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Performance of Lubricants for High Density Applications

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Abstract

As powder metallurgy usage broadens, the amount of challenging parts with higher densities increases. More sophisticated powder mixes and high performance lubricants are therefore required for such parts. In the case of high-density parts, the lubricant content must be minimized as much as possible in order to reach higher densities. Lubricants for high density applications must therefore provide the same lubrication performance at much lower concentrations than traditional lubricants. This paper presents the compaction and ejection behaviour of new lubricant systems specifically designed to reach densities above 7.3 g/cm³. These lubricant systems were evaluated under real production conditions. The physical and sintered properties of mixes produced from such lubricants are also discussed. The results are compared to commonly used lubricants in the PM industry.

Keywords: High density, lubricant, steel powder

Introduction

The growth of the P/M steel market is linked to a large extent to the improvement of the static and dynamic properties of components in a cost competitive way. The sintered properties of P/M steel parts can be improved by alloying, liquid phase sintering, high temperature sintering and heat treatments. Nevertheless, density of parts remains one of the most important parameters controlling sintered properties, especially the dynamic ones, and achievement of high density at the lowest possible cost is crucial for the PM industry. Techniques such as the double press/double sintering and forging are used to achieve densities in the range of 7.4 to 7.5 g/cm³ and near full density, respectively [1]. However, the processing costs of these techniques are quite significant, limiting to some extent their market penetration. A lot of effort was also devoted during the last decades to the development of single pressing/single sintering processes allowing achievement of densities of 7.2 g/cm³ and higher. Compaction techniques such as warm compaction, warm-die compaction and die wall lubrication (DWL) were introduced to achieve higher green densities than the conventional cold compaction. Except for the DWL technique, the key factor limiting the densification is the amount of lubricant added to the mix. Indeed, lubricant, which is required to ease the ejection of parts and obtain green parts without defect and cracks with good surface finish and avoid die and tool wear, has a strong effect on the maximum density practically achievable during compaction due to its low specific gravity (~ 1 g/cm³). The lower the lubricant content, the higher the maximum achievable density. New lubricants must be therefore more effective. This is the reason why a significant amount of efforts have been focused over the last decades to develop new families of lubricant with improved lubricating properties. Examples of such developments can be found in the following references [2,3,4,5,6].

In addition to its lubricating properties, the ideal lubricant should also be malleable enough at the targeted compaction temperature to ease the compaction. This is particularly true for high density applications. Ideal lubricants must also provide fast and consistent flow, good die-filling performance, excellent surface finish, clean burn-off without residues or stain on the parts, high green and sintered strength and be easy to admix to steel powder and additives. Achievement of all these properties is difficult and composite lubricants will likely be developed in the future to address all these goals.

Rio Tinto Metal Powder (RTMP) has initiated many years ago R&D programs aiming at the development of high performance composite wax lubricants for challenging applications. These lubricants are developed for the cold or warm-die compaction processes. In-progress results of a program specifically aiming the development of lubricants for high density applications were recently reported in 2010 [7], 2011 [8,9] and 2012 [10]. Different promising composite lubricants based on fatty acid waxes and polymers were identified, referred to as Lube HD-A and HD-C in combination or not with Lube HD-WP previously developed for warm pressing applications. In particular, laboratory tests showed that densities varying from 7.32 to 7.47 g/cm³ were obtained with FD-0200 mixes containing 0.4 to 0.45% composite lubricants all based on lube HD-C in reference [7]. The ejection performance of these lubricants was similar or superior to a mix with 0.75% EBS wax. However, little information on the ejection performance under industrial conditions was known and it was felt that acquiring such information was key for the development of new lubricants. The objective of the present work is to assess the performance of high density lubricating systems based on different combinations of lubricants HD-A and HD-C under typical industrial conditions using a fully instrumented mechanical press. In this study, the formulation selected was the MPIF FC0208 and all mixes were made with the RTMP most compressible grade, ATOMET 1001HP. Performance of new lubricants is compared to reference lubricants widely used in the industry.

Experimental Procedures

ATOMET 1001HP, the most compressible unalloyed water-atomised steel powder manufactured by RTMP, was used in this study. The powder was admixed with 1.8%wt. -100 mesh Cu, 0.7%wt. graphite and lubricants HD-A, HD-C and/or HD-WP in various concentrations as described in Table 1. The total level of lubricants varied between 0.3 and 0.5%wt. For group 1, Lubricants HD-A and HD-C were tested alone and together in different proportions. Group 2 corresponds to Mix D containing HD-C and HD-WP. This mix has already been described and tested in a production scale in ref [7]. Two additional mixes were prepared with lower lubricant content, 0.4 and 0.3%wt. (Group 3). Mixes with 0.7%wt. of Kenolube P11 and EBS wax (Acrawax C atomized), two widely used lubricants in the PM industry, were used as references. The pore free density, also given in Table 1, was about 0.06 g/cm³ lower for this formulation than the FD0200 formulation tested in references [7, 8 and 9].

