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ACHIEVING HIGH DENSITY BY SINGLE COMPACTION OF STEEL POWDER PREMIXES

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ABSTRACT

Achieving high density at relative low cost by single pressing/single sintering is of prime importance for the P/M 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 technologies. 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 gain in density due to temperature is typically of the order of 0.001 to 0.0015g/cm³/°C for steel powders. In addition to that, the powder mixes must provide excellent lubricity and very stable physical properties at the working temperature in order to achieve excellent part weight and density consistency. Furthermore, the mix, including both base powder and additives, must be formulated and optimized to maximize the pore free density, hence the green and sintered densities.

The paper describes special lubricant systems designed for high density applications. In the first part, FLOMET WP, a lubricant/binder system specifically engineered for the warm compaction process by Rio Tinto Metal Powders is presented and the properties of FLOMET WP are discussed in details. A case study describing how the FLOMET WP and the warm compaction technology are used for the production of high density structural components is then presented. In the second part, the properties of new type of lubricants designed for cold or warm-die compaction are presented. Results of a test carried out in real production conditions with these new lubricants are then presented and compared to that of parts produced with the warm compaction process.

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, which must compete against the machined, cast or forged counterparts in highly stressed applications. It is well known that 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 properties such as fatigue and impact strength, and achievement of high density at the lowest possible cost is crucial for the PM industry. Techniques such as the double press/double sintering (DPDS) and forging are well known and used to achieve densities in the range of 7.4 to 7.5 g/cm³ and to nearby full density respectively [1]. However, the production costs and cycle time associated with these techniques are quite significant, which limit to some extent the penetration of PM parts produced via these techniques. On the other hand, single pressing/single sintering significantly reduces the production cost versus DPDS and forging but densification is more limited and challenging, mainly because of the presence of an internal lubricant which limits the maximum density achievable and the high pressure required which may cause severe tooling damage and defects in the part. Compaction techniques such as warm compaction (WC) and warm-die compaction (WDC) were introduced to achieve higher green densities than the conventional cold compaction. These compaction techniques, which consist in pressing a pre-heated powder (WC) or a non-heated powder (WDC) in a heated die, take advantage of the improvement of the steel powder intrinsic compactability when compacting temperature is moderately increased. Gain in density due to temperature can reach up to 0.15 -0.20 g/cm³. The die wall lubrication technique (DWL) is another single pressing method used for high density parts. With the DWL technique, the level of internal lubricant in pre-mixes can be kept very low, typically at around 0.2%, which allows an increase of the green density achievable during compaction. However, the deposition of a uniform layer of lubricant on the die wall for every part in mass production remains a major challenge for that technology, especially in the case of long and complex parts. Significant progresses in that regard were reported by Lemieux et al [2, 3]. In summary, green densities varying between 7.25 to 7.5 g/cm³ can be achieved with the WC, WDC and DWL compaction techniques [4].

For single compaction processes, the performance of the lubricant admixed with the steel powder mixes or sprayed on the die walls remains the key factor in the achievement of high density. Lubricant is required to reduce the friction at die walls and ensure a good transfer of the compaction force throughout the part, low ejection forces and good surface finish and minimize tool damage. The lubricity at the die wall is proportional to the level of internal lubricant added to the mix, except for the DWL process. On the other hand, lubricants have very low specific gravity compared to steel and occupied a significant proportion of volume in the green part. The level of lubricant has a strong effect on the theoretical pore free density (density achieved if all porosities are eliminated) and the maximum density that can be practically achieved during compaction. The equation is thus very simple: the better the lubricant, the lower the quantity needed and the higher the density achievable. It is the reason why a significant amount of efforts have been focused over the last decades to develop and introduce new families of lubricant with improved lubricating properties.

This paper describes the compaction and ejection properties of FLOMET WP™, a proprietary lubricant/binder system specifically developed and designed for the warm compaction process and new ***High Density*** (HD) lubricants under development, specifically designed for cold and warm-die compaction. The compaction characteristics of FLOMET WP™ used in the serial production of a structural PM part are then presented. Results of a compacting trial performed with one of the experimental HD lubricant showing the best potential are also presented.

