Presented at the 2013 International conference on Powder Metallurgy & Particulate Materials held in Chicago, USA in June 2013 and published in the Advances in Powder Metallurgy & Particulate Materials-2013 conference proceedings available from the Metal Powder Industries Federation.

COMPARISON OF VARIOUS LUBRICANT SYSTEMS AND COMPACTION METHODS TO HIGH DENSITY APLICATIONS

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ABSTRACT

Achieving high density at relative low cost by single pressing/single sintering is of prime importance for the PM industry. Compacting methods such as warm compaction and warm-die compaction are known to provide enhanced green density when utilized with powder mixes specifically designed for these methods. The warm compaction and warm-die compaction are both using moderate compacting temperature, typically in the range of 60 to 130 °C, to enhance density. The die wall lubrication method is also known as an effective method to reach high density, mainly by limiting the amount of internal lubricant which limits the densification. In all cases, lubricant systems are of prime importance and must be especially efficient under high compacting pressure/temperature to ensure achievement of high density, excellent lubrication and very stable physical properties at the working temperature in order to achieve excellent part weight and density consistency. This paper describes the compaction and ejection behaviour of the recently developed HD warm die compaction lubricating system as compared to other systems designed for warm compaction and die wall lubrication.

INTRODUCTION

One of the main objectives of the PM steel market is the achievement of high density PM components, as static and dynamic properties are improved with increased density, while maintaining a reasonable cost. Sintered properties can be improved by several methods such as: alloying, liquid phase sintering, high temperature sintering and a variety of heat treatments. But in all cases, density of PM parts remains as one of the most important parameters controlling sintered properties. Depending on the compressibility of the iron base powder, the amount of additives and lubricant, part geometry and height, one can reach maximum densities of 7.05 to 7.25 g/cm^3 by conventional compaction. To achieve higher densities of 7.4 to 7.5 g/cm^3 double pressing/double sintering is employed as well as forging to attain full density[1]. Nevertheless, the production costs and cycle time associated with the latter techniques are quite significant, limiting market penetration.

The final sintered density and strength of PM parts are not only driven by the powder formulation, but also by the compaction process and compacting conditions used, the part characteristics and the sintering behavior. Therefore, densification and ejection performance of powder mixes remains one of the main factors to address when targeting high densities. The density level achieved when compacting a powder mix in a closed die is mainly a function of the intrinsic ability of the powder compressibility, the friction between the pressed powder particles and the die walls, and of the springback of the compacted part after ejection[2].

During the last decades many efforts were put together to develop new techniques that could reach densities above 7.2 g/cm^3 by single pressing/single sintering processes. Compaction techniques such as warm compaction (WC), which consists of pressing a preheated powder mix in a heated die and warm die compaction (WDC), where only the die is preheated[3], are very interesting alternatives allowing the achievement of higher density compared to cold compaction and at a lower cost compared to double pressing/double sintering and forging techniques. Typically, final densities in the range of 7.25 to 7.45 g/cm^3 can be achieved after a single press/single sinter step with WC and WCD[4]. These techniques enable the fabrication of parts with high density and green strength by increasing the ductility of the ferrous powder particles[5,6,7].

The die wall lubrication technique (DWL) is another single pressing method used for high density parts. This technique has been the object of several studies in recent years[8,9,10,11,12,13]. With the DWL technique, the level of internal lubricant in pre-mixes can be kept very low, typically at around 0.2%, allowing an increase of the green density while maintaining good lubrication at die walls during the compaction and ejection of parts[14,15,16].

In fact, for single compaction processes, the amount of lubricant has a strong effect on the maximum density that could be achieved during compaction, as lubricant has a very low specific gravity (~ 1 g/cm^3) compared to metallic powders. Indeed, the lower the lubricant content, the higher the maximum achievable density. On the other hand, the lubricant must also perform properly to facilitate ejection and produce parts with a good surface finish and no defects such as delamination, small holes, etc. Also die and tool wear shall be avoided, as lubricant reduces friction at die walls and ensures a good transfer of the compaction force throughout the part. The lubricity at the die wall is known to be proportional to the level of internal lubricant added to the mix, with exception for the DWL technique. Therefore, new lubricants must be more performing in order to be able to lower their concentration as much as possible while maintaining excellent ejection behavior. This is why a significant amount of effort has 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: 17, 18, 19, 20, 21 and 22. More particularly, a new lubricant system specifically designed for the WDC technique, called HD2, is characterized in this paper. Additional information on its characteristics can be found in reference 23.

The current paper compares the compaction and ejection behaviour, the green and sintered properties of mixes pressed with different high density compaction techniques, the WDC, WC and DWL. Lubricant systems specifically designed were used for the WDC and WC, while a conventional and an HD lubricant were used with the DWL technique. A mix containing 0.75% EBS wax was also pressed with the WDC technique and used as a reference.

EXPERIMENTAL PROCEDURE

Mixes containing different lubricating systems specifically designed for the warm die compaction (WDC), warm compaction (WC) and die wall lubrication were prepared in laboratory. The lubricant HD2, a proprietary lubricant described in reference 23 was used at two concentrations for WDC. A FLOMET WP mix was used for the WC process and two different mixes were prepared for the DWL technique. Also, as a benchmark a mix containing 0.7% ACRAWAX C atomized was prepared (called Ref). All powder mixes were prepared using ATOMET DB46, a diffusion alloyed steel powder containing 0.5% Mo – 1.75% Ni – 1.5% Cu produced by Rio Tinto Metal Powders mixed with 0.5% graphite and lubricants. Table 1 gives the mix names, lubricant contents and compaction methods used to compact rings with the mechanical production press.

Green and sintered properties were also measured with standard TRS specimens pressed with a laboratory hydraulic press at 552, 689 and 827 MPa according to MPIF Standards 15, 41-44[24]. The die was heated at 60 °C for all mixes except FLOMET WP which was pressed at 125°C. These conditions were used to obtain part temperatures similar to those achieved in the industrial press. For the DWL, Zn stearate was sprayed on the die wall prior to compaction of TRS specimens which were afterward sintered at 1130°C for 25 minutes under a nitrogen atmosphere containing 10% H_2.

