Presented at the Euro PM2013 congress held in Gothenburg, Sweden in September 2013 and published in the congress conference proceedings available from the European Powder Metallurgy Association (EPMA).

Evaluation of Innovative High Performance Lubricants for Compaction of Complex Powder Metallurgy Parts

V. Paris[1], Y. Thomas[2] and S. St-Laurent[1]

[1]*Rio Tinto Metal Powders, 1655 Marie-Victorin Rd, Sorel-Tracy, QC, Canada J3R 4R4*
[2]*National Research Council Canada, 75 De Mortagne Blvd, Boucherville, QC, Canada J4B 6Y4*

Abstract

A clear trend in the PM community for the past years is the development of increasingly more complex parts. Multi-level, tall and high green density parts are favoured in order to obtain the best mechanical performance while keeping the manufacturing costs as low as possible. To some extent, such advances are dependent on the metal powder feedstock. High performance lubricants added to the metal powder mix should, amongst others, promote a superior die-fill and an easier ejection from the die.

This article evaluates the behaviour of lubricant PR-2, a high performance lubricant developed by Rio Tinto Metal Powders. A water-atomised steel powder mix containing 2.0% copper, 0.55% graphite and variable amounts of the high performance lubricant was tested on an industrial press. The press was run in fully automatic mode during extended periods of time and the part-to-part weight variation was measured. The powder was also characterised for its die-filling behaviour. The lubricant is compared to other commonly used PM lubricants such as Acrawax C and Kenolube.

Introduction

In the powder metallurgy manufacturing process, lubricants are used in order to reduce the inter-particular friction during compaction and also the friction between the powder particles and the die itself. Up to this day, and despite many efforts to concentrate the lubricants at the die wall, the use of admixed lubricant can rarely be avoided. Admixed lubricants however have a significant drawback when higher densities must be achieved, or when complex parts require even density distribution. Since the specific density of most lubricants is much lower than the powder they are admixed to, the lubricants will occupy a larger volume fraction in the mix. This greatly reduces the green density at which parts can be compacted [1]. For parts with narrow sections, this could lead to a locally reduced density. Other techniques such as the die wall lubrication were developed to allow for a reduced usage of admixed lubricants. Results have shown that the quantity of admixed lubricants can indeed be lowered but that parts with a complex geometry still require admixed lubricants [2]. Furthermore, the die wall lubrication technology, although its efficiency is well demonstrated, remains complex and its commercial usage still limited [3].

Not surprisingly, considerable research and development efforts are still being pursued in the field of lubrication. In the last five years, several papers have described novel lubricants which promote higher densities and reduced ejection forces [4] [5]. In a previous publication, *Paris et al*. demonstrated that the proprietary lubricant PR-2, developed by Rio Tinto Metal Powders, provided better ejection behaviour for short (12.7 mm) cylinders both when compacted at room temperature and with the warm die compaction technique [5]. In a subsequent paper, it was demonstrated that this lubricant maintained its edge over conventional lubricants even for taller parts (25.4 mm) and at higher stroke rates [6].

This paper now focuses on the performance of this new family of lubricants under industrial-like conditions. Different powder mixes containing various formulations of lubricants were compacted on a 150 mt mechanical press in automatic mode for extended periods of time. The objective is to demonstrate that this new lubricant can provide a better part-to-part consistency than conventional lubricants currently used in powder metallurgy.

Experimental Procedure

Powder mixes weighing approximately 110 kg each were prepared from ATOMET 1001, a water-atomised steel powder manufactured by Rio Tinto Metal Powders. All four samples were prepared by admixing 2.0 wt% elemental copper (Acupowder 165), 0.55 wt% graphite (Timcal F-10) and variable amounts of lubricant PR-2. This lubricant is a proprietary, fatty-acid based lubricant recently developed by Rio Tinto Metal Powders. The exact composition of the samples is provided in Table 1. The two samples containing Acrawax C atomized (EBS wax) and Kenolube serve as benchmarks.

Table 1. Detailed composition of the powder mixes used in long compaction sequences.