Table 1: Description of mixes tested in this study.

Group	Mix	Lubricant HD-A, %wt.	Lubricant HD-C, %wt.	Lubricant HD-WP, %wt.	Lubricant Other	Total Organic, %wt.	Pore Free Density, g/cm³
1	B	0.4	0.1	-	-	0.5	7.502
1	E	0.5	-	-	-	0.5	7.502
1	F	0.3	0.2	-	-	0.5	7.502
1	G	-	0.5	-	-	0.5	7.502
2	D	-	0.21	0.29	-	0.5	7.502
3	J	0.2	0.2	-	-	0.4	7.551
3	M	0.15	0.15	-	-	0.3	5.602
REF	WAX	-	-	-	0.7%wt. EBS Wax	0.7	7.407
REF	KEN	-	-	-	0.7%wt. Kenolube	0.7	7.407

The flow rate and apparent density were measured using the Hall flow apparatus as described in MPIF Standards 3 and 4 [11]. Sintered properties were measured on standard TRS specimens pressed at 760 MPa (55 tsi) according to MPIF Standard 41 [11]. Die was heated at 65°C to obtain part temperature similar to that achieved in the industrial press. Specimens were sintered at 1130°C for 25 minutes under a nitrogen atmosphere containing 10% H_2.

Compaction and ejection behaviour was evaluated on a 150 t Gasbarre mechanical press, which is equipped with strain gauges to constantly monitor the forces on the top and bottom punch. This equipment is available at the NRC Institute in Canada. Rings were pressed in a tungsten carbide (WC/Co) die, 25.4 mm in diameter with a core pin of 14.2 mm in diameter. For this study, rings about 12.7 and 25.4 mm tall were compacted at stroke rates of 5 and 10 parts/min. Compaction was performed at about 480, 620, 710 and 820 MPa (35, 45, 52 and 60 tsi) with the cold compaction (CC) and warm-die compaction (WDC) techniques. CC was performed in a non-heated/non-cooled die while WDC was performed by heating the die at 60°C. Some additional tests were also carried out with a die heated at 40°C. In all cases, the powder was not heated prior to compaction. It is worth mentioning that part temperature varied between 32 and 40°C when cold compacted to 62 to 65°C by WDC at 60°C. The height, weight and green density were measured on each part. The water displacement technique was used for the density measurement. The outside diameter was measured after compaction with a CMM apparatus, model SmartScope Flash 300, with a precision of 1.5 µm. Measurement was done at 40 points around the circumference of the part, at mid-height. The press monitoring software outputs an ejection curve for each part produced. All of these ejection curves were treated with a software developed in-house in order to extract key information such as : the stripping force- the force required to start the ejection movement, the slide OUT force- the force recorded when part begins leaving the die cavity and the sliding force- the average force measured between the initiation and ending points. In order to account for part height variations, the forces obtained were converted to ejection shear stresses by dividing the corresponding force by the lateral surface of the specimen in contact with the die. Additional details on how the curves are treated can be found in reference [10].

Results and Discussion

Results at 12.7 mm

Fig. 1 shows the compressibility curves obtained for mixes from group 1 together with the two reference mixes with Kenolube and EBS wax for rings pressed by the CC and WDC-60°C techniques. For both conditions, mixes containing lube HD-A showed better compressibility than the two reference mixes. This is particularly true at 600 MPa and above, the difference in green density versus the references reaching ~ 0.08 g/cm³ at ~ 820 MPa. The density achieved at ~ 820 MPa was 7.30 g/cm³ for all mixes from group 1 with WDC-60°C, which represents 97.3% of the theoretical pore free density. Mix G with 100% lube HD-C had a compressibility similar to those of reference mixes cold compacted. However, its compressibility was significantly improved by WDC and was similar to those of mixes containing lube HD-A, especially above 700 MPa. Paris et al [8] already showed that this lubricant needs a little bit of heat to be softened and this showed up clearly in this study.