Table 1. Description of mixes and compaction techniques used for this study.

Mix	Lubricant	Compaction Method & Temperature
Ref	0.7% Wax	WDC/60°C
0.3%HD2	0.3% HD2	WDC/60°C
0.3%HD2-DWL	0.3% HD2	DWL/60°C
0.5%HD2	0.5% HD2	WDC/60°C
0.1%Wax-DWL	0.1% Wax	DWL/60°C
FLOMET WP	0.57% WP Lube+Binder	WC/115°C

Compaction and ejection behaviour was evaluated for five rings pressed on a 150 tons 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 National Research Council Canada. For this study, a tungsten carbide (WC/Co) die, 25.4 mm in diameter with a core pin of 14.2 mm in diameter, was used. Rings about 12.7 and 25.4 mm tall were compacted at a stroke rate of 5 parts/min. In order to construct compressibility curves, parts were pressed at different compaction pressures of about 480, 620, 710 and 820 MPa. Die temperature was set at 40 and 60 ^{o}C for the HD2 mixes, DWL and the reference mix. However, only results obtained at 60°C are presented in this paper. For DWL, Zn stearate was initially sprayed on the die walls but ejection behavior was definitively not appropriate. It was then decided to use our proprietary lubricant that was developed in a previous project[15]. For FLOMET WP, initial compaction tests were carried out with three die temperatures, 110, 115 and 125 ^{o}C to define the optimum temperature. The powder was not heated for these tests, as it is normally the case, so a waiting time of 2 min was used. The best results were obtained at 115°C, so that temperature was used to compare with the other mixes for the rest of the study. After each test, a standard mix with excellent ejection behavior 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 die/tool was not affected. In order to obtain data for the ejection shear stresses, the press monitoring software outputs an ejection curve for each stroke (Figure 1). Then, these ejection curves were treated with an in-house developed software to extract the stripping force (the force required to start the ejection movement), the out-die sliding 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 the procedure for data treatment can be found in Paris et al.[25].

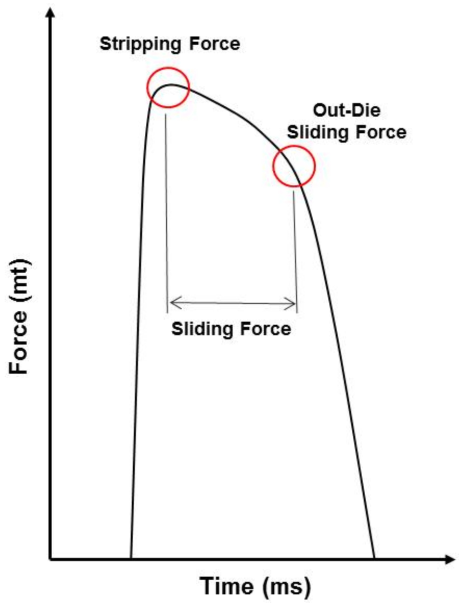

Figure 1. Schematic of a typical ejection curve, when monitoring the forces, on the mechanical press.

Measurements of height, weight and green density were taken for each part. The Archimedes method as well as the use of a coordinate measuring machine (CMM) were used for the density measurement. Another key characteristic to evaluate is the springback, since at high densities parts may expand significantly at ejection potentially causing delamination, small defects or a bad surface finish. To determine the radial springback (swelling of part after ejection of the die, expressed as a percentage), 40 measurement points around the circumference of the part, at mid height, were recorded with a CMM apparatus, model SmartScope Flash 300, having a precision of 1.5 µm.

RESULTS AND DISCUSSION

Results at 12.7 mm high parts

Figure 2 compares the compressibility curves for the HD2, DWL and REF mixes pressed at 60°C and WP mix pressed at 115 ^{o}C. Both sets of HD2 and DWL mixes follow similar trend, at every compacting pressure the green density obtained is almost the same. For the reference mix at pressures below 600 MPa the green densities obtained are similar or even higher than the latter cases, but at pressures above the 600 MPa green density considerably diminishes. At about 820 MPa, the green density obtained with the reference mix is 7.22 g/cm^3, while all the other mixes reach between 7.28-7.31 g/cm^3. The difference in density achieved with the different systems is not significant and proved that the new HD system can be used with the WDC technique to achieve similar density than the WC process at high pressures. Even DWL did not allow increasing density. Also, reducing the lubricant content from 0.5 to 0.3% only resulted in an increase in density of ~ 0.03 g/cm^3. FLOMET WP is significantly more compressible than the other mixes for low compacting pressures, the green densities being about 0.10 g/cm^3 higher for pressure between 400 and 600 MPa. The gain in density obtained with FLOMET WP is known to be linked to the improvement of the intrinsic compactability of the base power as temperature is increased. However, similar green density is achieved at higher pressure, due to the presence of internal lubricant which limits the pore free density, and thus the maximum practical density achievable.

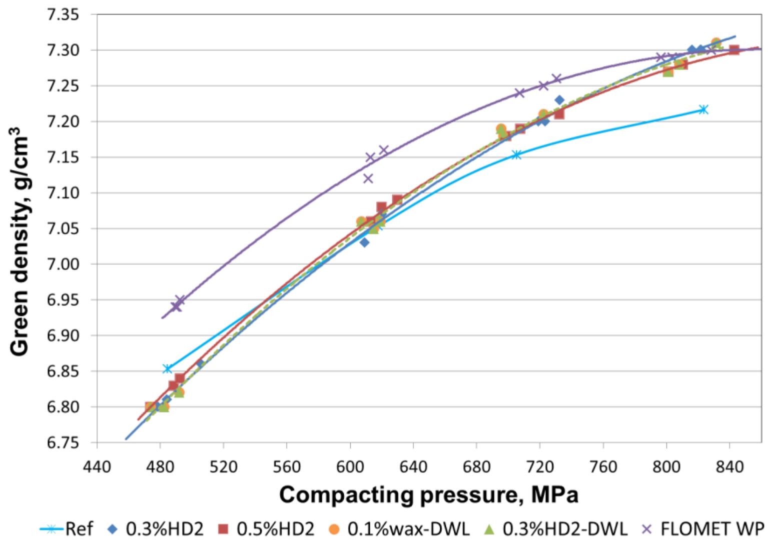

Figure 2. Compressibility curves for HD2 mixes, DWL mixes and the reference mix WDC at 60 °C along with FLOMET WP mix WC at 115 °C.

