure, there is little differentiation between all the lubricants other than Acrawax C. Some differences are arising at the targeted compaction pressure of 717 MPa (52 tsi) and above. While Apex Superlube 67/28 was clearly superior to its other version, the Apex Superlube 50/50 with a FC-0208 formulation, this is no longer the case using a FD-0208 powder. For those two high performance lubricants, the stripping and sliding shear stresses are very comparable throughout the entire compaction pressures range. A difference was measured in the out-of-die shear stresses where the Apex Superlube 67/28 had lower values than the Apex Superlube 50/50. This phenomenon is clearly visible in Figure 6 where the Apex Superlube 67/28 curve appears to be below the curve of the Apex Superlube toward the end of the ejection.



Figure 5. Stripping (A), sliding (B) and out-of-die (C) shear stresses for the FD-0208 formulation (DB46).

The reasons which can explain the different behaviour of high performance lubricants with a FD-0208 formulation are not known. It is possible that a different compaction behaviour of the diffusion-alloyed powder (deformation rate, porosity network development) may cause a varying amount of lubricant to be forced out from the part to the die walls. This, however, was not verified in this experiment.



Figure 6. Sample of an ejection curve for all the lubricants tested with the FD-0208 (DB46) formulation. (Curves were manually offset to avoid overlapping)

Green and Sintered Properties

For all lubricants tested, the compaction was performed at a fixed compaction pressure of 827 MPa (60 tsi). This methodology has the advantage of taking into account the compressibility of the powder mixes. However, it also underestimates the mechanical properties of less compressible samples, as many sintered properties are closely correlated to the sintered density. The results based on ATOMET 1001 are shown in Table 4 where the lubricants getting the best results are highlighted in blue while the least desirable results appear in red. For FC-0208 samples, the highest green density, 7.26 g/cm³, was obtained with Kenolube but other lubricants such as Acrawax C and Caplube L have very comparable green densities of 7.24 g/cm³. The Apex Superlube family trails behind with values of 7.19 and 7.16 g/cm³. Springback, which is a plastic deformation occurring after the pressure is released from the compact, appears also significantly higher for the Apex Superlube lubricants with values about 0.03% higher than the other lubricants. In a high performance lubricant, high springback is not favoured as it can lead to a lower final density and can also cause green cracks to appear. In terms of green strength, Kenolube and Caplube L have similar results, about 2100 psi while the Apex series has green strengths of about 1660 psi. The dimensional change, also important in the manufacturing of tight dimensional tolerance parts is also higher for both the Apex Superlube 50/50 and Superlube 67/28. Finally, the transverse rupture strength (TRS) varies greatly from one lubricant to another. In this case, Acrawax C obtains the best result.





Table 5 shows the same set of results but for the FD-0208 samples. The main trends remain unchanged from a FC-0208 to a FD-0208 formulation. For instance, the maximum green densities were again obtained with the Acrawax C, Kenolube and Caplube L samples. With the DB46 powder, Acrawax C has the highest green density, 7.32 g/cm³ compared to about 7.28 g/cm³ for both the Kenolube and Caplube L. In this case again, the Apex lubricants Superlube 50/50 and Superlube 67/28 offer the lowest green densities with respective values of 7.20 g/cm³ and 7.17 g/cm³. The highest green strength, 2629 psi was obtained with Kenolube. While most lubricants saw an increase in the green strength from a FC-0208 to a FD-0208 formulation, both Apex Superlube samples remained at values close to 1660 psi. The largest growth from die size was this time obtained with the Apex Superlube 67/28. Contrary to a FC-0208 powder mix, Kenolube has the largest growth from green size, 0.037% when admixed to a diffusion alloyed powder. All the lubricants had higher transverse rupture strength with the FD-0208 formulation but the tendency remained the same with Acrawax C the most resistant.





Sintering Aspect

While the esthetic aspect of sintered parts has virtually no effect on the mechanical properties which are obtained, it remains nevertheless an important property and stains-free parts are highly desirable. One reason which explains this is the automated quality control of parts using high definition imaging. This technique can lead to stained yet totally good parts to be discarded thereby increasing the scrap rate. Table 6 shows the appearance of small slugs compacted from mixes 1 through 10 and sintered under a standard atmosphere. Both Kenolube and Caplube L have formed stains. In the case of Kenolube, the staining is believed to originate from zinc stearate which is a component of the lubricant. The cause for stains formed with Caplube L can possibly be linked to the presence of metallic salts [10]. The cleanest parts were obtained with Acrawax C. Ethylene bi-stearamide (EBS), the main constituent of Acrawax C is known for its clean decomposition in sintering furnaces and this result was expected. Both Apex Superlube lubricants caused staining but in a lesser extent compared to Caplube L and Kenolube. It must be noted that no impact of the powder composition (FC-0208 vs FD-0208) was observed on either the presence or the severity of stains.





