Euro PM2013 - Lubricants

Manuscript refereed by Prof Lars Nyborg

Effect of Lubricant Particle Size Distribution on the Processing and Properties of P/M Ferrous Parts

Yannig Thomas, Fabrice Bernier, Sylvain St Laurent and Vincent Paris**

National Research Council Canada, 75 de Mortagne Blvd, Boucherville, J4B 6Y4, Québec, Canada
** Rio Tinto Metal Powder, 1655 Route Marie Victorin, Sorel-Tracy, J3R 4R4, Québec, Canada*

ABSTRACT

The role of lubricants during compaction is crucial and nowadays even more important, due to the increased number of PM parts with higher densities and/or challenging ejection conditions. The development of new lubricants for single compaction requires however an excellent understanding of the lubrication mechanisms and the behaviour of these new compounds.

In this paper, the effect of the lubricant particle size distribution on the compaction and ejection behaviour, but also on the static and dynamic properties of ferrous powder metallurgy parts is studied. Indeed, as parts reach higher densities, the influence of the size and shape of pores become dominant on the mechanical performance and particularly fatigue resistance of the parts. Water atomized powder mixes containing different particles sizes of ethylene-bis-stearamide (EBS) lubricant were compacted both on a laboratory press and on an industrial mechanical press at higher speed rates. In addition to compressibility curves and static sintered mechanical properties, complete ejection curves and fatigue tests results are presented.

INTRODUCTION

Lubricants are playing an important role in the manufacturing of powder metallurgy components. This is even truer nowadays as some parts manufactured using the PM route become more complex, taller or require higher densities. This creates demand for alternative lubricants that will have higher lubrication efficiency, but which can also respond to increased concerns about the environmental impacts of many industrial processes as well as other more practical considerations (parts appearance, compressibility, shelf-life of metal powders).

Conventional P/M lubricants are typically fatty acid based waxes such as ethylene bis-stearamide (EBS) or metallic stearates (mainly zinc stearate (ZnSt) and lithium stearate (LiSt)). The latter have seen their usage shrink, or their respective proportion reduced because of environmental concerns and residuals left in the furnaces and on parts after sintering. Kenolube, which is a specially designed blend of EBS and ZnSt, was developed long time ago. Still, it is one of the best lubricants, leading to low ejection forces. Its tendency to sometimes leave stains on sintered parts due to the presence of ZnSt as well as its sensitivity to very humid environments have however bolstered the P/M community to search for alternatives. In addition to polymeric lubricants that were developed in the late 90's, mainly for high green strength and warm pressing applications, the last decade has seen new lubricants being developed. Those improvements addressed the formulation and processing of new composite waxes, still mainly based on fatty acids components but having more efficient lubricating properties and leaving no or very low levels of residuals after burn-off. These lubricants are the foundation of the new warm die compaction process (50-80°C) and high density developments. In addition to their chemical formulations and their delubrication and part cleanliness properties, the particle size distribution and shape of these lubricants are key characteristics which are worth being considered since they could also significantly affect efficiency of the lubricants.

Even though it is recognized in the PM community that larger particle sizes will improve the ejection performance, relatively few papers were published on that subject. Most were published mainly fifteen years ago [1, 2, 3]. In particular, Lawrence et al. [1] evaluated large lubricant particle size range on laboratory equipment and showed that coarse particles lead to better ejection performance, higher green strength at the expense of lower compressibility. Detrimental effects on static sintered properties were observed due to larger voids left after lubricant burn-off. Some experimental composites lubricants using fine and coarse particles were said to be a good compromise.

As for the static mechanical properties, larger lubricant particle size might also impact the fatigue behaviour of PM steels, which is affected by porosity and the microstructure. Any local phenomenon has greater importance for fatigue behavior than for resistance under static stress. Indeed, early work on the evaluation of fatigue limit for PM steels [4] showed that not only the amount but also size of the pores affect the endurance limit; the largest pores being more detrimental than smaller ones [5, 6, 7]. In fact, microcracks usually originate at or near the surface of pores with the highest stress concentration factors [8, 9, 10, 11]. More quantitatively, Danninger et al. [6], applied the Kitagawa plot to explain the influence of pore size on the fatigue limit of PM steels by applying a critical pore size criteria, under which the pores do not affect fatigue limit. However, for pores larger than the critical pore size, the fatigue limit decreases with increasing defect size. Consequently, it is not so much the average size of the pores which influences the fatigue limit rather than the size of the largest pore present in the volume subject to repetitive stress loading. The influence of the size and shape of pores on the mechanical performance and particularly fatigue resistance of the parts becomes even more important as density of part increases.

