Development of Advanced Lubricants for New PM Applications

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Abstract

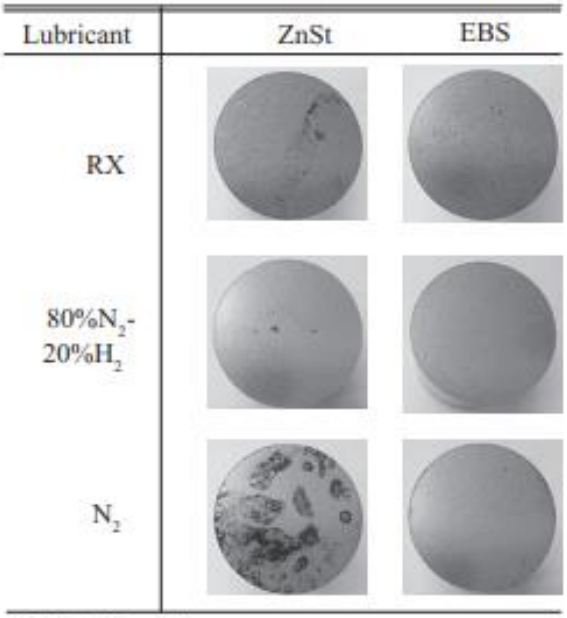
As the powder metallurgy (PM) industry continues to evolve, the need for highly-effective, clean-burning, environmentally-friendly lubricants becomes more evident. PM parts are becoming more complex and require higher strength with less weight, necessitating a push for components with higher green and sintered density. While many lubricants have been developed over the years, newer advanced lubricants have been conceived that are both more lubricious and cleaner burning than their previous counterparts, opening the compaction operating window. Superior lubricants can be used at lower additions to achieve higher density without the need for heated tooling into a tight temperature range. They can also allow for easier de-lubrication during sintering, which lessens their environmental impact, results in cleaner parts, and improves magnetic performance when used in soft magnetic applications.

Introduction

Powder Metallurgy (PM) lubricants and binders, typically consisting of metallic stearates, amide waxes, or a combination thereof, are commonly utilized to allow for proper compaction and ejection of parts in press-and-sinter applications [1-3]. These lubricants have historically been used at additions between 0.5-1.5 wt.% and while non-metallic additions to powder premixes are undesirable, admixed organic lubricants have proven to be necessary in the production of PM parts. Alternative solutions such as die wall spray have never proven to be a robust production alternative for most parts producers [4-5].

The ideal PM lubricant solution should offer optimal lubricity without negatively affecting the blending of the powder premix, the compaction process, sintering of the compacted parts, or mechanical properties of the sintered parts. Desired lubricant properties include high apparent density and good flow of the powder premix, low ejection forces, minimal scoring on parts and compaction tooling, high green strength and green density of compaction parts, clean burnout with low environmental or safety implications, and excellent mechanical strength of sintered parts. Finding new lubricants to meet all the above criteria can be a challenge.

As mentioned previously, metallic stearates (such as zinc and lithium stearate) have been used in the PM industry for many years. While these lubricants blend well in premixes and offer many benefits, they do not burn out as cleanly as other alternatives, as shown in Figure 1 [6-8]. This figure shows a comparison of sintered parts produced using a common metallic stearate and amide wax lubricant, sintered in various atmospheres. This type of staining, in addition to other cleanliness and environmental concerns, makes the use of a metallic stearate lubricant an unattractive choice for today's industry.



RX: Endothermic gas

Figure 1: Comparison of sintered surface finish between common metallic stearate and amide wax in various sintering atmospheres [6]

Amide waxes, such as ethylene bis stearamide (EBS), shown in the figure above, offer a much cleaner-burning alternative, making them the most popular choice for conventional powder metallurgy parts. And while amide waxes are effective for most applications, automotive industry demands over the past several years are requiring greater mechanical properties, most commonly and cost-effectively achieved through an increase in green and sintered part density. To increase part density, beyond simply increasing compaction pressure, it is essential to reduce lubricant content due to the low density of the lubricant itself. Therefore, new lubricants have been developed with equivalent or superior part ejection characteristics, even at lower premix lubricant additions in the range of 0.25-0.60 wt% [9-12]. These newly developed lubricants are also effective over a wider temperature range, allowing them to be used in conjunction with heated compaction tooling to drive density levels even higher [13-15]. Figure 2 below shows the effect of increasing die temperature on green density for an advanced premixed lubricant at 0.25 wt.% addition.