50/50	67/28	
-------	-------	--

Conclusion

The aim of this paper was to produce a review of commercially available high performance lubricants, demonstrate their key advantages and compare those with a well-known and widely used lubricant, in this case EBS wax Acrawax C. The lubricants were evaluated using two different powders, an unalloyed steel powder (ATOMET 1001) in a FC-0208 formulation and a diffusion-alloyed powder (DB46) in a FD-0208 formulation. The samples were evaluated on an array of characteristics which should be sought after in a high performance lubricant. The apparent density, flow rate, compressibility, ejection behaviour, green and sintered properties and sintered aspect were evaluated.

In order to offer a fair comparison, the lubricants were tested at a concentration of 0.7% by weight. This amount of lubricant is quite common in the ferrous powder metallurgy community. It is assumed that the relationship between the ejection forces recorded on the press and the amount of lubricant in a powder mix is inversely proportional. That is, as the concentration of lubricant is increased, the ejection forces required to eject the parts should decrease. However, it is possible that some lubricants will perform better or behave differently at higher or lower loading levels. Different concentrations of lubricant were not evaluated in this study. Furthermore, all compactions were completed with a die heated to 60°C. Some lubricants may have performed differently at other compaction temperatures.

Furthermore, it must be noted that the Apex Superlube lubricants are provided already admixed with some graphite. The Apex Superlube 50/50 contains 50% w/w lubricant and 50% w/w graphite while this proportion is 67% w/w lubricant and 28% w/w graphite in the Apex Superlube 67/28. Due to this characteristic, one sample (mix 9) contained 0.1% w/w more graphite than the other four FD-0208 samples. Some variation, especially in the sintered properties, may arise from this difference.

It was demonstrated that Kenolube and Caplube L are very comparable lubricants in terms of apparent density, flow rate, ejection behaviour and sintered aspect. Some differences were observed in the compressibility, where Kenolube remains superior and in the sintered properties. Both Apex Superlube lubricants cannot achieve densities as high as the other two lubricants tested, at a loading level of 0.7% w/w. They both exhibited lower apparent densities and slower flow rates. Their sintered aspect, however, appeared superior to Kenolube and Caplube L. In terms of parts cleanliness, Acrawax C remains a very attractive choice, producing stain-free parts in standard sintering conditions.

Acknowledgments

The authors would like to thank Olivier Bouchard (RTMP), Ian Bailon-Poujol (RTMP) and Patrick England (NRC-Canada) for their contribution to this study.

References

- 1 Whittaker, David. High Density, High Performance Powder Metallurgy Components. *International Powder Metallurgy Directory*, 15 (2012-2013), 67-74.
- 2 St-Laurent, Sylvain, Paris, Vincent, and Thomas, Yannig. Performance of Lubricants for High Density Applications. In *2012 Powder Metallurgy World Congress and Exhibition* (Yokohama (Japan) 2012), Japan Society of Powder and Powder Metallurgy.
- 3 Ahlin, Åsa, Ahlqvist, Anna, and Litström, Ola. Newly Developed Lubricants for High Performance Powder Mixes. In *Euro PM2008 Congress and Exhibition* (Mannheim (Germany) 2008), European Powder Metallurgy Association, 205-210.
- 4 Larsson, Mats, Allroth, Sven, Dahlberg, Mikael, and Knutsson, Per. Selection of Lubricants and Mixes for Efficient Production of High Performance PM Components. In *EuroPM 2013* (Göteborg (Sweden) 2013), European Powder Metallurgy Association, 151-156.
- 5 McQuaig, Kylan, Schade, Chris, and Sokolowski, Peter. Development of a Lubricant System for Improved Performances of Premixes. In *International Conference on Powder Metallurgy and Particulate Materials* (Chicago, IL (USA) 2013), Metal Powders Industries Federation, 46-59.
- 6 Hammond, Dennis. Advanced Lubrication Technology. In 2013 International Conference on Powder Metallurgy and Particulate Materials (Chicago, IL (USA) 2013), Metal Powder Industries Federation, 27-44.
- 7 METAL POWDER INDUSTRIES FEDERATION. *Materials Standard for PM Structural Parts*. Metal Powder Industries Federation, Princeton, NJ (USA), 2012.
- 8 METAL POWDER INDUSTRIES FEDERATION. *Standard Test Methods for Metal Powders and Powder Metallurgy Products*. Metal Powder Industries Federation, Princeton, NJ (USA), 2012.
- 9 Thomas, Yannig, Bernier, Fabrice, St-Laurent, Sylvain, and Paris, Vincent. Effect of Lubricant Particle Size Distribution on the Processing and Properties of P/M Ferrous Parts. In *EuroPM 2013* (Göteborg (Sweden) 2013), European Powder Metallurgy Association, 157-162.
- 10 H.L. BLACHFORD. Caplube L WHMIS Material Safety Data Sheet. 2009.