To improve the understanding of the lubrication mechanisms and help in the development of new lubricants for single compaction of parts with excellent sintered properties, the effect of the lubricant particle size distribution on the compaction and ejection behaviour, but also on the static and dynamic properties of ferrous powder metallurgy parts deserves more attention. It is therefore the objective of this paper to discuss the effect of using ethylene-bis-stearamide with different particle size distributions on the compressibility, ejection, static and fatigue properties of water atomized powder mixes compacted on both a laboratory press and an industrial mechanical press at higher speed rates.

EXPERIMENTAL PROCEDURE

Materials

ATOMET 1001HP, the most compressible unalloyed water-atomised steel powder in Rio Tinto Metal Powders' offering was used in this study. The powder was admixed with 1.8%wt. Cu, 0.7%wt. graphite and 0.7%wt. of four different particle sizes ground powder EBS lubricant. Two reference mixes were also prepared containing 0.7 %wt. of either Acrawax C atomized or Kenolube. The particle size distributions of the lubricants were determined with a Coulter laser diffractometer, model LS230 using a dry «Tornado» disperser (Fig. 1). Key parameters of the distributions are summarized in Table 1 where a diameter Dx is reported at various volume percent of the particle size cumulative distribution of the sample. It is worth mentioning that the particle size characteristics of the Acrawax C are very close to that of EBS-5 µm.

Table 1: Particle size characteristics of lubricants

	D_{10}	D_{50}	D_{90}	$D_{99.9}$
EBS_2.5µm	0.7	2.4	5.5	18.8
EBS_5µm	0.9	4.5	12.4	22.8
EBS_15µm	1.9	12.5	27.2	71.5
EBS_25µm	3.8	23.0	54.2	101.1
ACRAWAX C atomized	1.1	5.8	14.6	27.3
Kenolube	2.0	22.7	60.3	92.1

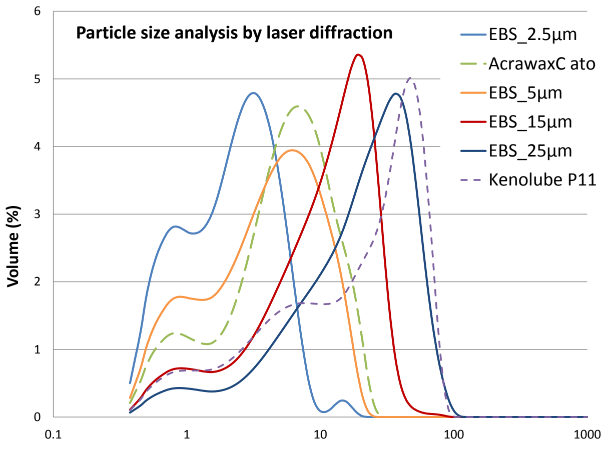

Fig. 1: Particle size distribution of lubricant waxes

Compressibility and lubrication evaluation

Compaction and ejection behaviour was evaluated on an industrial 150 mt Gasbarre mechanical press, which is equipped with strain gauges to constantly monitor the forces on the top and bottom punches. In order to construct compressibility curves, rings (Din 14.2mm, Dout 25.4mm, height 12.7mm) were pressed in a tungsten carbide die, at a stroke rate of 5 parts/min at 480, 620, 710 and

820 MPa (35, 45, 52 and 60 tsi) with the cold compaction (CC) and warm-die compaction (WDC) techniques. The CC process was performed in a non-heated/non-cooled die while the WDC process was performed by heating the die at 60°C (WDC-60°C). In all cases, the powder was not heated prior to compaction. The part temperature varied between 31 to 37°C when cold compacted and from 62 to 65°C when warm-die compaction was performed. 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. Measurements were taken at mid-height at 40 points around the circumference of the part. The press monitoring software outputs an ejection curve for each part produced. These ejection curves were treated with an in-house software in order to extract key information such as the stripping force (the force required to start the ejection movement), the sliding force (the average force measured between the initiation and ending points) and the out-die sliding force (the force recorded when part begins leaving the die cavity) (Fig. 2). In order to account for part height variations, the forces obtained were converted to 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 [12]. Following each test, a standard reference mix with excellent ejection behaviour was compacted to clean the die and verify if the die was not damaged during previous tests. The ejection shear stress of that standard mix was stable all along the tests, confirming that tooling had not deteriorated.