The effect of the level of lube HD-A on density at ~820 MPa is better illustrated in Fig. 2. The higher the proportion of lube HD-A, the higher the density when CC is used. However, with WDC at 60°C, the proportion of each lubricant had no significant effect, both lubricants showing basically the same compressibility. It is very interesting to note that the gain in density achieved with WDC versus CC is around 0.09 g/cm³ for mix G containing 100% lube HD-C, even if part temperature was only increased by ~25°C. For a similar gain in temperature, the gain in density was only 0.01 g/cm³ for Mix E with 100% lube HD-A and between 0.01 to 0.03 g/cm³ for EBS wax and Kenolube. It is quite obvious from a densification point of view only that lubricant HD-A should be preferred over lube HD-C, especially if die is not heated. Density obtained with mix D containing lube HD-C together with HD-WP is also given in Fig. 2. As it was the case for mix G, the density achieved with CC was quite low, which is not a surprise considering that both HD-C and HD-WP required some heat to soften and be activated. This is even more important for lube HD-WP, which was developed for Warm Compaction. Green density reached about 7.28 g/cm³ with WDC at 60°C, the gain being of ~ 0.07 g/cm³. This gain is mainly attributed to lube HD-C.

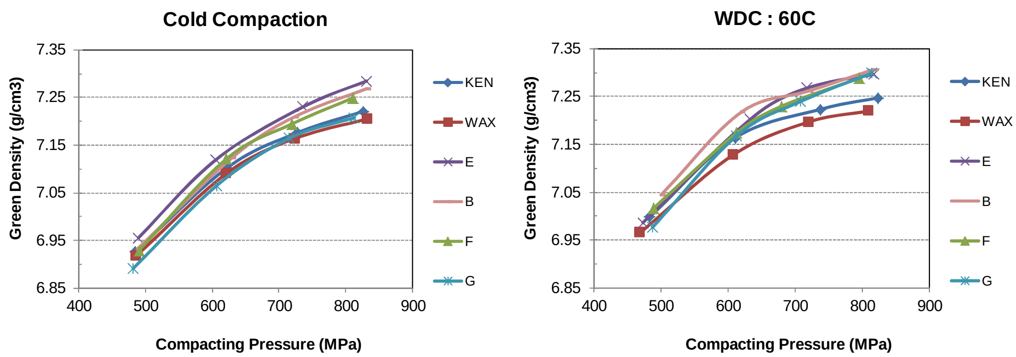

Fig. 1- Compressibility curves for group 2 obtained by CC and WDC- 60°C (12.7 mm, 5 SPM).

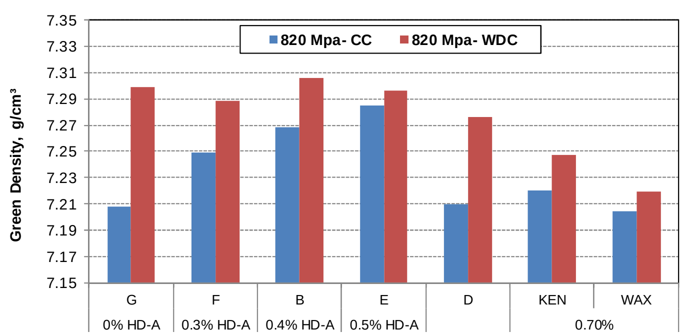

Fig. 2- Green density for different mixes at ~ 820 MPa (12.7 mm, 5 SPM).

The stripping and slide OUT ejection shear stresses obtained by the CC and WDC processes for mixes from group 1 and the reference mixes are given in Fig. 3 and Fig. 4 respectively. The first point that strikes the attention is the large difference between cold and warm-die compaction on the ejection shear stresses for all mixes. Indeed, the ejection shear stresses are reduced by 20 to 35% when WDC is used. It is also interesting to note that the difference between mixes is relatively low at ~ 480 MPa (35 tsi) for both compaction conditions. This result is somewhat surprising considering the relatively low density achieved at that pressure and the low concentration of lubricant in mixes B, E, F and G. Both conditions should result in very low quantity of lubricants expelled out at the die wall. This result indicates that lubricants HD-A and HD-C are quite efficient even if low quantity is used. The difference in stripping and slide OUT pressure between mixes increased significantly as pressure was increased. Mix with Kenolube gave the best results at pressures above ~ 600 MPa. For this mix, the ejection shear stress dropped slightly as pressure was increased. All the other mixes behave quite similarly, with some differences depending on the conditions. Mixes F and B, containing both lubricants HD-A and HD-C, gave the best results amongst group 1, better than EBS wax when die was not heated and at least equivalent to EBS wax with WDC at 60°C.