The stripping, sliding and out-die sliding shear stresses are also compared for the HD2, DWL and WP systems as well as the reference mix in Figure 3. Mixes processed with the DWL technique gave much lower ejection shear stresses than the other mixes/compacting systems, around 50% lower. Mix with 0.3% HD2 gave the lowest ejection stress amongst the 2 DWL mixes tested, confirming the role of the internal lubricant. This was expected as the use of a DWL technique with an appropriate lubricant deposit a uniform layer of lubricant on the die wall creating an interface between the die and the part that allows decreasing the ejection shear stresses. The excellent ejection performance is maintained at all compacting pressure tested, even at pressure above 800 MPa[26]. Besides the DWL systems, all other mixes show much higher shear stresses, which are on the other hand typical for mixes with internal lubricant pressed with more conventional compacting methods. Mix with 0.5%HD2 and FLOMET WP (0.57% binder/lube) behave as the reference Mix (0.7% lube) for the stripping shear stress, while 0.3%HD2 presents stripping shear stress up to 10-20% higher versus the others. For the 0.3%HD2 mix, all ejection shear stresses measured increase with compacting pressure.

We can also observe in Figure 3 that the ejection shear stress drops during the ejection process for all the mixes (Stripping> Sliding > Out-Die Sliding). The drops in ejection gives a very good estimate on how efficient is the lubricant during ejection movement. A calculation that is done to evaluate the efficiency of lubrication consists in dividing the out-die sliding shear stress by the stripping shear stress. The result is a ratio that indicates how the shear forces vary during ejection, the lower the ratio, the better the behavior of the lubricant under dynamic conditions[23]. As seen in Figure 4, mix with 0.5% HD2 lubricant gave the lowest ratio amongst the mixes/compacting methods tested. Mix with 0.3%HD2 and 0.7%EBS wax (Ref) behave quite similarly while mixes 0.1%wax-DWL and FLOMET WP gave the highest ratio. In particular, as the pressure is increased the ratio increases for mix 0.1%wax-DWL. This is an indication that the internal lubricant (0.1%wax) is not sufficient. Much better behavior is obtained with 0.3%HD2-DWL mix. Finally, it is seen that very high ratio is obtained with FLOMET WP for compacting pressure of 700 MPa or lower. However, the ratio decreases quite significantly when pressure reaches 800 MPa, suggesting that the WP lubricant+binder system requires high pressure to effectively migrate at the die walls and performs better as density approaches the pore free density. It also indicates that the lubricant system sustains very well the high pressure at the die wall.

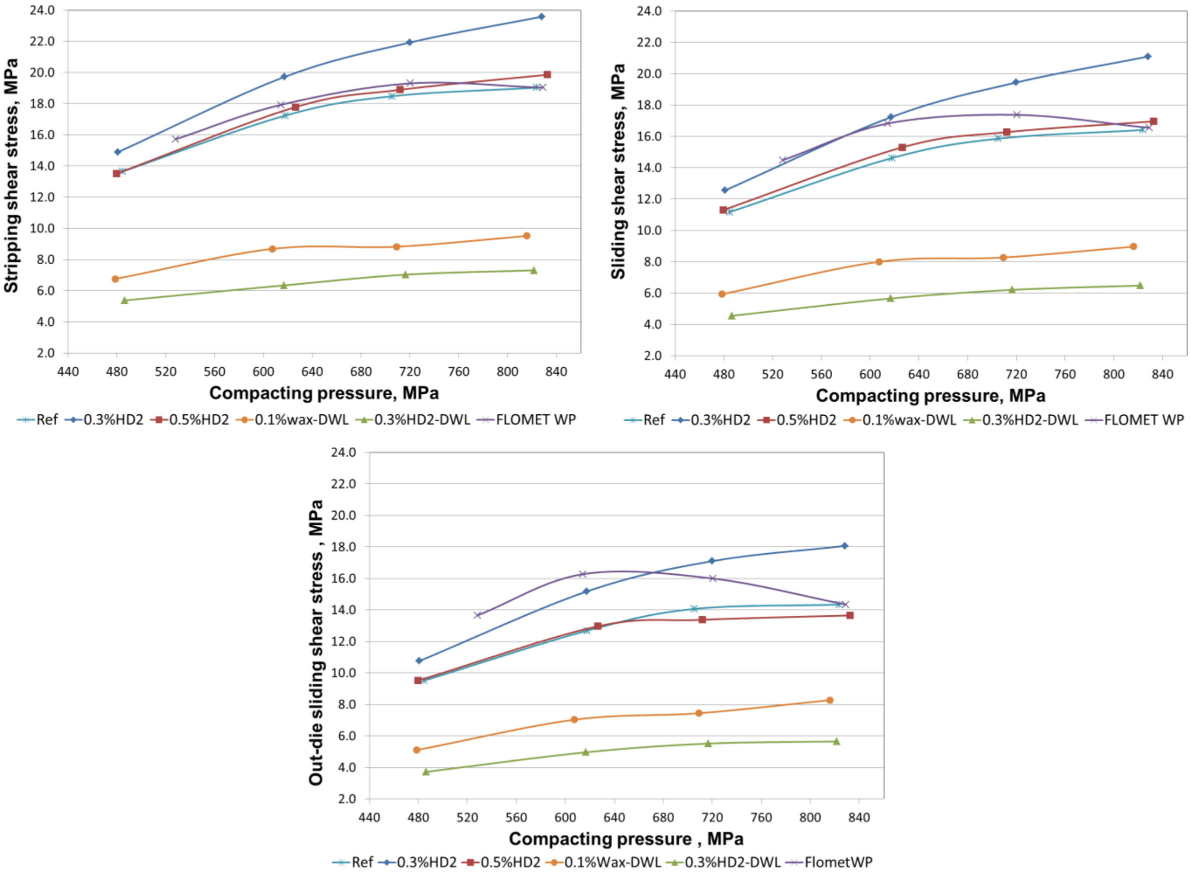

Figure 3. Stripping, sliding and out-die sliding shear stresses for HD2 mixes, DWL mixes and the reference mix WDC at 60 °C along with FLOMET WP mix WC at 115 °C.