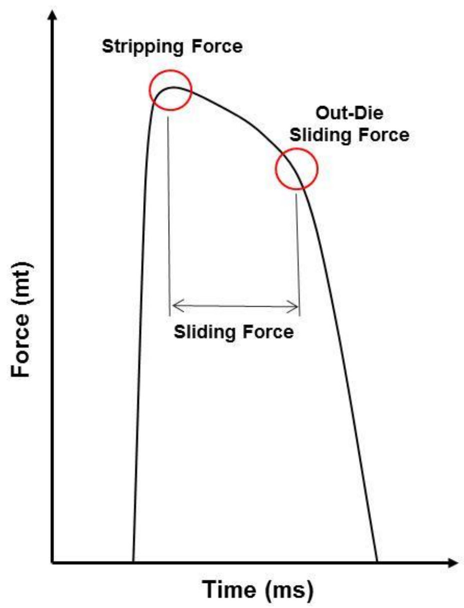

Fig. 2: Schematic of a typical ejection curve

Green and sintered properties were also measured on standard TRS specimens pressed on a laboratory hydraulic press at 480, 620, 710 and 820 MPa according to MPIF Standards 15, 41-44 [13]. Die was either at room temperature or heated at 60 °C for all mixes. Specimens were sintered at 1130°C for 25 minutes under a nitrogen atmosphere containing 10% H_2. Fatigue tests were carried out on 7.15 g/cm^3 TRS specimens using a load control fatigue machine with a three-point bending apparatus operated at a frequency of 85 Hz and a R-ratio (σmin/ σmax) of 0.1. The staircase method was used to determine the endurance limit, using a 2.5 million cycles run-out limit [14].

RESULTS

Compressibility and springback

Fig. 3 shows the compressibility curves obtained for mixes containing the different particle size EBS lubricants together with the two reference mixes with Kenolube and Acrawax C, for rings pressed on the industrial press by the CC and WDC-60°C techniques.

As expected, the lubricant particle size affects slightly the compressibility of the powder mixes both at CC and WDC-60°C on the whole pressure range. At 820 MPa, a reduction of 0.07g/cm^3 is observed between the EBS-2.5µm and the EBS-25µm, but it is only 0.04-0.05g/cm^3 between Acrawax C and the EBS-25µm. It is worth mentioning that despite the fact that both EBS-5µm and Acrawax C atomized

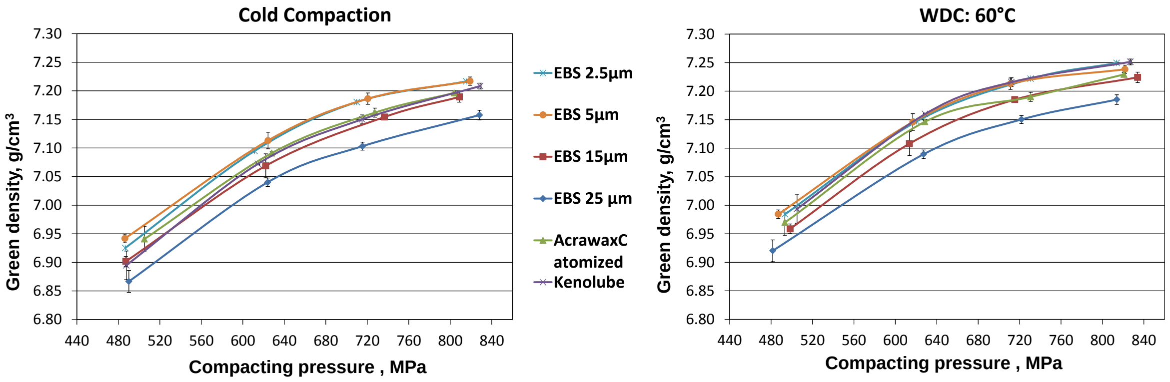

Fig. 3: Compressibility curves obtained by a) CC and b) WDC-60°C

are ground and have very similar particle size distribution, the compressibility of mix containing Acrawax C is lower at pressures higher than 600 MPa. This behaviour is observed both with CC and WDC-60°C. Also, the particle size of the lubricant is not the only factor affecting the green density since both Acrawax C and Kenolube have similar compressibility using CC even if their size distribution are significantly different. When WDC-60°C is used, the compressibility of Kenolube is even slightly improved compared to Acrawax C.