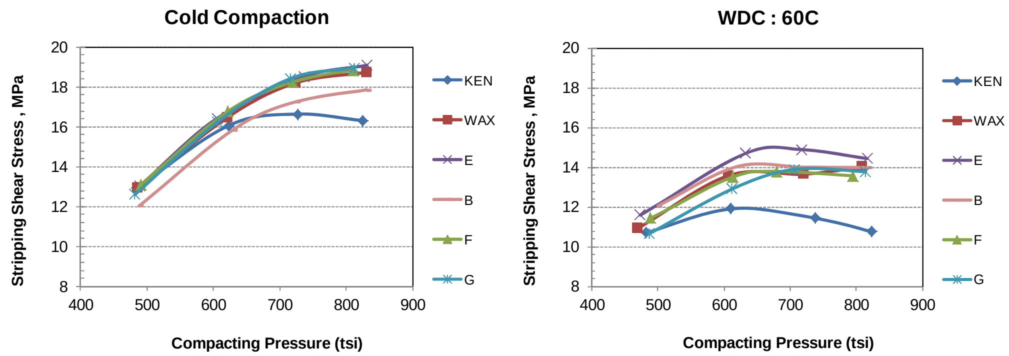

Fig. 3- Stripping shear stresses for specimens pressed by CC and WDC-60°C (12.7 mm, 5 SPM).

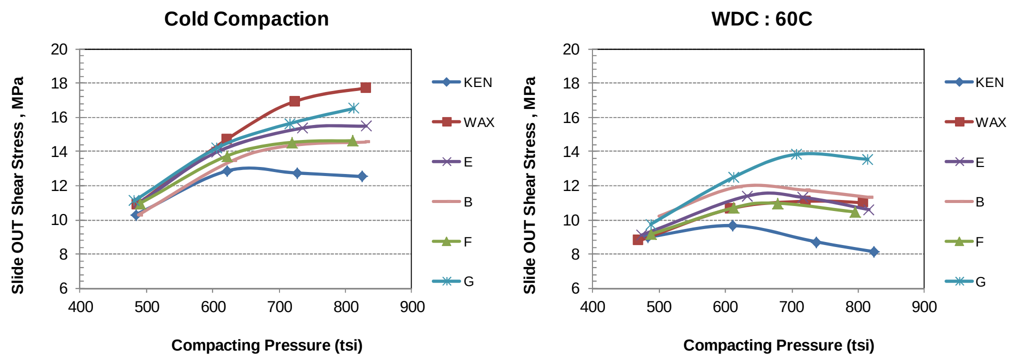

Fig. 4- Slide OUT ejection shear stress for specimens pressed by CC and WDC-60°C (12.7 mm, 5 SPM).

One way to measure the efficiency of lubrication at die walls is by comparing the ejection shear stresses at beginning and end (when parts begins exiting the die cavity) of the ejection cycle. This is done by calculating the Slide OUT/Stripping ratio. Indeed, this ratio is indicative of how the sliding force varies during ejection. The lower this ratio, the better the lubrication. Fig. 5 shows this ratio for groups 1 and 2 and reference mixes at 820 MPa. It is worth mentioning that this ratio remains quite constant as a function of the compacting pressure. Mixes B, E and F containing lube HD-A gave quite low values, similar to that of Kenolube. Also, the compaction method had almost no effect on the ratio. The ratio for Mix G with 100% lube HD-C was higher, especially with WDC where it reached almost 1. Similar results were obtained with mix D which also contains lube HD-C. In that case, lube HD-WP did not contribute to improve the lubrication like HD-A did for mixes F and B. Finally, interestingly, the ratio for mix WAX, which was ~0.95 when cold compacted, improved significantly when WDC was used and was similar to that of Kenolube, suggesting that a slight increase in temperature is beneficial for the lubricating behaviour of EBS wax.

Results obtained with 0.5%wt. lubricant showed that Mix F offers the best compromise in term of compaction and ejection. This formulation is constituted of 60%HD-A/40%HD-C. Two mixes with a total lubricant content of 0.4 and 0.3%wt. were prepared with a very similar lubricating formulation (group 3). The ratio of HD-A/HD-C was 50/50. The compressibility curves are given in Fig. 6 for these mixes, Mix F and the two reference mixes. Reducing the lubricant content had no effect on density at low pressures but resulted in an increase in green density at 700 MPa and above for both compaction conditions. Density reached ~ 7.35 g/cm³ for mix M (0.3% Lube) by WDC at 820 MPa, which is ~ 0.11 g/cm³ higher than the density reached with the references. The gain in density with WDC was between 0.04 and 0.08 g/cm³ for the entire pressure range for mixes with composite lubes.