1 PERFORMANCE OF HIGH DENSITY LUBRICATING SYSTEMS

1.1 Material Description

Mixes were prepared according to the FD-2000 formulation from MPIF standard 35. ATOMET DB46, a diffusion-alloyed steel powder containing 0.5%Mo -1.75%Ni - 1.5%Cu produced by Rio Tinto Metal Powders, was admixed with 0.3% natural graphite and various lubricant/binder systems. Table 1 gives the description of mixes prepared. The term ATOMET refers to the conventional dry mixing technique while FLOMET WP ™ refers to the binder-treated process specifically designed for Warm Compaction mixes. Mix WP is currently used for mass production of several parts by the warm compaction process by Porite Yangzhou. Four different versions of HD lubricant systems were tested in this study, all of them containing from 0.1 to 0.4% of a lubricant identified as Lube HD-C. The characteristics of this lubricant are described in references 5 and 6 (identified as Lube C in the paper). It was used alone in mix HD-1 and in combination with other lubricants for mixes HD-2 to HD-4. It is worth mentioning that the level of lubricants in mixes HD varied between 0.4 and 0.45%, which is significantly lower than the level of lubricant and binder used in mix WP. As a result, pore free density was notably higher for mixes HD-1 to HD-4.

Table 1. Description of Mixes Prepared for this Study

Mix Name	Admixing Type (1)	Lubricant system	Pore Free Dens., g/cm³
WP	***FLOMET WP ™***	***0.58% Lube + Binder WP***	***7.52***
HD-1	***ATOMET***	***0.40% Lube HD-C***	***7.61***
HD-2	***ATOMET***	***0.25% Lube HD-C + 0.20% Lube WP***	***7.58***
HD-3	***ATOMET***	***0.25% Lube HD-C + 0.20% EBS Wax***	***7.58***
HD-4	***ATOMET***	***0.30% Lube HD-A + 0.1% Lube HD-C***	***7.61***

(1)- ATOMET refers to conventional dry mixing method. FLOMET WP refers to the Binder-treatment admixing method specifically designed for Warm Compaction mixes.

1.2 Experimental Procedures

The compressibility curves were evaluated by pressing standard 6.35 mm thick transverse rupture strength (TRS) specimens on a lab hydraulic press at a temperature of ~75°C for the HD mixes and 130°C for WP mix.

The compaction and lubricating performances were evaluated in more details with an instrumented single action compacting press known as the Powder Testing Center (PTC). The PTC allows continuous recording of the moving punch displacement and pressures applied to the moving punch and transmitted to the stationary punch all along the compaction and ejection process, allowing determination of the key compaction characteristics such as the slide coefficient, the ejection forces and the compactability. These characteristics are described below.

The slide coefficient η, which characterizes the efficiency of transferring the compaction force throughout the part, can be defined by an empirical relation developed by Gasiorek and al. [7,8] for a single action press as follows:

$$\eta = \left(\frac{Pt}{Pa}\right)^{\left[\frac{4F}{SH}\right]}$$

where *Pa* is the pressure applied to the compacting punch,
Pt the pressure transmitted to the stationary punch,
F the cross-section area,
S the cross-section perimeter,
H the height.

The factor *4F/SH* represents the compact aspect ratio or compact geometry factor. For a cylindrical compact, the factor *4F/SH* is equal to *D/H* where *D* is the diameter of the compact. η can vary between 0 and 1, representing an infinite friction and no friction respectively. The higher the η, the lower the friction loss and the better the lubrication and densification uniformity. For a given in-die density and die material, the slide coefficient is a very good parameter to evaluate the lubrication behavior of powder mixes containing different types of lubricants [9].

The compactability, which is defined as the intrinsic ability of a powder to be densified in the absence of friction at die walls [10], can be evaluated by the relation between the green density and the average pressure seen by the compact called Net Pressure (P_{NET}). P_{NET} is given by the following equation for a cylindrical compact,

$$P_{NET} = Pa * \eta^{\left(\frac{H}{2D}\right)} = (Pa * Pt)^{1/2}$$

The stripping and sliding ejection shearing stresses were obtained by dividing respectively the maximum force required to initiate the ejection cycle and the average force necessary to maintain the ejection movement over a certain travelling distance by the surface area of the compact in contact with the die wall, corresponding to πDH for a cylindrical specimens.

Cylindrical specimens 10 mm tall were pressed in a tungsten carbide die (WC) having a diameter of 9.525 mm at a compacting rate of 1 mm/sec. The aspect ratio of these cylindrical specimens pressed in the PTC is 3 times higher than that of standard 6.35 mm thick TRS bars. Those compacts are therefore more representative of parts commonly produced in a production scale. The compaction tests were run at 70°C and 830 MPa.