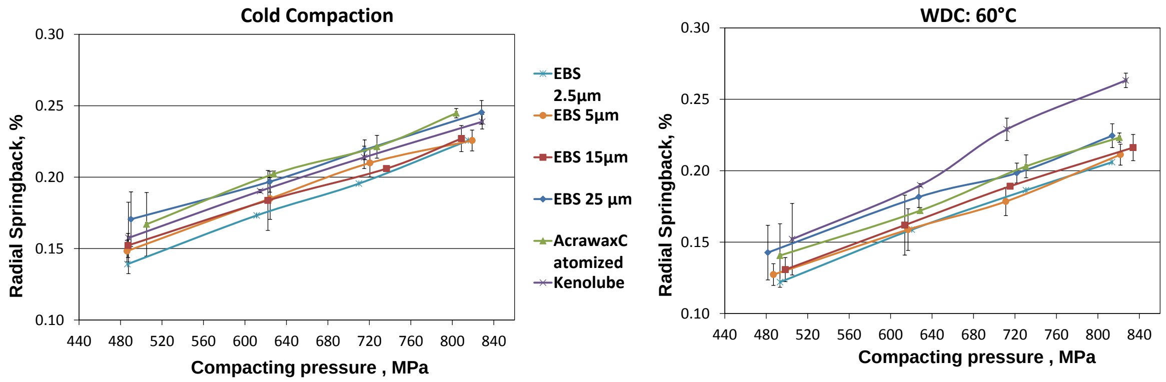

Fig. 4: Green expansion (springback) obtained by a) CC and b) WDC-60°C

Fig.4 shows that the green expansion of compacted rings increases with the compacting pressure. The different lubricants follow the same trend both under CC and using WDC-60°C, with values slightly below for WDC-60°C except for Kenolube. A slight increase of springback is however observed when the particle size of EBS lubricant is increased (about 10% difference between EBS-2.5µm and EBS-25µm). Only the behaviour of Kenolube is different with WDC-60°C, leading to significantly higher springback when compacting at pressures higher than 600 MPa. This correlates with the presence of some delamination or green cracks observed at the surface of the parts made with Kenolube mix.

Ejection performance

The main effect of the variation of the lubricant particle size distribution is on the ejection behaviour of the powder mixes. This is particularly evident for cold compaction as shown in Fig.5. A decrease of up to 10% in stripping shear stress is observed when increasing the D50 particle size of EBS from 2.5µm to 25µm. At 820 MPa, stripping shear stresses of 20 MPa and 18 MPa were measured for the EBS 2.5µm and EBS 25µm, respectively. Surprisingly the Acrawax C atomized lubricant - which has similar particle size distribution as the EBS-5µm - presents the highest stripping shear stresses. The Kenolube lubricant recorded the lowest values but also showed a different behaviour with a stabilization of the stripping shear stress as compacting pressure increases. Fig. 5 shows also that it is important not only to look at what is often called the ejection peak at the start of the ejection step, but also at the complete ejection curve. Indeed, when using the lower EBS particle size distribution, the amount of lubricant expelled at the surface of parts seems not to be enough to prevent the increase of friction when the ring is moving out of the die. This is demonstrated by a higher out-die sliding shear stress than the stripping shear stress for the EBS-2.5µm mix. The out-die sliding/stripping shear stresses ratio is therefore a good parameter to compare the behaviour of the different lubricants. It is clearly seen that this ratio greatly diminishes when the lubricant particle size increases. Based on this parameter, the specific behaviour of Kenolube stands out. The powder mixes containing the finer EBS have higher out-die sliding/stripping shear stresses ratios which also increase with progressively