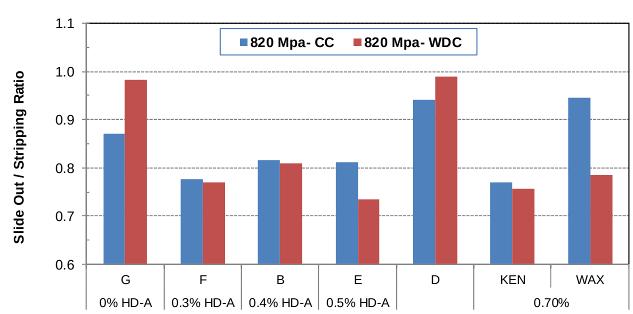

Fig. 5- Slide OUT/Stripping ratio for mixes pressed by CC and DWC at 60°C (12.7 mm, 5 SPM).

The ejection performance for these mixes is given in Fig. 7. Only the sliding average shear stress is plotted, very similar trends being observed with the stripping and Slide OUT. As it was the case for the other mixes, the sliding shear stress was significantly lower with WDC. No difference was observed at 480 MPa with the CC, which is again surprising considering the very low level of lubricant expected to be squeezed to the die walls. However, the sliding shear stress slightly increased with the WDC for mixes J and M. The difference in ejection between these mixes and the Kenolube one further increased as the pressure was raised for both pressing conditions. However, the performance of mix J with 0.4% lubricant was very close to that of Mix with Wax. In addition, we can observe with WDC that the sliding shear stress went down slightly as the pressure was increased above 700 MPa for Mixes F, J and M. A similar behavior is seen with Kenolube. This behavior clearly shows that the lubricant is efficiently transferred to the die walls and remains efficient even under very high applied pressure.

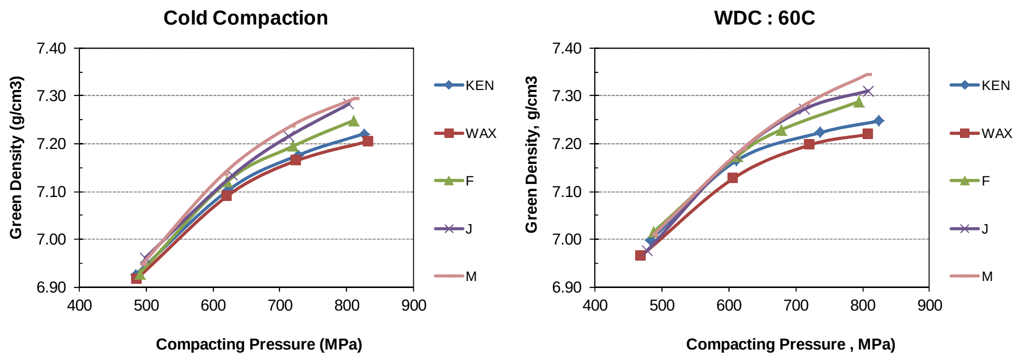

Fig. 6- Compressibility curves obtained for Mixes F, J and M obtained by CC and WDC- 60°C (12.7 mm, 5 SPM).

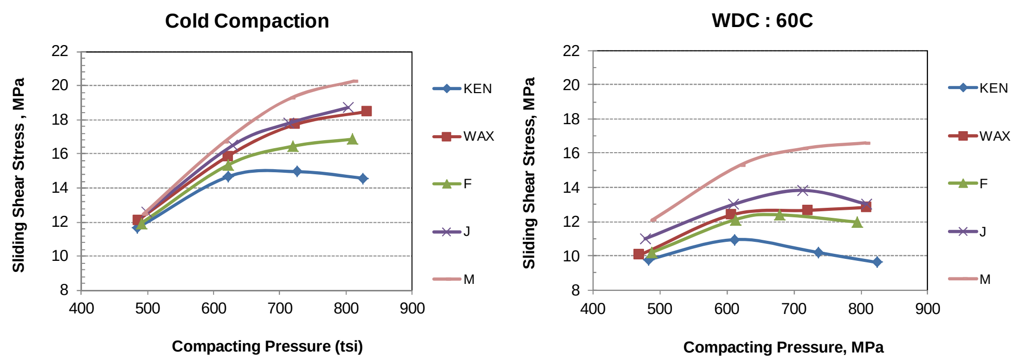

Fig. 7- Ejection performance (Sliding AVG) for Mixes F, J and M obtained by CC and WDC- 60°C (12.7 mm, 5 SPM).