1.3 Compaction behavior

The compressibility curves as obtained by pressing standard TRS specimens at 130°C for mix WP and 75°C for the other materials are given in Figure 1. A mix with the same formulation and 0.7% EBS wax, identified as Ref, was also tested. Mixes WP and Ref showed quite similar compaction behavior except that density was between 0.05 to 0.07 g/cm³ higher for Mix WP. The higher density obtained with mix WP is linked to the higher compacting temperature and the lower amount of lubricant versus the Reference mix. In both cases, relative density of about 97.5 and 98% was reached at 690 and 830 MPa, respectively, which corresponds to the practical limit for these materials.

The compressibility behavior of HD mixes was quite different than that of WP and Ref mixes. Indeed, at 550 MPa, green density was similar to the reference mix but about 0.07 g/cm³ lower than the WP mix. However, the gain in density obtained by increasing the compacting pressure was more important than that achieved with the two other mixes. Green density reached values varying between 7.42 and 7.47 g/cm³ at 830 MPa for HD mixes, which is between 0.05 and 0.10 g/cm³ higher than density achieved with FLOMET WP. Best results were achieved with mix HD-1 and HD-4. The difference in behavior is due to the lower amount of lubricant in HD mixes and the fact that a certain amount of pressure must be applied to activate the lubricant.

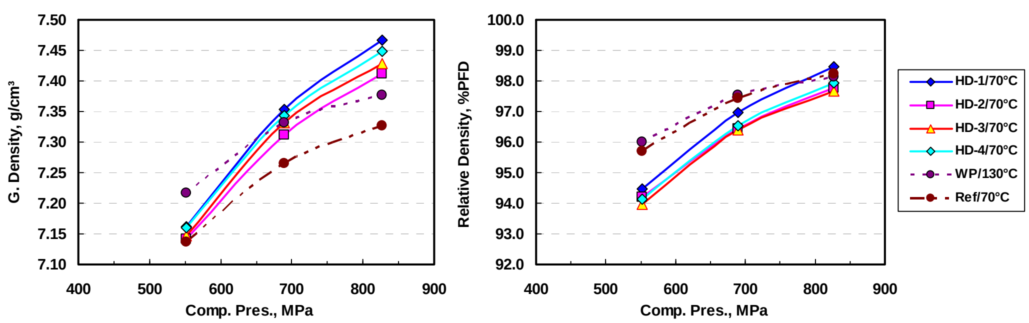

Figure 1. Compressibility curves obtained with TRS specimens.

Results of tests realized with the PTC in a WC die at 70°C are summarized in Table 2. Because of the limitation in the maximum compacting temperature that can be used with the WC die, mix WP was not tested at ~ 130°C. The objectives of these tests were mainly to compare the lubricity behavior of each lubricant system during compaction and ejection.

Table 2. Results of tests carried out with the PTC (Compaction done in a WC Die at 830 MPa and 70°C).

Mix	Dens. In-Die, g/cm³	Green Dens. g/cm³	%PFD	Slide Coef	Net Pres., MPa	Stripping, MPa	Sliding, MPa
HD-1	*7.399*	*7.344*	*96.6*	*0.789*	*741*	*17.8*	*13.2*
HD-2	*7.398*	*7.342*	*96.8*	*0.778*	*739*	*17.6*	*13.0*
HD-3	*7.402*	*7.353*	*97.0*	*0.752*	*722*	*21.4*	*14.8*
HD-4	*7.416*	*7.382*	*97.1*	*0.721*	*712*	*24.2*	*15.7*
WP	*7.341*	*7.290*	*97.0*	*0.739*	*719*	*22.6*	*16.3*

Green densities varying between 7.34 and 7.38 g/cm³ were achieved with all HD mixes, best result being obtained with HD-4. It is also interesting to note that this result was achieved with the lowest net pressure amongst all the mixes tested, clearly indicating that the intrinsic compactability of this mix is superior to the others. This is confirmed in Figure 2 that shows the green density as a function of the Net pressure. It should be noted that mix WP gave the lowest density amongst the material tested. This result is partly explained by the fact that the compacting temperature used was not the optimum one for that lubricant system.