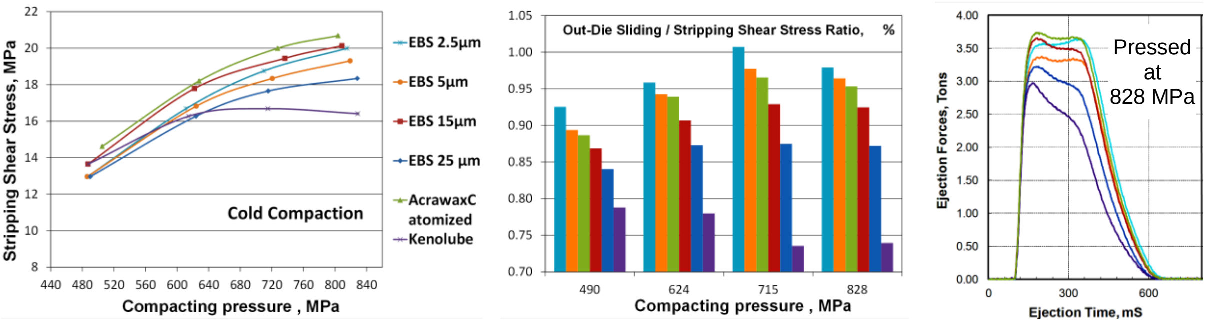

Fig. 5: Stripping shear stresses, Out-Die Sliding/Stripping Shear Stress ratio & Ejection curves for rings pressed by CC

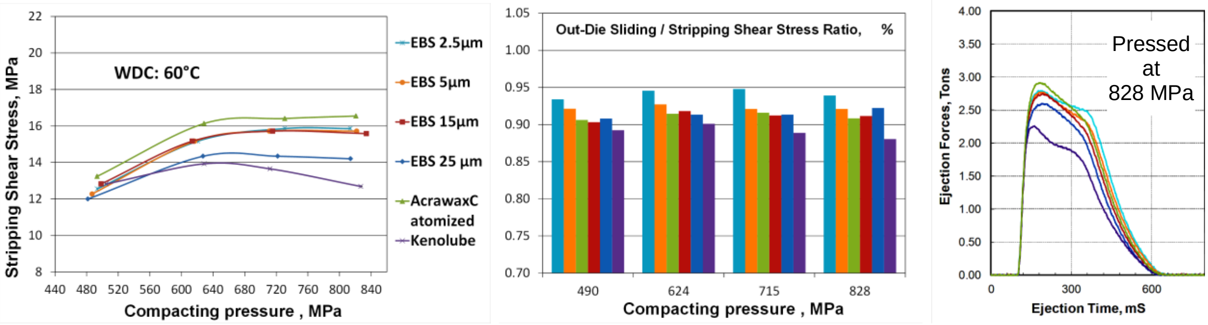

Fig. 6: Stripping shear stresses, Out-Die Sliding/Stripping ratio and Ejection curves for rings pressed by WDC-60°C

higher compacting pressures. As progressively coarser lubricants are used, the shear stresses ratios are more stable over the entire compaction pressures range. For the Kenolube-based mix, these shear stresses ratios are even diminishing as the compaction pressure increases.

For WDC-60°C, the effect of the lubricant particle size on the stripping shear stress is similar to that observed by CC. However, all the values are significantly lower than those obtained with CC. Also, when the compaction pressure increases, most lubricant exhibit stable stripping shear stresses as demonstrated in Fig.6. In the case of Kenolube, the stripping shear stress even diminishes as pressure increases above 600 MPa. The levelling and even reduction of the ejection pressure with progressively higher compaction pressure was also described previously [15].The shape of the ejection curves are completely different from what was observed at CC. Indeed, similar out-die sliding/stripping ratios are observed whichever lubricant is used. This might be explained by lower viscosity and/or higher deformability of the EBS lubricants at higher compacting temperature which reduces significantly the effect of the particle size distribution on their migration towards the die wall/part interface.