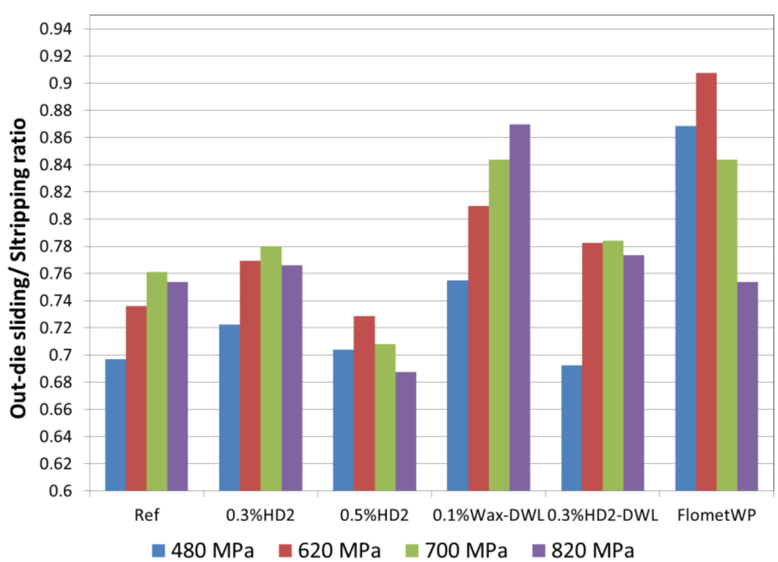

Figure 4. Out-die sliding/Stripping ratio at different compaction pressures for HD2 mixes, DWL mixes and the reference mix WDC at 60 °C along with FLOMET WP mix at 115 °C.

The expansion of the parts after ejection is denoted as radial springback. Springback is dependent on part size or aspect ratio, base powder intrinsic characteristics, die stiffness and compacting conditions. Since in the current experiments the part geometry, base powder and die were kept constant, the differences in springback result from the compacting technique and the lubricant used. It is expected to have larger part expansion after ejection with mixes containing larger amounts of lubricants/binders and when reaching high densities (such as at high compacting pressures). The nature of lubricant is also very important. As depicted in Figure 5, all systems had a relatively similar springback, which remains below 0.28% in the worst case. Only at lower compacting pressure FLOMET WP (even when having 0.57% lubricant) resulted in the smallest springback despite the fact that it reaches the highest green densities for given compaction pressures. Mixes with 0.5%HD2 and 0.3%HD2-DWL give the highest springbacks for compacting pressure above 650 MPa. Interestingly, Mix with 0.3%HD2 pressed by WDC gives lower springback than the same mix processed by DWL, suggesting that the external lubricant may have an effect on the springback by preventing air or internal lubricant to be expelled out of the parts. It is worth mentioning that reducing the compacting temperature with HD2 helps reducing the springback. Indeed, tests ran at 40 ^{o}C showed that the 0.5%HD2 mix had springbacks similar or even slightly lower than the reference mix.

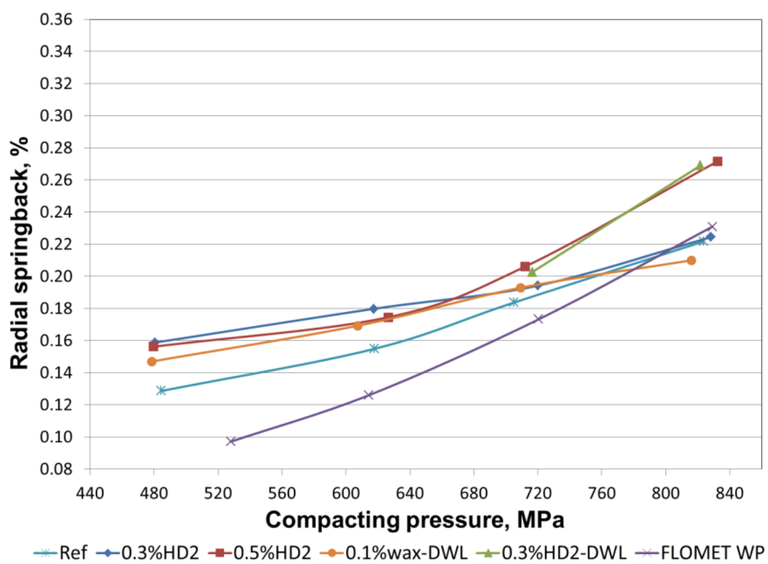

Figure 5. Radial springback for HD2 mixes, DWL mixes and the reference mix WDC at 60 ^{o}C along with FLOMET mix WP at 115 ^{o}C.

Results at 25.4 mm high parts

Figure 6 presents the compressibility curves of parts two times taller for the HD2 mixes (0.3 and 0.5%), 0.3%HD2-DWL, FLOMET WP and compares them to the reference mix. First of all, for all mixes, the maximum density achieved at ~ 820 MPa was lower by 0.01 to 0.06 g/cm^3 versus density achieved with 12.7 mm parts. The drop in density was particularly noticeable for FLOMET WP, especially at compacting pressure lower than 700 MPa. It is also interesting to note that the reference mix gave very good density for low compacting pressure, clearly indicating that for tall parts, higher lubricant content is beneficial for densification. It is observed that the 0.3%HD2 mix leads in higher green densities than the reference mix above 720 MPa, while for the 0.5%HD2 mix this occurs earlier at 640 MPa. At about 720 MPa the HD2 mixes surpass the FLOMET WP mix in green densities. At all compacting pressures the 0.3%HD2-DWL mix performs better obtaining the highest green densities. As it was the case for 12.7 mm parts, difference in maximum density between WDC, WC and DWL is very small. These tests also confirm the benefit of using the new HD2 system with a friendly user compacting method versus the two other methods in terms of density.