Despite the very good densification characteristics of mix HD-4, it did not provide as good lubrication performance as the other lube systems. In fact, mixes HD-1 and HD-2 gave the highest slide coefficient and the lowest ejection forces. It should be noted that all mixes gave slide coefficient above 0.7, which is considered

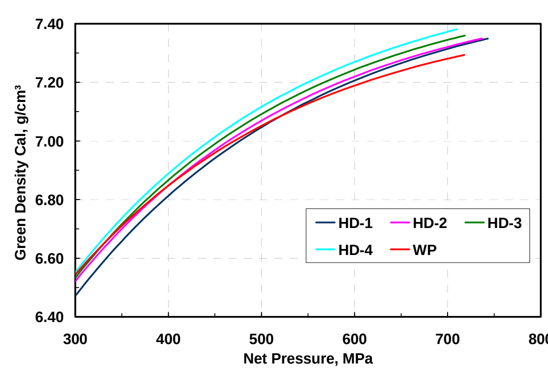

Figure 2. Green density as a function of Net pressure (WC Die, 830 MPa, 70°C).

as a good lubrication performance. Figure 3 illustrates the variation of slide coefficient as a function of the relative density. The typical curve obtained with 0.75% EBS wax (FC0205 formulation) is given for comparison. It is seen for all lubricant systems that the slide coefficient remained quite stable during compaction with a slight increase when density exceeds about 93% PFD. The increase in slide coefficient as density approaches the maximum density achievable is likely due to a higher amount of lubricant moving to the die walls. The increase is much more spectacular with EBS wax, especially when relative density approaches 98%. This is believed to be linked to the high level of EBS wax in this mix (0.75%), which is between 30 and 88% higher than in the other mixes. For the entire density range, mixes HD-1 and HD-2 showed better lubricating properties than the mix with EBS wax even with lower lubricant content. The behavior of Mix WP was also very similar to the mix with EBS wax, again with a lower lubricant content (0.58% vs 0.75%).

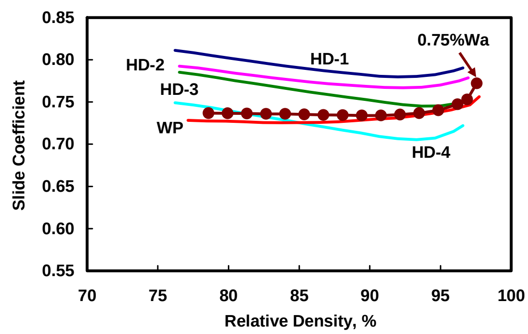

Figure 3. Relation between the slide coefficient and the relative In-Die density (WC Die, 830 MPa, 70°C).

Figure 4 shows the stripping and sliding forces measured during ejection. Typical results obtained with a FC0205 mixes containing 0.75% EBS wax and Kenolube are also shown. Mixes with EBS wax and Kenolube were pressed at 690 MPa instead of 830 MPa for the other mixes. It is seen that the ejection behavior of mixes HD-1 and HD-2 were quite comparable to that of mixes with EBS wax and Kenolube. Mix HD-4 gave the worst ejection performance followed closely by mix WP. However, these values remain lower compared to the ejection forces normally measured with a HSS die as shown in Figure 5. In the case of mix HD-4, it contains only 0.4% total lubricant with 0.1% Lube HD-C. The level of lube HD-C is the lowest amongst all the mixes tested. It is quite clear from results in Table 2 that lube HD-C provides very good lubrication performance. Increasing the level of HD-C and slightly reducing the level of HD-A should allow to significantly improved ejection performance.

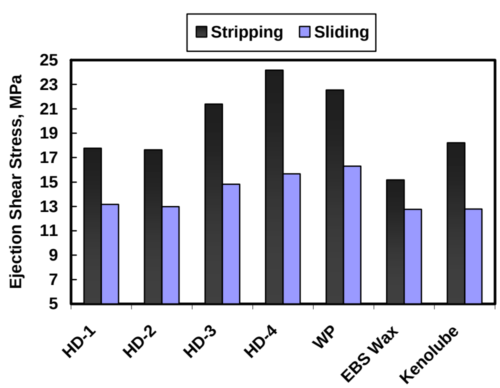

Figure 4. Ejection forces measured in a WC Die (830 MPa, 70°C).

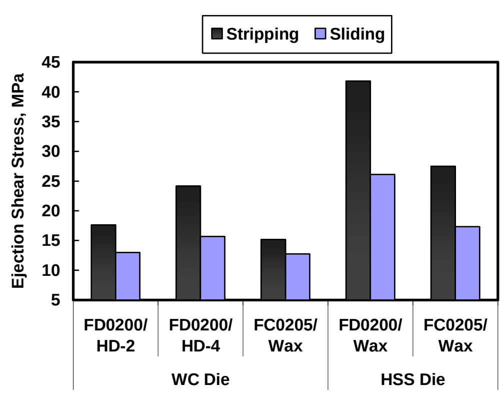

Figure 5. Ejection forces in different die materials at 70°C and 830 MPa (690 MPa for FC0205).