Results at 25.4 mm

In order to validate the behavior of lubricants under more severe conditions, compaction tests with the industrial press were pursued with an increased die fill to reach a length of ~ 25.4 mm after ejection. These tests were performed with the WDC at 60°C only with Mixes F, J and M and the two reference mixes. Parts were first compacted at four different pressures at a rate of 5 parts/min. The variation of density and ejection obtained as a function of the pressure is illustrated in Fig. 8. The comparison with the previous test at 12.7 mm will be discussed later. In term of density, the same trend already described in previous section was observed : 1- no difference in density was observed at 480 MPa, 2- higher density is achieved with mixes F, J and M for pressures higher than 600 MPa, the highest density at 820 MPa being inversely proportional to the lubricant content. Similar trend was also obtained for the average sliding shear stress. However, the best result was still obtained with the Kenolube mix while Mixes F and J were quite comparable to the mix with EBS wax. We can see for all the mixes that the sliding shear stress increased first with the pressure to reach a maximum between 600 and 700 MPa and then goes down above 700 MPa. This trend is also observed with mix M containing 0.3% lubricant except that the maximum is obtained at a slightly higher pressure.

Additional tests were also carried out at 10 parts/min to validate the effect of increasing the compacting rate on density and ejection. Results, not shown here for the entire pressure range, were very similar to those achieved at a stroke rate of 5 parts/min except that slightly lower densities were achieved for all mixes at all pressures as anticipated. On the other hand, the ejection shear stresses were in average slightly lower, the difference being about 5%. The density and ejection shear stress obtained at 820 MPa by WDC for parts 12.7 and 25.4 mm tall are compared in Fig. 9. Slightly lower green density was achieved for all the mixes by doubling the part height, the drop in density being of ~0.025 g/cm³ for mixes containing the composite lubricant and

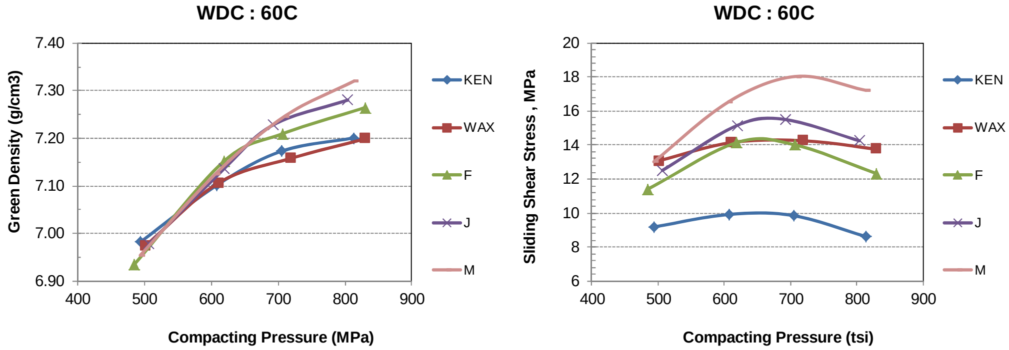

Fig. 8- Green density and sliding shear stress as a function of compacting pressure for parts 25.4 mm tall (5 SPM).

0.05 g/cm³ with Kenolube. An additional loss in density was obtained by increasing the stroke rate, about 0.03 g/cm³ for mixes F, J and M and 0.06 g/cm³ for mix with Kenolube. Mix with Kenolube was particularly affected by the part height and stroke rate while mix with Wax was not so affected, the total loss in density being only 0.04 g/cm³ versus 0.10 g/cm³ for Kenolube.

The average sliding shear stress was not greatly affected by the part height and stroke rate for mixes F, J, M and WAX, the difference remaining below 1.2 MPa or 8%, even for mix M containing only 0.3% lubricant. This result indicates that height did not affect the lubricating efficiency of Lube HD-A/HD-C, even if the sliding distance was doubled. The ejection was improved by increasing part size and doubling the stroke rate for Kenolube, the difference being of the order of 2 MPa or ~20%. Finally, Mix J containing 0.4%wt. lubricant performed almost equally to the mix WAX containing 75% more lubricant.

It is difficult to compare the lubricating efficiency of the new composite lubricants to that of Kenolube since their levels are not the same. Fig. 10 shows the variation of the ejection sliding shear stress as a function of the lubricant content for mixes F,J, M and KEN. It is seen that the ejection varied inversely with the lubricant content for mixes with the new composite lubricants. If we extrapolate these results, the ejection performance of a mix with 0.7% composite lubricant would be equal or even slightly better than the reference mix with Kenolube. This conclusion is obtained for all conditions tested.