Figure 7 shows the ejection shear stress for 25.4 mm tall parts. Very similar trends as those observed for 12.7 mm tall parts can be seen. Indeed, as it was the case, the DWL provides the lowest stripping, sliding and out-die sliding shear stresses while 0.3%HD2 mix pressed by WDC gives the highest values, the difference increasing with compacting pressure. The 0.5% HD2, FLOMET WP and the reference mixes give very similar ejection stresses. The global trend resembles that seen on 12.7 mm parts, and despite the fact that the part height was doubled the ejection shear stresses were quite similar. Only the DWL technique shows an increase of the ejection pressure for taller parts.

The out-die sliding/stripping ratio is presented in Figure 8 for 25.4 mm tall parts in order to evaluate the efficiency of lubrication as done with 12.7 height parts. Contrary to what was observed on 12.7 mm parts, the FLOMET WP mix shows the lowest ratio that is further improved with increased pressure. Mixes with 0.3%HD2-DWL, 0.5%HD2 and 0.7%EBS behave quite similar. The mix containing 0.3%HD2 resulted in the highest ratio as the out-die sliding shear stress remained elevated.

Figure 9 shows the radial springback as a function of the compacting pressure for the 25.4 mm tall parts. Overall, springback was slightly higher at 25.4 mm than at 12.7 mm, more particularly at higher pressures. The FLOMET WP mix maintains the lowest expansion after ejection as it was the case at 12.7 mm. On the other hand, mix with 0.3%HD2-DWL gives the highest springback at all compacting pressures, similarly to what was observed at 12.7 mm.

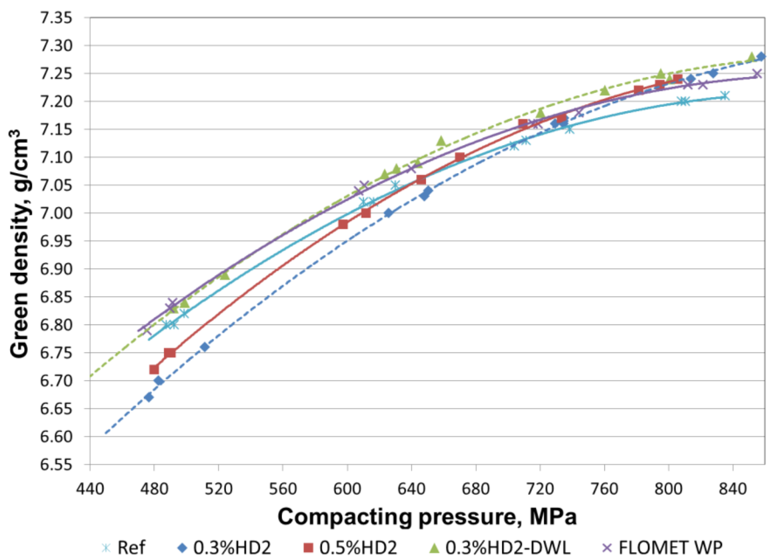

Figure 6. Compressibility curves for HD2 mixes, HD2-DWL mix and the reference mix WDC at 60 °C along with FLOMET WP mix at 115 °C for parts 25.4 mm height.

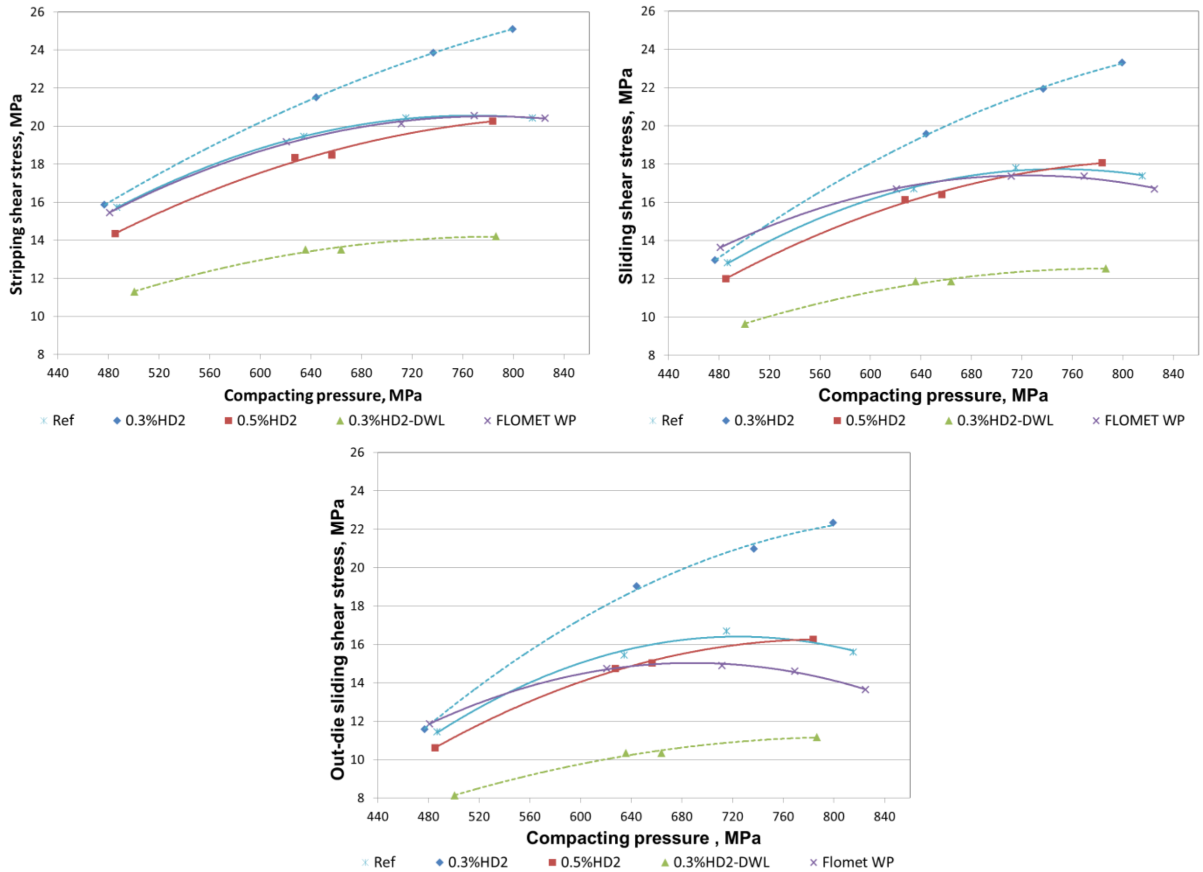

Figure 7. Stripping, sliding and out-die sliding shear stresses for HD2 mixes, 0.3%HD2-DWL mix and the reference mix WDC at 60 °C along with FLOMET WP mix at 115 °C for parts 25.4 mm height.