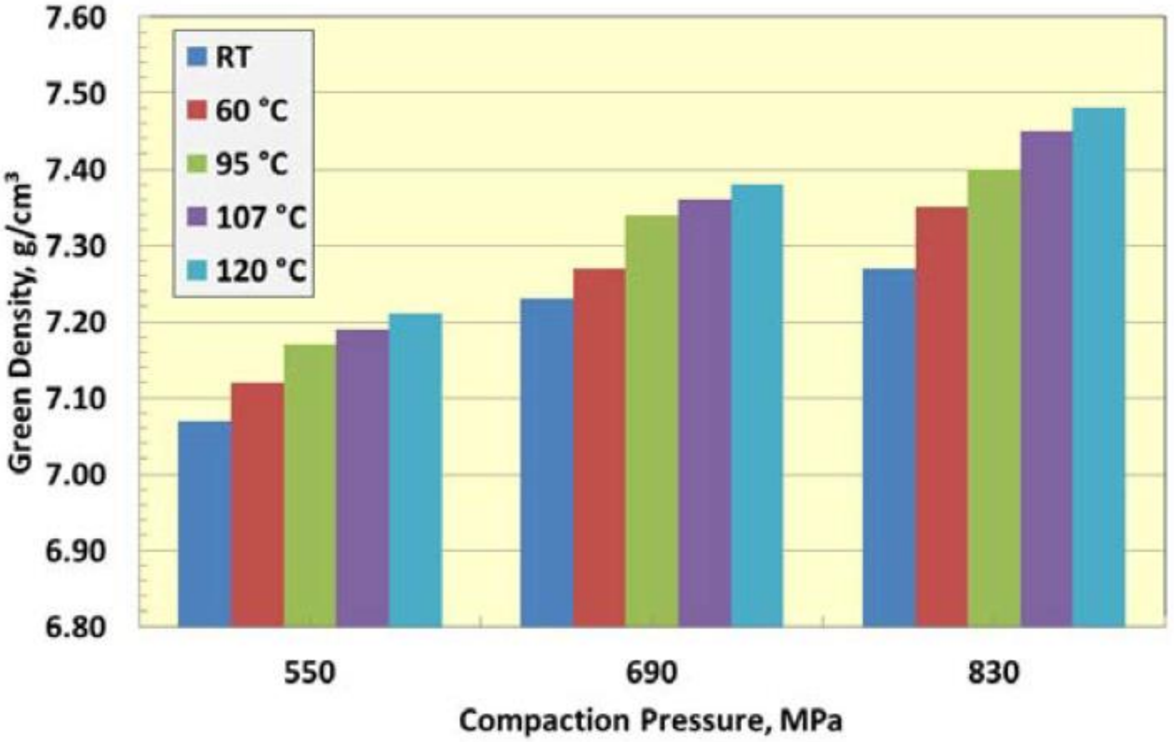


Figure 2: Effect of die temperature on compacted green density at three different compaction pressures with an addition of 0.25 wt.% advanced lubricant [14]

As market demands on final part properties continue to increase, even more advanced lubricant options will need to be developed. This work outlines the development and testing of AncorLube LV, an admixed premix lubricant superior to any other known admixed lubricant on the market today. It has been rigorously tested through all stages of the production process and benchmarked against other commonly-used PM lubricant options. The lubricant is most attractive due to its lubricity even at low lubricant additions, wide temperature range of operation, and clean burn out.

Experimental Procedure

After development and optimization of the AncorLube LV chemistry and particle size distribution for maximum lubricity, further experiments were completed in several stages to ensure ideal performance in all areas of the standard PM production process. First, the ejection forces were measured on FC-0205 premixes with 0.75 wt.% admixed lubricant, comparing AncorLube LV to commonly-used alternatives, EBS and Kenolube. This was completed on bushing samples with an OD of 14.3 mm, an ID of 9.0 mm, and an M/Q ratio of 10 using a compaction pressure of 700 MPa and a room temperature die.

Once complete, the green density and ejection characteristics were then repeated on rectangular bars measuring 32 x 12.7 x 12.7 mm dimensions according to MPIF Standard 15 [16], where the initial ejection pressure (strip) and pressure applied as the bar is exiting the die (slide) were measured over time. Figure 3 shows a schematic of a standard pressure vs. time graph, including the points at which strip and slide pressures were recorded [3]. This round of testing was completed on F-0000 compositions, using the same three lubricants as before and two different lubricant additions (0.4 and 0.6 wt.%). Testing was completed using a compaction pressure of 760 MPa and a die temperature of 70 °C.