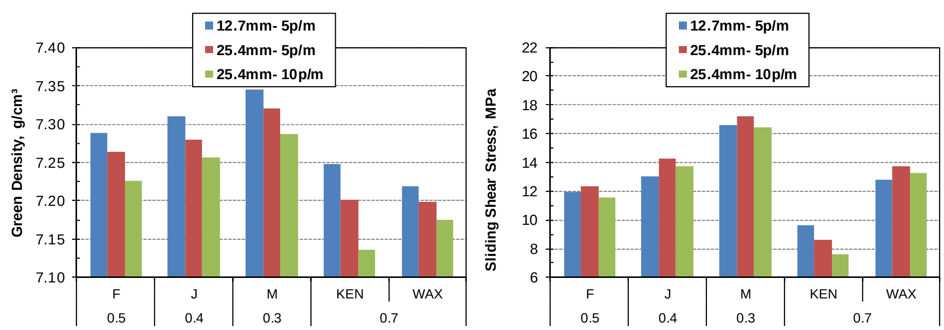

Fig. 9- Comparison between tests carried out at 12.7 and 25.4 mm by WDC at 60°C and 820 MPa.

Springback

Another key characteristic is the springback or the expansion that parts undergo at ejection. Indeed, when reaching high densities close to the pore free density, parts may expand significantly at ejection, causing delamination or small defects. Springback is affected by several factors such as die stiffness, compacting conditions, base powder intrinsic characteristics, part height and geometry. The lubricant may also affect springback as we know it may partially melt during compaction. Fig. 11 illustrates the variation of the springback for mixes B, KEN and WAX for specimens pressed by CC and WDC under different conditions at 820 MPa, which is the most challenging pressing condition used in that study. Similar trend was observed with all mixes containing Lube HD-A. Mix with EBS wax was not very sensitive to the compacting conditions, springback remaining almost unchanged. Mixes containing Kenolube and the composite lubricant were more sensitive. First, heating the die led to an increase of the springback for both mixes. Increasing the height had no effect for Kenolube but led to another increase of the springback for mix B. Finally, increasing the stroke rate at 10parts/min led to an increase of the springback for all the lubricants.

Additional tests were conducted to better quantify the effect of die temperature on springback. A new mix was prepared and tested by CC and WDC at 40 and 60°C were carried out with Mix B. The effect of die temperature on density, springback, ejection sliding shear stress and part temperature at 820 MPa is shown in Fig. 12. Part temperature as well as density increased with the die temperature as expected. The ejection sliding shear stress followed the opposite trend. It is interesting to note that the springback did not change between “No heating” and 40°C but only increased when die was heated to 60°C. The springback achieved at 40°C was equivalent to that of EBS wax. Parts produced at 40°C had a much better surface finish than at 60°C. Considering all that, it is recommended for lube HD-A/HD-C to maintain the part temperature below ~ 50-55°C to limit springback.

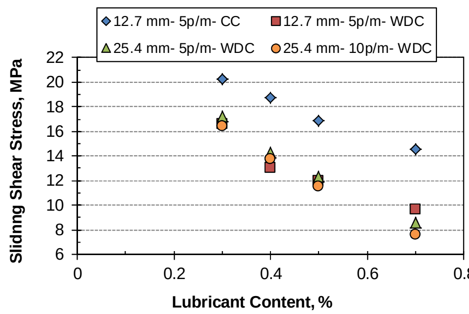

Fig. 10- Sliding shear stress versus the lubricant content.

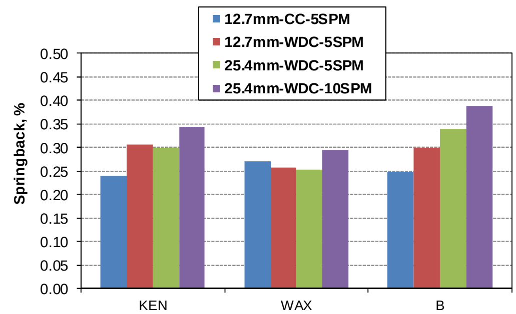

Fig. 11- Springback for different compacting conditions.