Mix	Base Powder	Copper	Graphite	Lubricant
1	AT-1001	2.00% Cu165	0.55% F-10	0.7% PR-2
2				0.6% PR-2
3				0.8% Kenolube
4				0.8% Acrawax C

The apparent density and the flow rate of each sample were evaluated according to the MPIF Standard 4 and 3, respectively [7]. Although the Hall flow rate as evaluated in the MPIF Standard 3 is a useful and common way to rate the flow behaviour of a sample mix, it fails to clearly establish how the powder flows into a die. To complete the Hall flow rate measurement, each powder mix was submitted to a die-fill evaluation. This test is performed on an in-house rig composed of ten small rectangular die cavities of increasingly smaller width. The cavities are placed such that their longer side is in-line with the die-fill direction. The rig is equipped with a variable speed shoe which automatically dispenses powder to fill each die cavity. Once the shoe has completed its course over the length of the rig twice, the powder in each die cavity is emptied and weighed. The test is repeated 10 times over for each powder mix. Reported results include the weight dispersion from the 10 tests as well as the in-die bulk density. In this experiment, the shoe was run twice over the cavities and the shoe speed was set at 8 cm s^{-1}.

Long compaction sequences were performed on a 150 mt Gasbarre mechanical press at the National Research Council Canada (Boucherville, Canada). This production press is equipped with strain gauges able to monitor the forces on both the upper and lower punches. The parts pressed were cylindrical slugs with an outer diameter of 25.4 mm and a core pin diameter of 14.2 mm. The parts were 25.4 mm high with an M/Q ratio of 9.07. The M/Q ratio is the ratio of the surface in contact with the die over the projected area of the part [8]. The compaction was completed at a stroke rate of 10 parts per minute with a WC-Co die heated to 60°C at a targeted compaction pressure of 485 MPa. For each powder mix tested, about 1000 parts were pressed under steady state conditions. Prior to beginning each test sequence, the die fill of the press was adjusted such that the green parts were 25.4 mm tall at the targeted compaction pressure.

Throughout each sequence, parts were sampled at regular intervals to measure their weight, temperature, height, green density and radial springback. Following the compaction sequences, for some of the collected parts, exact dimensions were measured using a SmartScope Flash 300 CMM with an accuracy of 1.5 µm. From these dimensions, the radial springback and the green density were calculated.

Table 2. Sampling and measurements frequencies of the parts produced during the long compaction sequences.

Sampling Frequency	n	Measurements			
		Weight	Temperature	Dimensions	Green Density
Every 10 parts	~100	X			
Every 30 parts	25			X	X (from CMM)
Every 50 parts	20		X		X (water displacement)

Results and Discussion

Table 3 provides the apparent density and the Hall flow rate measured on each mix. Kenolube is the lubricant which provides the second highest apparent density and the faster flow rate. This is no surprise, as Kenolube contains zinc stearate. Metal stearates are known to be excellent flow enhancers [9]. Mixes 1 and 2 (containing 0.7 wt% and 0.6 wt% PR-2) show excellent apparent densities which are close to, and for Mix 2, even higher than Kenolube. The flow rates of Mixes 1 and 2, albeit slower than the Kenolube-based mix, are definitely improved when compared to the Acrawax reference mix.

Table 3. Apparent density and Hall flow rate for all mixes tested in the long run sequences.

Mix	Lubricant Content	Apparent Density	Hall Flow Rate
	(wt %)	($g{\cdot}cm^{-3}$)	($s{\cdot}50g^{-1}$)
1	0.7% PR-2	3.15	30.5
2	0.6% PR-2	3.21	29.8
3	0.8% Kenolube	3.16	26.8
4	0.8% Acrawax C	3.10	34.0

Figure 1 shows the die-fill behaviour for all mixes. The die-fill is evaluated by measuring the mass of powder accumulated after one test in each die cavity, once the feed shoe has filled them. The standard deviation (σ) is calculated and divided by the average weight (μ) according to Equation 1, which provides the relative standard deviation (RSD).

$$\%RSD = \frac{\sigma}{\mu} \times 100 \qquad (1)$$

The in-die apparent density is the actual weight of powder collected from each cavity over the cavity volume. Mixes 1 and 2, both containing the PR-2 lubricant have much lower relative standard deviation for the narrower die cavities. The relative standard deviation for the 3 cm^3 die cavity is only 0.17% and 0.23% for both Mixes 1 and 2, respectively. This compares to about 0.28% for the Kenolube mix and 0.30% for the Acrawax C mix. The lower relative standard deviation for narrow die cavities suggests that the lubricant PR-2 can provide more stable parts in serial production, particularly if the part design includes narrow sections.