Static and dynamic sintered properties

The transverse rupture strength (TRS), dimensional change from green size as well as apparent hardness and pore sizes were measured for all the powder mixes pressed in the different conditions. However, only TRS as a function of sintered density (Fig.7) are reported here. For CC, all the powder mixes gave similar results except for the Acrawax containing mix which showed 3% increase in TRS value for all the density range compared to the average of all other mixes. Except for Acrawax and EBS-25µm, parts pressed by WDC-60°C showed about 10% increase compared to CC for density below 7.05 g/cc. At higher densities, TRS values for Kenolube levelled off and even decreased for higher pressures. This may be related to higher green expansion observed previously leading to some internal delamination which can affect sintered properties. On the other hand, similar TRS results were obtained for the Acrawax and EBS-25µm mixes under WDC-60°C and CC.

As for the effect of lubricant particle sizes on mechanical properties; the larger particle size lubricant EBS-25µm lead under WDC-60°C to 4-7% lower TRS values at high densities than lower particle sizes EBS, with values at 7.15 g/cm^3 of ~1230 MPa for EBS-25µm as compared to ~1275-1310 MPa. The same trend is observed for the fatigue behaviour of the four EBS mixes pressed by WDC-60°C, as illustrated in the stress vs. number of cycles in Fig.8 and in table 2. The EBS-25µm shows a decrease of ~8% of the endurance limit compared to the lower particle sizes lubricants (EBS-2.5µm, EBS-5µm and EBS-15µm). These results tend to suggest that the mechanical behaviour is only

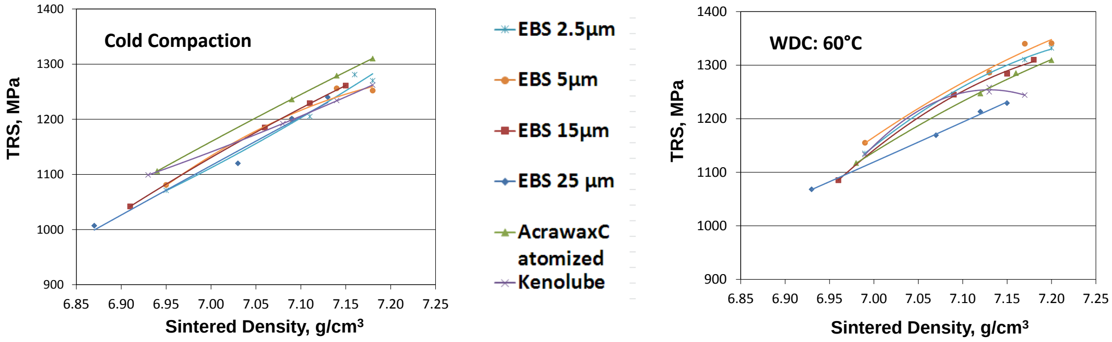

Fig. 7: Transverse rupture strengths vs. density for sintered TRS standard specimens pressed by CC and WDC-60°C

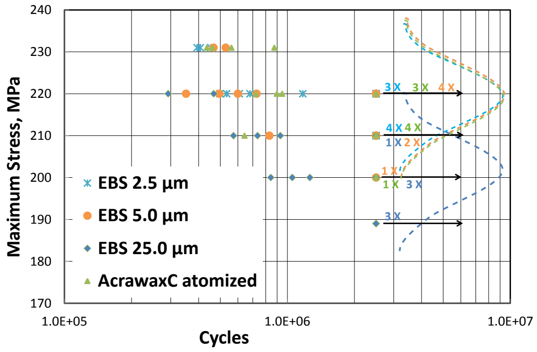

Fig. 8: Fatigue properties of 7.15g/cm^3 sintered TRS specimens pressed under WDC-60°C

Table 2:
Static and dynamic properties of 7.15g/cm^3 sintered parts pressed under WDC-60°C

	TRS (MPa)	Fatigue Limit (MPa)	
		$\sigma_{max50\%}$	STDEV
EBS 2.5 µm	1302	220	5
EBS 5.0 µm	1310	217	7
EBS 15.0 µm	1285	217	7
EBS 25 µm	1230	202	9
AcrawaxC atomized	1275	220	9

affected by the particle sizes of the lubricant when they reach a critical value. Another hypothesis would be that the greater green expansion with EBS-25µm under WDC-60°C might have induced some decohesion between particles resulting in slightly reduced mechanical properties.