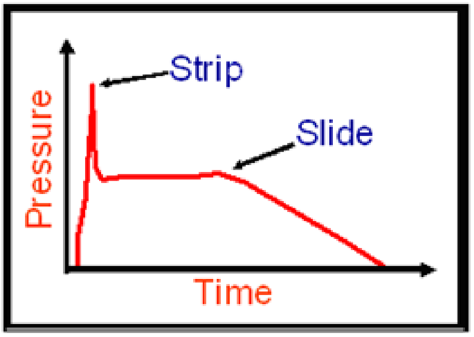


Figure 3: Schematic of ejection pressure measurements [3]

In order to observe the compaction consistency of a premix made with the AncorLube LV, a larger volume FC-0205 premix was produced with 0.6 wt.% lubricant addition. From this premix, VVT stator components were produced using a press rate of 10 strokes per minute and the weight of every fifth part was measured up to 1,400 parts. Parts were compacted to a green density of 6.9 g/cm^3 at a die temperature of 65 °C.

Finally, the burn-out characteristics of each of the lubricants was characterized in two different ways. First, FC-0208 premixes were produced with 0.6% lubricant additions of AncorLube LV, Kenolube, and EBS. Using these premixes, slugs with a nominal diameter of 38 mm and nominal height of 25 mm were compacted to a green density of 7.0 g/cm^3 and sintered in an Abbott belt furnace at 1120 °C in an atmosphere of 90 vol.% N_2 and 10 vol.% H_2 for approximately 20 minutes at temperature. After the surface condition of the sintered slugs was documented, the weight loss of ~50 mg each lubricant was measured using thermogravimetric analysis (TGA) on a Netzsch STA 449 C. The total weight loss was measured in nitrogen over a temperature range from room temperature (~20 °C) to 800° C in order to simulate lubricant burn-out during sintering.

Results and Discussion

The key aspect of any PM lubricant is its ability to aid in compaction and ejection of parts. Therefore, during lubricant development, cylindrical parts with high surface area and M/Q ratio (lateral area divided by compacted area) of 10 were utilized to replicate difficult part compaction and ejection. As shown in Figure 4, an FC-0205 premix containing 0.75 wt.% EBS compacted using this geometry required an ejection force of 175 MPa. EBS represents a good baseline value as the most common lubricant in the PM industry, widely used due to low cost and clean burn-out. Kenolube, another highly-utilized lubricant in the PM industry, was found to reduce the necessary ejection forces under the same conditions by ~36%. Meanwhile, the AncorLube LV lubricant reduced ejection forces by 55% from the EBS baseline when utilizing the same premix composition, lubricant content, and die geometry.