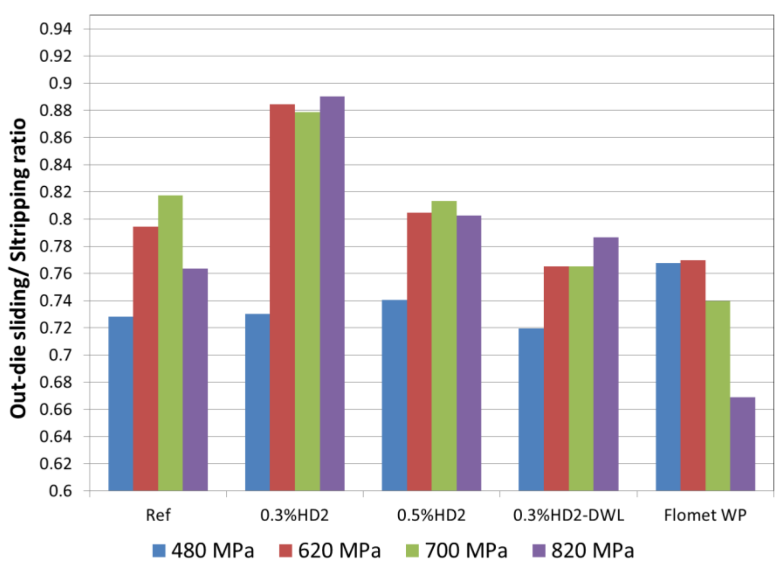

Figure 8. Out-die sliding/Stripping ratio at different compaction pressures for HD2 mixes, 0.3%HD2-DWL mix and the reference mix WDC at 60 °C along with FLOMET WP mix at 115 °C for parts 25.4 mm height.

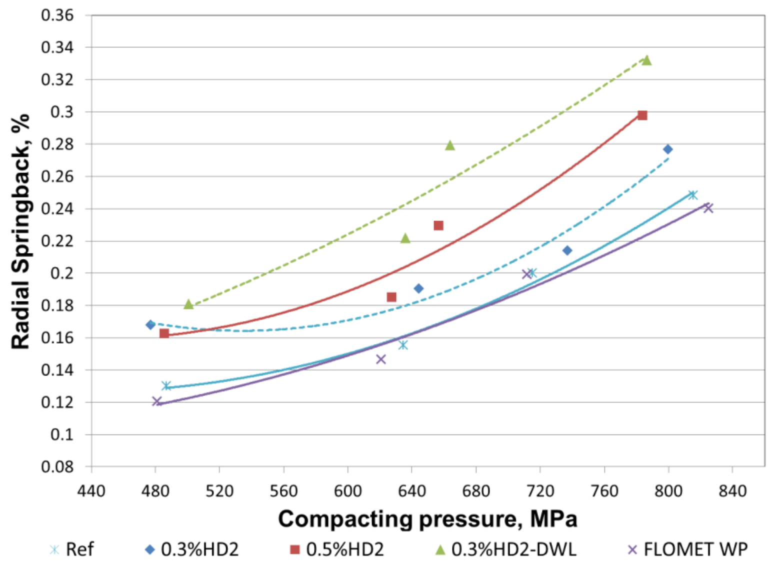

Figure 9. Radial springback for HD2 mixes, 0.3%HD2-DWL mix and the reference mix WDC at 60 ^{o}C along with FLOMET WP mix at 115 ^{o}C for parts 25.4 mm height.

Green and sintered properties as measured in laboratory

Green and sintered properties were evaluated by pressing standard TRS specimens at three different compacting pressures (552, 689 and 827 MPa). The results are shown in Figure 10. As anticipated, green density increases with compacting pressure for all the systems tested. Much higher density was obtained compared to the tests carried out in the production press. Indeed, densities were between 0.08 to 0.15 g/cm^3 higher than those previously presented. This is explained by the M/Q ratio, which is defined as the lateral area over compacted area, the higher the ratio the larger the die wall friction, therefore the greater the difficulty to compact to higher densities[27]. This ratio is 4.54 for the rings (12.7 mm height) while it is only 1.4 for a 6.35 mm height standard TRS bar.

For green strength, the HD2 lubricants and EBS wax gave quite similar results while the mix pressed by DWL gives slighlty higher green strength, namely because of the low concentration in lubricant. As expected, FLOMET WP mix reaches remarkable green strength values up to 47 MPa, which is more than double of what the other mixes provided. This is the result of the higher compacting temperature used and the proprietary WP lubricant/binder system.

After sintering, significant difference in dimensional change from die size was observed between the materials, partly due to the difference in green density but mainly to the lubricant system itself. The HD2 lubricant favors less shrinkage during sintering. On the other hand, FLOMET WP performs very similarly to the reference mix. For all mixes, increasing the compacting pressure resulted in more positive dimensional change. As expected, TRS and apparent hardness increase with density. However, it is interesting to note that the TRS increases much more significantly for the 0.1% wax mix pressed by DWL. This is due not only to the higher density reached at a given compacting pressure but also to the lower level of internal lubricant, which is known to favor higher strength. This is clearly an advantage for the DWL process. Mix with 0.3% HD2 also shows a higher increase in TRS while reaching higher densities than the other mixes, confirming the benefit of using less lubricant on sintered properties.