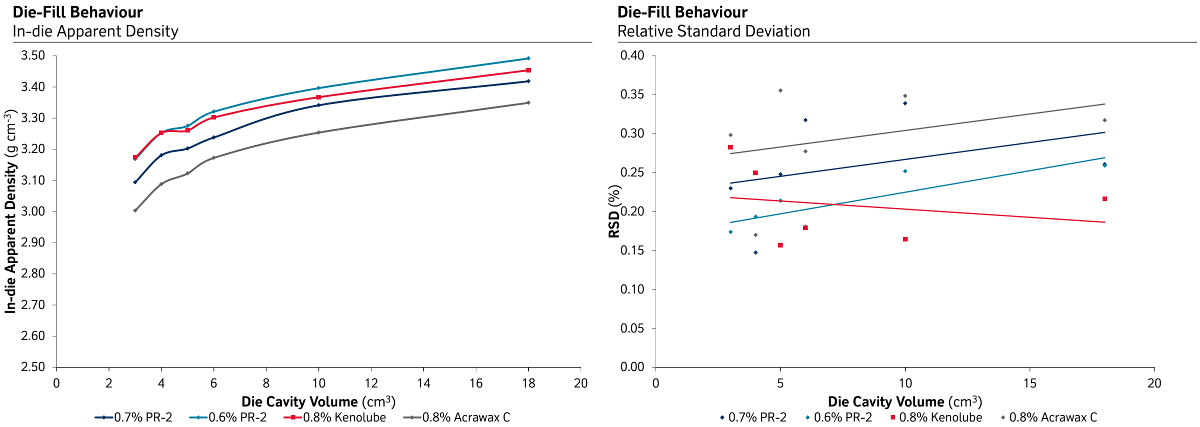

Figure 1. Die-fill behaviour of the various mixes.

Due to the differences in the apparent densities shown in Table 3 and to varying compressibility of each powder mix, it was necessary to adjust the die fill of the press in order to obtain parts that are as close as possible to the desired 25.4 mm height, while maintaining the targeted compaction pressure of 485 MPa. This procedure had an effect on the weight of powder filling the die, and consequently on the final parts weight. As demonstrated in Table 4, the die-fill height was the lowest for Mix 2 (0.6% PR-2) and the highest for the Acrawax mix. Mixes 1 (0.7% PR-2) and 3 (0.8% Kenolube) were compacted using very similar die-fill heights, yet slightly higher for mix 3. As a consequence, parts pressed with mix 3 are slightly heavier than the parts made from mix 1. The average compaction pressure varies for each mix. As a general rule, the more the average compaction pressure departs from the targeted compaction pressure, the less stable are the parts weight. This is a consequence of the instability of the powder mix to provide consistent parts, stroke after stroke. This is particularly obvious for Mix 4 (0.8% Acrawax C) where the average compaction pressure is much higher than the targeted compaction pressure. This implies that as the press was running on the Acrawax mix, unstable die-filling occurred. The deteriorating die-fill is clearly shown in Figure 2. As the press keeps running, the powder mix containing Acrawax C produces increasingly heavier parts. This phenomenon occurred for mix 2 as well, although it was much less severe. This is confirmed by the slopes of the linear regression curves shown in Figure 2 which are negative, yet very close to zero for mixes 1 and 3. Mix 4 has a much steeper slope, about four times as much as mix 2.

To effectively compare each lubricant against one another, the normal distribution density function of the parts weight distribution was obtained, using Equation 2.

$$f(x,\mu,\sigma) = \frac{1}{\sqrt{2\pi}\sigma} e^{-\frac{(x-\mu)^2}{2\sigma^2}} - \mu \qquad (2)$$

where: $x =$ part weight, in grams.
$\mu =$ population mean, in grams.
$\sigma =$ population standard deviation, in grams.

Table 4. Compaction parameters and partial results from the long compaction sequences.