2 PRODUCTION TRIALS

2.1 FLOMET WP Warm Compaction

2.1.1 Experimental Procedure

Mix WP described in Table 1, a FLOMET WP™ FD-0200 formulation already used for mass production of several parts at Porite Yangzhou, was used for the validation of the FLOMET WP™ and warm pressing technologies. Cam gears as illustrated in Figure 6 and used for the power hand tools were pressed with a 60 metric tons mechanical press at a stroke rate of 12 parts/min. The powder mix and the die were heated to 70 and 90°C respectively. The green parts were compacted to a density of 7.2 g/cm^3. About 100 pieces were produced for this test run. The compacting tonnage, part weight, dimension and sintered hardness were measured to validate the consistency of the WP powder mix.

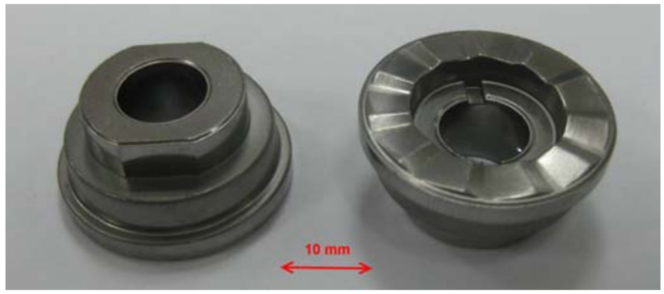

Figure 6. Picture of the cam gear produced with a FD-0200 FLOMET WP™ premix (Mix WP).

2.1.2 Results

Results of tests are summarized in Table 3. The premix WP performed well in terms of consistency of compacting pressure, part weight, green and sintered dimensions and hardness, which were well within the lower and upper limits. These results are typical of behavior of that material. The variation in weight corresponds to ± 0.49%, which clearly indicates that the flowability of the FLOMET WP ™ premix is good when heated at a temperature of ~ 70°C. Very small variation in OD after ejection was obtained, also indicating a very consistent springback. It is worth mentioning that the same consistency was maintained after sintering.

The surface finish of parts was excellent, which indicates that the lubrication of the WP Lube was more than adequate. The green density targeted for this test was relatively low for this mix, which can potentially reach density of around 7.35 g/cm³ (at 98% PFD). It is planned to validate this lubricant system under more severe conditions against the new experimental lubricants.

Table 3. Summary of the compacting, green and sintered properties of the FD-0200 FLOMET WP™ mix.

	Specification	Min.	Max.	Range	Sigma
Tonnage, Tons	***50 / 53***	***50.8***	***52.5***	***1.7***	***0.33456***
Weight, g	***46.7 / 47.3***	***46.79***	***47.25***	***0.46***	***0.09778***
Height, mm	***15.4 / 15.55***	***15.447***	***15.507***	***0.06***	***0.01277***
Outside Diameter green, mm	***32.9 / 33.1***	***33.00***	***33.02***	***0.02***	***0.00577***
Outside Diameter Sintered, mm	***32.9 / 33.1***	***33.04***	***33.06***	***0.02***	***0.00535***
Hardness, HRB	***80 / 90***	***82***	***85***	***3***	***0.69962***

2.2 New HD Lubricating System

2.2.1 Experimental Procedure

Three HD lubricant systems were of particular interests: HD-1, HD-2 and HD-4. HD-1 and HD-2 gave the best lubrication behavior. However, HD-1 was eliminated because of its higher cost compared to the others and because it did not offer real benefit compared to the HD-2 system. The HD-4 system gave the best densification behavior. However, as shown in Table 4, mix HD-2 had a much better flow rate than mix HD-4. Also, mixes HD-2 and WP had very similar flow rate and apparent density. System HD-2 was then selected for the validation under real production conditions at Porite Yangzhou. It is worth mentioning that the HD-2 lubricant system is composed of lubricants HD-C which provides very good lubrication and compaction behavior at a moderate temperature and lube WP that is currently used in FLOMET WP™ technology.

Table 4. Physical properties for Mixes HD-2, HD-4 and WP.