CONCLUSIONS

In addition to their chemical formulations that affect both their efficiency and their cleanliness properties, the particle size distribution of EBS lubricants were shown in this paper to have a significant effect of the ejection efficiency. This was particularly true when parts were pressed by cold compaction on the industrial mechanical press. Indeed, for smaller particle size lubricant, the amount of lubricant expelled at the surface of parts was not sufficient to lubricate the die walls leading to an increase of the ejection forces when the rings were moving out of the die. The out-die sliding/stripping ratio was shown to be a good parameter to track this phenomenon and to better compare the behaviour of the different lubricants. Using WDC-60°C, this phenomenon was less pronounced which might be explained by the lower viscosity and/or higher deformability of the EBS lubricants at WDC-60°C that reduces the effect of the particle size distribution on their migration towards the exterior surface of the rings. In return, using EBS lubricant having a D_{50} particle diameter of ~25 µm led to a 4-8% reduction of both powder mix compressibility and mechanical properties as compared to mixes made with EBS lubricants having a D_{50} particle diameter of 15 µm or under.

ACKNOWLEDGEMENTS

The authors would like to acknowledge P. England, S. Mercier and D. Simard from NRC as well as O. Bouchard from RTMP that performed experimental work presented in this paper.

REFERENCES

[1] Lawrence, A.I.; Luk, S.; Hamill, J.A.; *"A Performance Comparison of Current P/M Lubricants and Routes to Improvement"*, MPIF, Adv. Powder Metall. Particulate Mater., 1997, Vol.1, pp. 4.3-4.21.

[2] Griffo, A. ; Marszalek, A.; German, R.M.; *"Statistical Analysis of lubricant particle size effect on ferrous P/M alloys"*, Int. J. Powder Metall., 1998, 34(5), pp. 55-65.

---

[3] Suzuki, H.; Lutheran, M.E.; "*Study of various lubricants for P/M ferrous materials",* MPIF, Adv. Powder Metall. Particulate Mater., 1998, pp. 11.15-11.24.
[4] Haynes, R.; "*Fatigue Behaviour of Sintered Metals and Alloys*", Powder Metall., 1970, 13(26), 465-510.
[5] Cimino, T. M.; Rutz, H. G.; Graham, A. H.; Murphy, T. F.; *"Effect of microstructure on fatigue properties of ferrous P/M materials"* , MPIF, Adv. Powder Metall. Particulate Mater., 1997, Vol. 13, pp. 137-149.
[6] Danninger, H.; Spoljaric, D.; Weiss, B.; *"Microstructural features limiting the performance of P/M steels*", Int. J. Powder Metall., 1997, 33(4), pp. 43-53.
[7] Beiss, P.; Lindlohr, S.; *"Porosity statistics and fatigue strength",* Int. J. Powder Metall., 2009, 45(2), pp. 39-48.
[8] Holmes, J.; Queeney, R. A.; *"Fatigue Crack Initiation in a Porous Steel"*, Powder Metall., 1985, 28(4), 231-235.
[9] Hardboletz, A.; Weiss, B.; *"Fatigue behaviour of iron based sintered material: A review"*, Int. Mater. Reviews, 1997, 42(1), 1-44.
[10] Drar, H.; *"Metallographic and fractographic examination of fatigue loaded PM-steel with and without MnS additive",* Materials Characterization, 2000, 45(3), 211-220.
[11] Chawla, N., & Deng, X.; *"Microstructure and mechanical behavior of porous sintered steels"*, Materials Science and Engineering A, 2005, 390(1-2), 98-112.
[12] Paris, V.; Thomas, Y.; St-Laurent, S.; *"Novel High Performance Lubricants for Conventional and High-Density Compaction"*, MPIF, Adv. Powder Metall. Particulate Mater., 2012, pp.3.35-3.47.
[13] "*Standard Test Methods for Metal Powders and Powder Metallurgy Products"*, MPIF, Princeton, 2012.
[14] W. Weibull; *"Fatigue testing and analysis of results",* Pergamon Press, New York, 1961.
[15] Beiss, P.; Broeckmann, C.; Drygalov, M.; Wassenberg, R. B.; Zafari, A.; "*Frictional Shear Stresses during Compaction and Ejection of Pure Iron in Tool Steel Dies"*, Proc. Int. Conf. on Tool Steels, 2009, Aachen, Germany.