Mix	Die Fill Height	In-die Apparent Density	Average Compaction Pressure	Average Part Height	Average Part Weight
	(mm)	(g cm^{-3})	(Mpa)	(mm)	(mm)
1	52.4002	3.32	489.6	25.400	60.56
2	50.8762	3.40	501.5	25.423	60.32
3	52.4510	3.32	491.8	25.396	60.72
4	54.6100	3.21	508.8	25.455	60.98

By subtracting the mean part weight from the normal density value for each data point, the density functions can be centered at zero, which makes the comparison much easier. The normal density functions for all mixes are shown in Figure 3. Mix 1, containing 0.7% of PR-2 has the narrowest curve, indicating that the parts weight are more stable than with any other lubricant tested. The Kenolube-based mix has a nearly identical weight distribution compared to Mix 2, yet it contains 25% more lubricant. This demonstrates that even at significantly lower lubricant content, it is possible to control the part-to-part weight variation using lubricant PR-2. Mix 4, containing the EBS wax demonstrates a very wide part weight distribution which is slightly skewed to the left. The shape of the bell curve for the Acrawax C mix demonstrates that the parts weight was not as constant as for the other mixes.

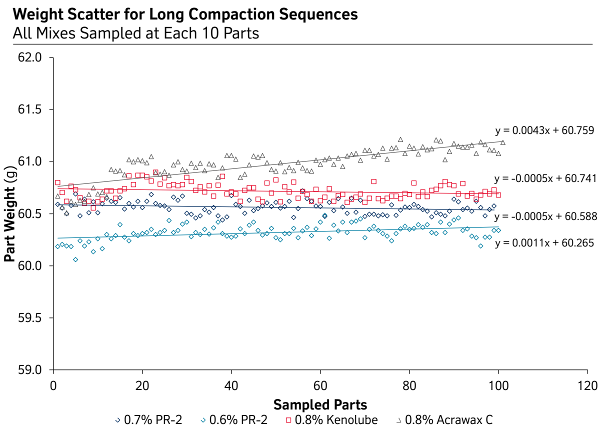

Figure 2. Evolution of the part weight measured throughout the long compaction sequences.

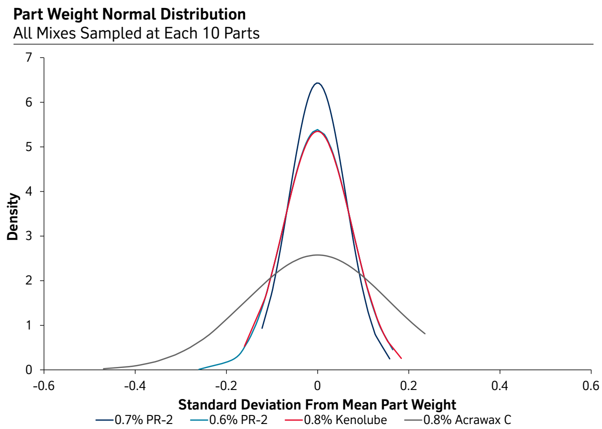

Figure 3. Normal distribution of weight calculated from the sampled parts.

Table 5 shows select data from the long compaction sequences which are representative of the stability of the compaction process. The relative standard deviation of the parts weight was calculated according to Equation 1 for all mixes. Similar to Figure 3, these values measure the spread of the parts weight during the long compaction sequences. The mix containing 0.7% of PR-2 lubricant had the lowest relative standard deviation whilst mixes 2 and 3 were equal. Not surprisingly, the Acrawax C based mix had the highest relative standard deviation, echoing the results presented in Figure 3.

Table 5. Process stability data calculated from the data collected during the long compaction sequences.

Properties		Mix			
		Mix 1 (0.7% PR-2)	**Mix 2 (0.6% PR-2)**	**Mix 3 (0.8% Kenolube)**	**Mix 4 (0.8% Acrawax C)**
Average Parts Weight (g)		60.56	60.32	60.72	60.98
Relative Standard Deviation (%)		0.102	0.123	0.123	0.254
Average Parts Density ($g \cdot cm^{-3}$)		6.83	6.80	6.85	6.87
Relative Standard Deviation (%)		0.119	0.092	0.105	0.134
Part-to-part Weight Stability	C_p	8.154	5.328	6.153	2.728
	P_{pk}	2.688	2.251	2.235	1.076
Part-to-part Density Stability	C_p	1.824	2.285	1.763	1.377
	P_{pk}	2.056	2.673	2.321	1.809

Conclusion

A high performance lubricant should be able to offer consistent parts in order to achieve satisfying results in an industrial application. Consistency can be described in several ways, but the part green density and the part weight are two variables where stability and limited variation are especially important. In previous papers, most results were obtained on an industrial press but in manual mode. Although this method enabled the compaction at high shear stresses, it lacked the strain of continued, automated production. In order to demonstrate that the high performance lubricant PR-2 can be effective under industrial conditions, it was necessary to assess the behaviour of the lubricant during long compaction sequences conducted in fully automated mode.