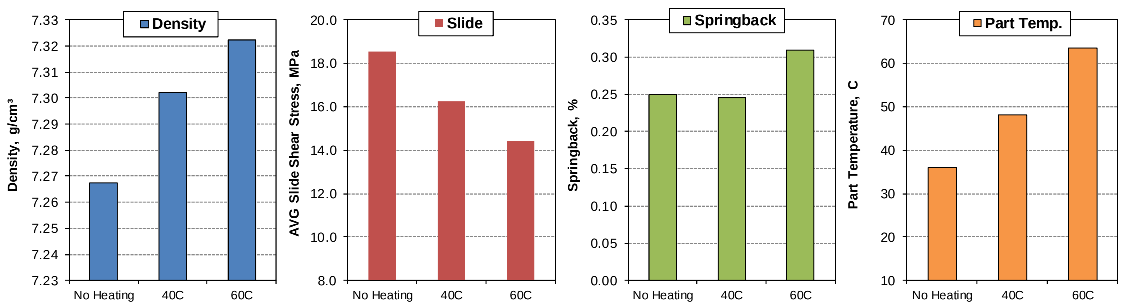

Fig. 12- Effect of die temperature on density, ejection slide shear stress and springback (12.7 mm, 5SPM)

Physical and Sintered Properties

The apparent density, flow and sintered properties are given in Table 2. Mixes containing Lube HD-A/HD-C (B, F, J and M) gave low apparent density of ~2.90 g/cm³. Flow rate was quite good at ~33 s/50g. Paris et al [10] showed that significant improvement in flow rate and higher apparent density can be obtained by changing the blending procedure. Mix with Kenolube gave higher apparent density and better flow rate while the flow rate and apparent density were typical for mix with EBS wax. Mix D containing lube HD-A/HD-WP had the highest apparent density and best flow amongst the mixes tested. This result is in accordance with results presented in reference [7].

The sintered strength and apparent hardness obtained were very similar for all the mixes with best results being achieved with Mixes J and M which had the highest density. Mix with EBS wax also gave very good sintered strength considering the relatively low density achieved with that mix. Several sintering tests confirmed that result. Dimensional change of mixes with Lube HD-A and HD-C was more positive, which is not a total surprise considering the higher density obtained with these formulations.

Table 2. Physical and sintered properties for various mixes [(1)].

	KEN	WAX	D	B	F	J	M
AD, g/cm³	3.15	2.99	3.36	2.97	2.82	2.86	2.88
Hall Flow rate, s/50g	29.0	37.5	27.2	32.9	34.1	33.5	33
G. Dens., g/cm³	7.21	7.25	7.29	7.33	7.31	7.33	7.36
TRS, Mpa	1240	1330	1260	1300	1250	1260	1350
App. Hard. HRB	88	90	87	88	88	90	91
Dim. Change vs Die, %	0.34	0.41	0.60	0.52	0.54	0.53	0.55

(1) Pressing was done at 760 MPa by WDC at 65°C. Sintering was done at 1130°C for 25 min in a 90% N_2-10% H_2 atmosphere.

Conclusions

This paper presented in-progress results of a R&D program specifically aiming the development of lubricants for high density applications using cold or warm-die compaction processes. Specifically, the performance of different promising composite lubricants, based on fatty acid waxes and polymers identified previously, as measured with a fully instrumented mechanical press enabling the monitoring of the ejection forces during all the ejection cycle were presented for a FC0208 formulation. The present work confirmed the excellent compressibility and lubricating performance of new composite lubricants at concentrations varying from 0.3 to 0.5%wt. under different compacting conditions (stroke rate and part aspect ratio). The effect of the proportion of two different lubricant types was characterized and the best formulations were shown to lead to significantly higher densities (0.08g/cm^3 or higher) at high pressure while maintaining similar ejection performance as a mix with 0.7%wt. EBS wax.

In fact, all the results confirmed that the ejection shear stresses were significantly reduced when WDC was used. It was also shown that the difference in ejection between mixes was minor at ~ 480 MPa (35 tsi) whatever the compaction conditions used (stroke rate and aspect ratio). This result was somewhat surprising considering the relatively low density achieved at that pressure and the low concentration of lubricant in the mixes, which should normally result in very low quantity of lubricants at die walls. This result indicates that the new lubricating systems are quite efficient and active even if at low concentration.

The expansion that parts undergo at ejection was also closely monitored by CMM. Important difference in springback was observed depending on the lubricating formulations and the compacting conditions used, particularly at high densities close to the pore free density. The die temperature was found to have a great influence on springback, particularly for Kenolube and the new composite lubricants. Based on tests carried out, it would be recommended to maintain the part temperature below 50-55°C to optimize both the springback and surface finish.

Further work will be performed to validate these results on long production runs. It is also planned to characterize new efficient lubricating systems (PR-1 family) at low concentrations for high density applications. Indeed, tests ran in parallel showed that these types of lubricant offered better ejection performance than Kenolube even for tall parts.

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