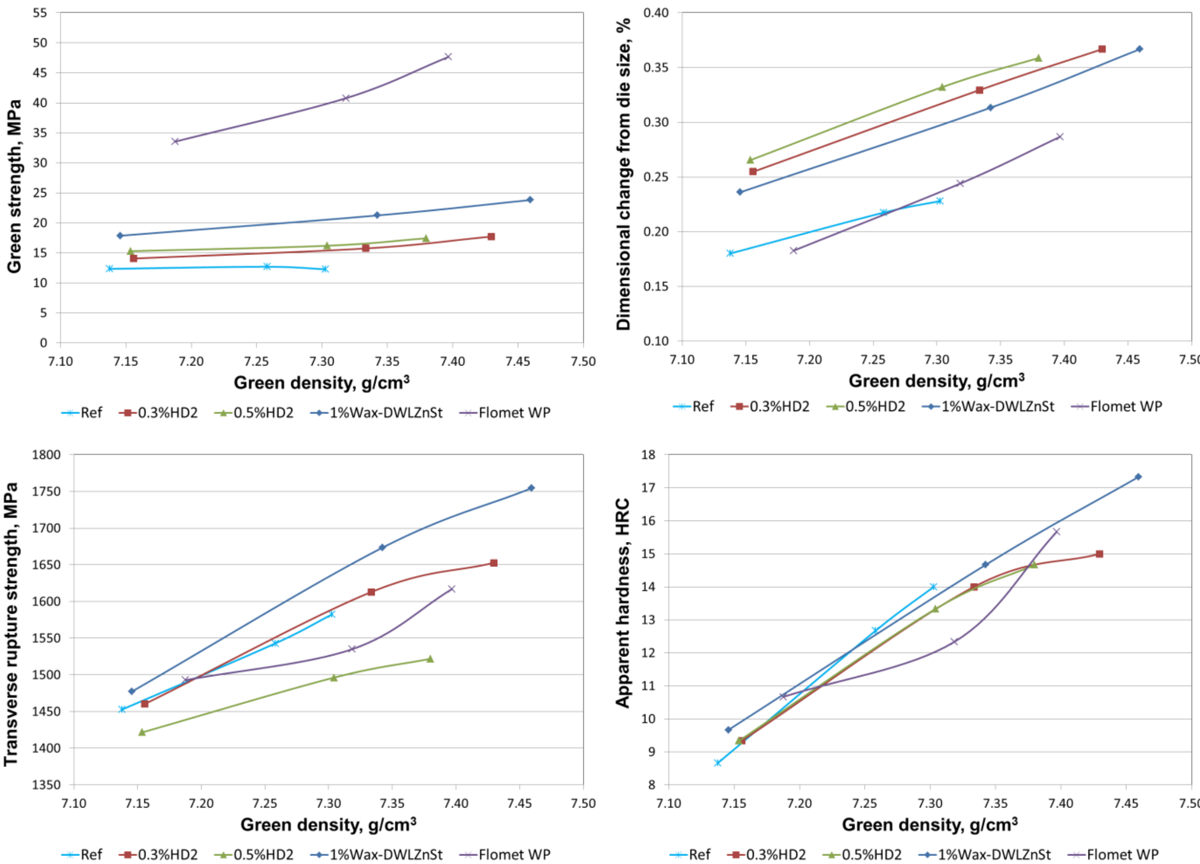

Figure 10. Green and sintered properties measured on standard TRS specimens for the HD2 mixes, 0.1%Wax-DWL and FLOMET WP mix.

CONCLUSIONS

The objective of this study was to compare different high density compacting methods. All compacting methods tested (WC, WDC and DWL) with appropriate lubricant systems (HD2, FLOMET WP) show certain advantages and disadvantages ones from the others. It merely depends on the final product requirements and technology available that will allow a PM manufacturer to choose the appropriate system. The advantage/disadvantages of the different method are described below.

- The warm compaction (WC) method using the FLOMET WP proved to be very good performer when higher densities should be reached at lower pressures. The compressibility curve obtained for the 12.7 mm height parts was indeed significantly higher for all the pressure range, while maintaining the lowest springback. Nevertheless, gain in density is much lower when part height is increased.
- FLOMET WP mix ejection behavior is also similar to a conventional mix with 0.7% wax except that it is less efficient during sliding as indicated by the high out-die sliding/stripping ratio. The lubricant performance improves significantly at high compacting pressures above 800 MPa.
- The use of DWL with a performing external lubricant and an optimized mix containing an efficient lubricant (as HD2) can reach high green densities with extremely low ejection shear stresses at all compacting pressures. Therefore, for complex parts (or parts having a bigger aspect ratio) 0.3%HD2-DWL system gives the best results.

- A new lubricant system for WDC, called HD2 allows reaching very similar green densities at high compacting pressure to the WC and DWL techniques, which are more difficult to monitor on a day to day production basis (die wall lubricant or heated powder with higher compacting temperature). In addition the HD2 lubricant system provides ejection performance similar to conventional wax at much lower concentration and the best dynamic performance as indicated by the very low out-die sliding /stripping ratio obtained.
- Due to its excellent dynamic ejection behavior, lubricant HD2 maintains its good ejection properties for taller parts (25.4 mm). A concentration of 0.5% HD2 is recommended for both heights tested in this study.
- Lowering the lubricant content favors an increase of sintered strength (TRS). In that regard, using DWL system with an appropriate external lubricant and a mix with low concentration of internal lubricant allow achieving high density parts, excellent ejection performance at least 2 times lower than standard press-ready mixes and better sintered properties, especially at high compacting pressures.
- Good green and sintered properties are achieved with the HD2 lubricants, very similar or slightly lower values result from the increase of the organic content, as seen for 0.3%HD2 versus 0.5%HD2.

The new HD formulation (HD2) compacted by the WDC technique appears as the best compromise among the different methods in terms of simplicity and robustness of the process, by reaching similar high densities at high pressures while maintaining excellent ejection performances despite the low level of admixed lubricant (<0.5%wt). Nevertheless, research work is still ongoing to further improve the compaction and ejection performance of lubricants to be used with the cold compaction and warm-die compaction techniques, especially for bigger and/or more complex parts.

ACKNOWLEDGEMENTS

The authors wish to express their gratitude to the following people for their dedicated involvement in the present study: Olivier Bouchard (RTMP) for the preparation and characterisation of mixes in the quality control laboratory, and Patrick England and Daniel Simard (NRC-Canada) for all the tests on the industrial press and characterization of parts.