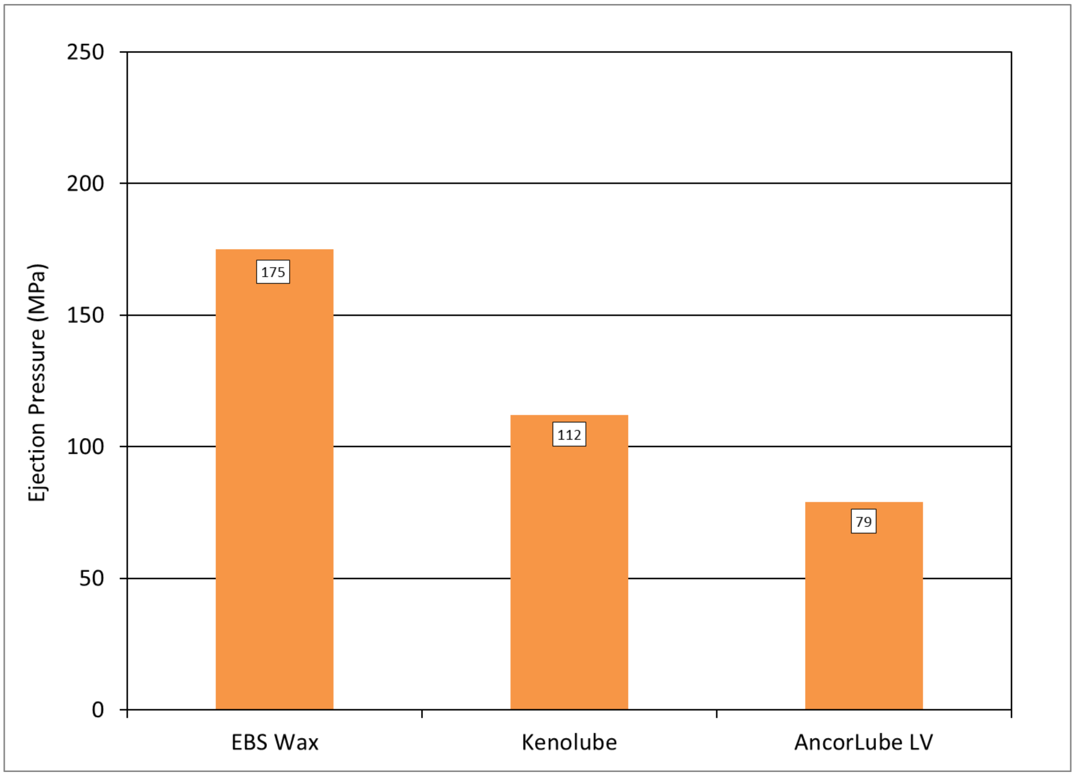


Figure 4: Ejection pressure of three lubricants (0.75 wt.% addition) in FC-0205 premixes

To further analyze and compare lubricity of the various materials, standard F-0000 premixes were made with 0.6 wt.% and 0.4 wt.% admixed lubricant. A standard MPIF TRS bar geometry was used, compacted to a height of approximately 12.7 mm at a compaction pressure of 760 Mpa and a die temperature of 70 °C. As shown in Figure 5 with 0.6 wt.% lubricant addition, the EBS samples had the highest ejection forces, despite bars achieving the lowest green density of the group. This is not surprising considering knowledge of EBS and the previous results. The Kenolube premix had lower, improved ejection and slightly higher green density, while the AncorLube LV showed superior compressibility and ejection behavior to both alternatives.

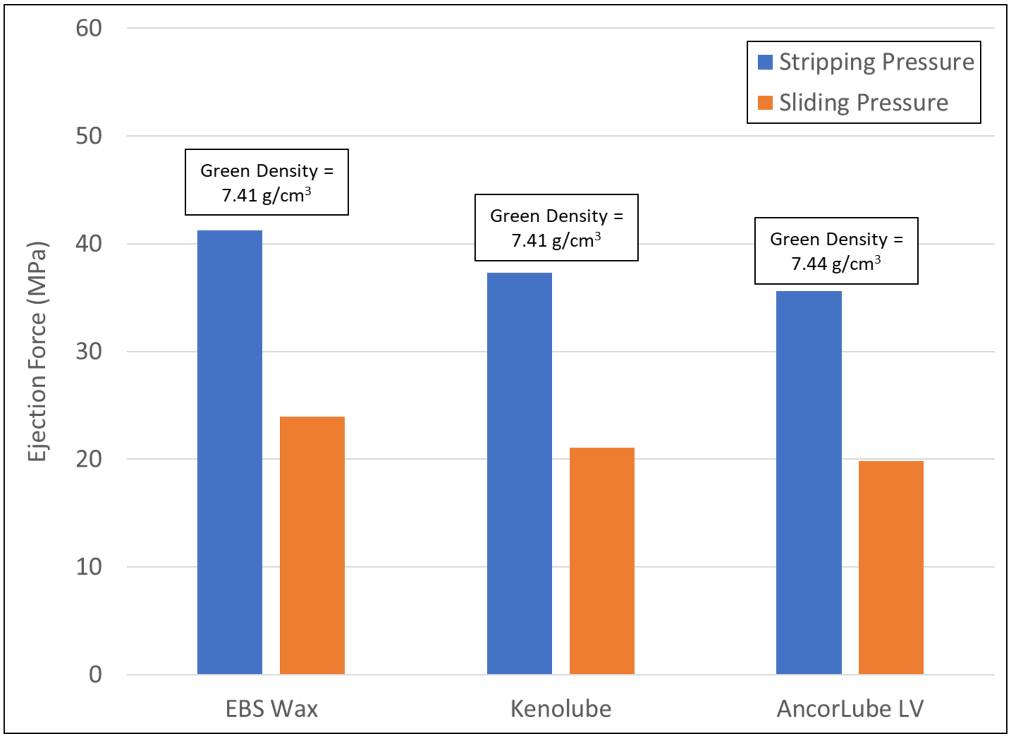


Figure 5: Ejection characteristics for rectangular samples made from F-0000 premixes with 0.6 wt.% lubricant compacted at 760 MPa at 70 °C