Powder mixes manufactured using the PR-2 lubricant offer high apparent densities and very good flow rates without resorting to the addition of neither flowing agents nor metallic stearates. Die-filling tests have shown that powder mixes containing the PR-2 lubricant will flow better within narrow die cavities therefore enabling complex and thin-walled parts to reduce any green density gradients. With a more even green density throughout the parts, it is reasonable to believe that the mechanical properties or the shape of the parts made using the PR-2 lubricant can be improved.

The realisation of long compaction sequences of about 1000 parts each enabled the gathering of sufficient amount of data to present a statistical analysis on some parameters. It was found that the lubricant PR-2 used at a concentration of 0.7 wt% can provide more stable values than Kenolube. The results from this experiment establish that the high performance lubricant PR-2 can provide a parts manufacturer the capability of making more consistent parts in actual production conditions. This remains true even if the concentration of lubricant is decreased in the metal powder feedstock.

Acknowledgements

The authors would like to thank the following contributors to the realisation of this paper:

Olivier Bouchard (RTMP) who significantly contributed to the development of several powders mixes, for the preparation and the characterisation of the powder mixes (apparent density, flow rate, die-fill behaviour, mechanical properties and the long compaction sequences).

Patrick England (NRC - Boucherville) for the compaction on the industrial press of several mixes and the responsibility of the long compaction sequences.

André Lacombe (RTMP) (and his team) for having prepared and overseen the transportation of the large samples amid busy working days.

References

1 Hanejko, Francis G., Luk, Sydney H., Rawlings, Arthur J., and Hale, Timothy J. Applications of High Performance Binder-Treated Materials. In *Advances in Powder Metallurgy & Particulate Materials* (Princeton, NJ (USA) 1998), Metal Powder Industries Federation, 11.67-11.79.

2 Lemieux, Patrick, Thomas, Yannig, Mongeon, Paul-Emile, Pelletier, Sylvain, and St-Laurent, Sylvain. Benefits of Die Wall Lubrication for Powder Compaction. In *Advances in Powder Metallurgy & Particulate Materials* (Princeton, NJ (USA) 2003), Metal Powders Industries Federation, 16-25.

3 St-Laurent, Sylvain, Thomas, Yannig, and Azzi, Lhoucine. High Performance Lubricants for Demanding PM Applications. In *Advances in Powder Metallurgy& Particulate Materials* (Princeton, NJ, USA 2006), Metal Powders Industries Federation, 1-13.

4 Ahlin, Åsa, Ahlqvist, Anna, and Litström, Ola. Newly Developed Lubricants for High Performance Powder Mixes. In *Euro PM2008 Congress and Exhibition* (Shrewsbury (UK) 2008), European Powder Metallurgy Association, 205-210.

5 Paris, Vincent, Thomas, Yannig, and St-Laurent, Sylvain. Novel High Performance Lubricants for Demanding PM Applications. In *International Conference on Powder Metallurgy and Particulate Materials* (Princeton, NJ (USA) 2012), Metal Powder Industries Federation.

6 Paris, Vincent, Thomas, Yannig, and St-Laurent, Sylvain. High Performance Lubricants for Conventional Medium Density Compaction. In *2012 Powder Metallurgy World Congress and Exhibition* (Shimogamo 2012), Japan Society of Powder and Powder Metallurgy.

7 METAL POWDER INDUSTRIES FEDERATION. *Standard Test Methods for Metal Powders and Powder Metallurgy Products*. Metal Powder Industries Federation, Princeton, NJ (USA), 2012.

8 Beiss, P, Broeckmann, C, Drygalov, M, Wassenberg, R. B., and Zafari, A. Frictional Shear Stresses during Compaction and Ejection of Pure Iron in Tool Steel Dies. In *International Conference on Tool Steels* (Aachen 2009).

9 McCall, James M. Manganese Stearate as a Lubricant for Powdered Metals. In *Advances in Powder Metallurgy & Particulate Materials* (Princeton, NJ (USA) 2003), Metal Powder Industries Federation, 26-32.