REFERENCES

1 F. Chagnon and S. St-Laurent, “Optimizing Powder Mix Formulations and Processing Conditions for Warm Compaction”, *Proceeding of the 2000 Powder Metallurgy*, World Congress in Kyoto, Japan, November 2000.

2 S. St-Laurent, F. Chagnon and Y. Thomas, “Study of Compaction and Ejection Properties of Powder Mixes Processed by Warm Compaction”, *Adv. Powder Metall. & Particul Mater.*, 2000, pp.3.79-3.91

3. V. Musella and M. D'angelo, "Process for Preheating Metal in Preparation for Compacting Operations", *U.S. Patent* No. 4,955,798.

4 F. Chagnon and Y. Trudel, “Effect of Compaction Temperature on Sintered Properties of High Density P/M Parts”, *Advances in Powder Metallurgy and Particulate Materials*, MPIF, Princeton, N.J., 1995, Vol. 2, p. 5-3.

5 F. Chagnon, C. Gélinas and Y. Trudel, “Development of High Density Materials for P/M Applications”, *Adv. Powder Metall.& Particul. Mater*, Vol.3, 1994, pp. 199-206.

6 M. Gagné, “Warm and Cold Compaction”, *Adv. Powder Metall.& Particul. Mater*, Vol.1 , 1997, pp.319-206.

7 G.F. Bocchini, “Warm Compaction of metal powders. Why it works, why it requires a sophisticated engineering approach.”, *Adv. Powder Metall. & Particul Mater.*, Vol.1, 1998, pp.4.29-4.42

8 W.G. Ball, P.F. Hibner, F.W.Hinger, J.E.Peterson and R.R.Phillips, “Replacing internal with external lubricants: Phase III. Tribostatic Application of Lubricants onto Die Walls” *Advances in Powder Metallurgy & Particulate Material*, Vol. 2, Part 6, compiled by Terry M. Cadle and K.S. Narasimhan, Metal Powder Industries Federation, Princeton, NJ, 1996, pp. 3-15.

9 Technical data sheet Gasbarre Products Inc.

10 I. I. Inculet, J. D. Brown, P. Castle and P. Hansen, “Powder Metallurgy Apparatus and Process Using Electrostatic Die Wall Lubrication”, *U.S. Patent* No. 5,682,591.

11 P.F. Hibner, R.C.Bennett and J.C.Costello, “Apparatus for Dispensing Lubricating Powder”, *U.S. Patent* No. 5,992,772.

12 T. M. Cadle, J.H. Mandel and P.R. Roskopf, “Dry Die Wall Lubrication”, *U.S. Patent* No. 6,190,605 B1.

13 P-E. Mongeon, S. Pelletier, A. Ziani, “Die Wall Lubrication method and Apparatus”, *U.S. Patent* No. 6,299,690.

14 P. Lemieux, S. Pelletier, P.-E. Mongeon, L.P. Lefebvre, Y. Thomas, F. Chagnon, “A New Approach to Die Wall Lubrication for P/M Applications”, *Adv. Powder Metall. & Particul Mater.*, 2001, pp.3.1-3.13.

15 P. Lemieux, L. Azzi, Y. Thomas, S. Pelletier, P.E. Mongeon, S. St-Laurent, “Pressing Challenging Parts on a Production Scale by Using Die Wall Lubrication Technology”, *Advances in Powder Metallurgy and Particulate Materials- 2005*, MPIF, Princeton (USA), 1995, Vol. 2, p. 3.71-3.83.

16 P. Lemieux, K. Chattopadhyay and M. Hasan, “Computer Modelling of the Die-Wall Lubrication Process Using the Confining Block”, *Advances in Powder Metallurgy and Particulate Materials-2010*, *MPIF*, Hollywood (USA)

17 D. L. Hammond, “ Lubricant system for use in powdered metals”, *US Patent* 6,679,935, issued on January 20, 2004.

18 F. G. Hanejko, “Methods for preparing metallurgical powder compositions and compacted articles made from the same”, *US Patent Application* 20070186722, filed on August 15, 2006.

19 S. St-Laurent, Y. Thomas and L. Azzi, “High Performance Lubricants for Demanding PM Applications”, *Proceedings of the International Conference On Powder Metallurgy and Particulate Materials,* 2006, San Diego, California, , 2006-06-18

20 L. Azzi, Y. Thomas and S. St-Laurent, “Lubricants for high-density compaction at moderate temperatures”, *International Journal of Powder Metallurgy*, 2007, Vol. 43 (6).

21 A. Ahlin, A. Ahlqvist and O. Litström, “Newly developed lubricants for high performance metal powder mixes”, *Euro PM2008*, International Powder Metallurgy Congress & Exhibition, Mannheim, Germany, September - October 2008; EPMA, Vol. 3.

22 L. Wimbert, K. Dollmeier, V. Kruzhanov and R. Lindenau, “Advanced Lubricants – A Complex Challenge for Powder Metallurgy: Density, Lubricity”, *Environment*, Proceedings of the World PM2010 Congress & Exhibition, Florence, Italy, October 2010, EPMA, Vol. 1.

23 S. St-Laurent, V. Paris and Y. Thomas, “Performance of Lubricants for High Density Applications”, *Powder Metallurgy World Congress and Exhibition.* 2012, Yokohama, Japan, 10-14-2012.

24 *MPIF Standard Test Methods for Metal Powders and Powder Metallurgy Products – 2012 Edition*, Princeton, NJ (USA): Metal Powder Industries Federation, 2012.

25 V. Paris, Y. Thomas, and S. St-Laurent, “Novel High Performance Lubricants for Conventional and High-Density Compaction”, *Proceeding of 2012 International Conference on Powder Metallurgy and Particulate Materials*, Princeton, NJ (USA), MPIF, 2012

26 P. Lemieux, Y. Thomas, P.-E. Mongeon, S. Pelletier and S. St-Laurent, “Combining Electrostatic Die Wall Lubrication and Warm Compaction to Enhance Green and Sintered Properties of P/M Components”, *P/M World Congress*, Orlando, Florida, 2002.

27 E. Ernst, F. Thummler, P. Beiss, R. Wähling and V. Arnhold, “Friction Measurements during Powder Compaction”, *Powder metallurgy Int.*, Vol.23, 1991, pp.77-84.