	HD-2	HD-4	WP
App. Dens. g/cm³	***3.37***	***2.99***	***3.36***
Hall Flow rate, s/50 g	***24.8***	***35.4***	***27.6***

An eccentric inner gear as shown in Figure 7 was selected for this production trial. This part is currently produced with the warm compaction technology at 110°C using a FD-0200 material named FD0200-Ref for the reminder of the text. This material contains about 0.6% lubricant. A larger batch size mix with the same formulation as HD-2

(Table 1) was produced. Mix is named FD0200/HD-2 to avoid confusion. Parts were pressed with a 60 metric Tons mechanical press at a rate of 12 parts/min and to a green density of 7.25g/cm^3. Only the die was heated to a temperature of ~60°C. Friction generated during compaction was sufficient to rise the part temperature to about 70°C. For the Warm Pressed mix (FD0200/WP), the die and powder were both heated at 110°C. For each test run, 100 parts were collected to validate the consistency as well as the appearance of parts.

2.2.2 Results

Results of tests carried with the inner gear are reported in Table 6. It should be noted that green density met the specification for both materials. The tonnage required to reach the desired density, weight and height was slightly lower with the HD lube system even if compacting temperature was significantly lower. The consistency of mix with the HD-2 lubricant was also very good and comparable to that obtained with the current warm pressing mix used in production. The part weight variation was ± 0.33%, which is very good. The surface finish of parts was also very similar for both materials, indicating that 0.45% lubricant HD-2 was sufficient for this part. However, longer pressing runs should be done to confirm the results on a longer period. Results are nevertheless very promising and it is planned to pursue the tests.

Figure 7. Picture of the eccentric inner gear used for the HD lubricants Test.

Table 6. Results of tests carried out with FD0200 mix with a new experimental lubricant system HD-2.

Mix	Items	Tolerance	Min.	Max.	Range	Sigma
FD0200/WP	Tonnage, Tons	-	45.7	46.7	1.0	0.24288
	Weight, g	0.4	29.733	29.95	0.217	0.05060
	Height- green, mm	0.2	10.15	10.2	0.05	0.01223
FD0200/HD-2	Tonnage, Tons	-	44.2	45.1	0.9	0.18062
	Weight, g	0.4	29.892	30.092	0.200	0.03703
	Height- green, mm	0.2	10.24	10.29	0.05	0.00891

2.2.3 Next steps

The objective of this first validation trial was to confirm the potential of new experimental lubricant systems under real production conditions. It is well known in the industry that the behavior of organic lubricants may vary significantly between laboratory and production conditions. Several differences exist but the compaction rate is certainly the factor that may contribute the most to change the behavior of the lubricant. So it was very important to confirm this at the start. The very encouraging results obtained now open the road for the next series of tests, which is the determination of the practical limit in term of densification and lubrication of these new lubricating systems. It is planned to use fully instrumented mechanical presses capable of recording ejection forces and press parts with different size and shape. Longer runs with pre-production scale batches should also be realized to validate the consistency of the green and lubrication properties. At the same time, research and development continue to find and develop more efficient and cost effective lubricants.

CONCLUSIONS

The properties of two lubricant systems for high density applications, the FLOMET WP™ lube system specifically designed for the warm compaction technology and currently used for the mass production of several parts, and new experimental HD lubricants designed for the cold compaction and/or warm-die compaction were described. In particular, the compaction and ejection characteristics as evaluated with an instrumented laboratory press were presented in the first section while results of compaction trials performed with parts currently in production with the warm compaction technology at Porite Yangzhou were presented in the second section. The following conclusions can be drawn from these tests.

- The FLOMET WP ™ lube system gave higher green density and very similar lubricating properties during compaction and ejection compared to a mix containing EBS wax even if its concentration was about 30% lower.
- Four different HD lubricant systems were evaluated at levels varying from 0.4 to 0.45%. Green densities higher than 7.40 g/cm³ were achieved with all the HD lube systems at 800 MPa or higher, which represent a significant gain compared to a reference mix with EBS wax and the FLOMET WP™ lube system.
- The lubricating properties of HD lube systems (0.4 to, 0.45%) were superior to that of FLOMET WP™ (0.58%) and EBS wax (0.75%) even if their concentration in the mix was significantly lower.
- Despite the very good lubricating properties of HD lubricants, high compacting pressure must be applied to obtain high green density approaching 97-98% of the maximum theoretical density.
- Compaction trials performed with FLOMET WP ™ and one of the HD lubricant systems tested in this paper confirmed the excellent part-to-part consistency and the very good compaction and lubricating properties of these lubricant systems.
- Further tests are planned to validate the real potential of HD lubricants in term of density and lubricity, especially in the case of more challenging parts.

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