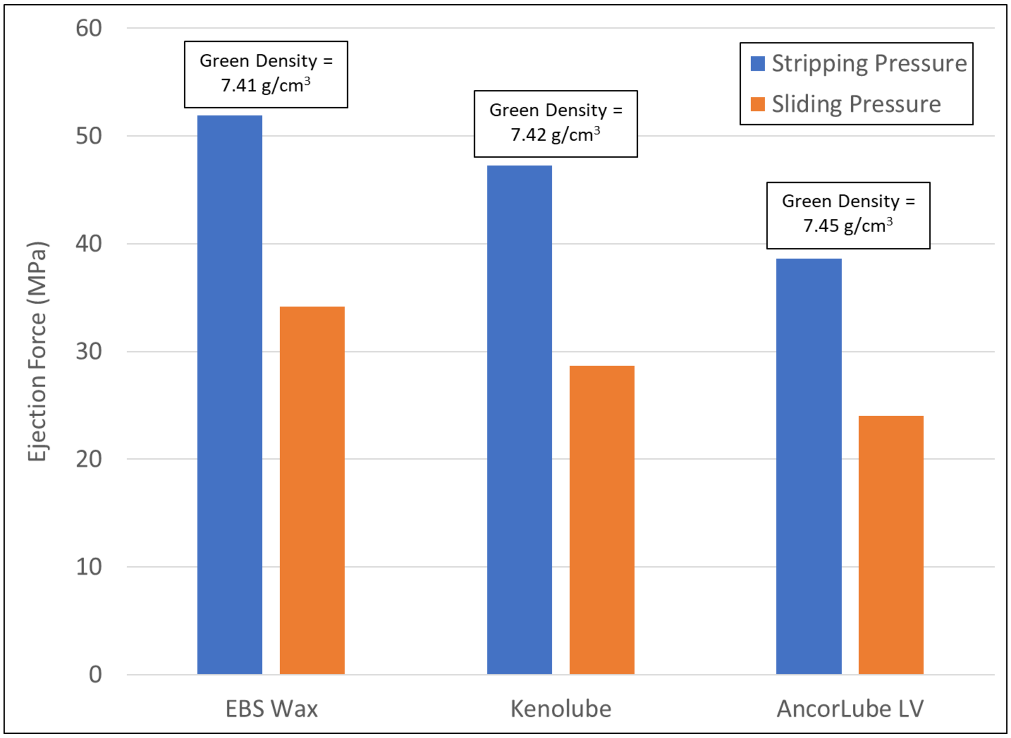


Figure 6: Ejection characteristics for rectangular samples made from F-0000 premixes with 0.4 wt.% lubricant compacted at 760 MPa at 70 °C

When comparing Figure 5 to Figure 4, it is surprising there is not a larger difference between the advanced lubricants and the EBS wax when tested at 0.6 wt.% addition. This is likely due to the much lower M/Q ratio with the TRS bar geometry (estimated at ~1.5), making this a much "easier" part to compact. The differences between the various lubricants became far more apparent in Figure 6 when the lubricant content was reduced to 0.4 wt.%. With less lubricant present, the limitations of the various admixed lubricants become clear. The EBS and Kenolube both see an increase of greater than 20% in their needed stripping pressure and an increase greater than 25% in their sliding pressure when reduced to 0.4 wt.% addition. Meanwhile, the AncorLube LV is less affected due to its extremely high lubricity. Even with a reduced lubricant content, only an 8% increase in stripping pressure and 18% increase in sliding pressure was observed with this lubricant.

This result is further confirmed when looking at the surface of the actual compacted and ejected bars TRS bars, as shown in Figures 7 and 8. Here, the longest side of each bar is displayed, with the vertical scratches visible parallel to the compaction direction. As expected, increasing overall lubricant content and moving towards more advanced lubricants in this study showed a marked improvement in the surface condition of the samples following ejection. The heaviest scoring was observed when the EBS was utilized at 0.4% lubricant addition.

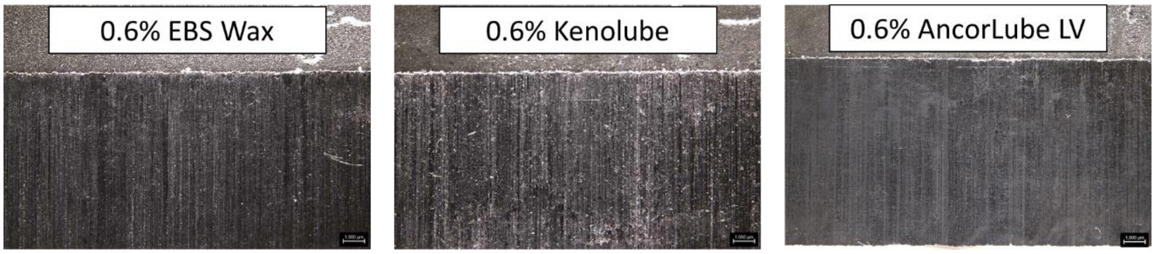


Figure 7: Stereoscope images of rectangular sample surfaces made from F-0000 premixes with 0.6 wt.% lubricant compacted at 760 MPa at 70 °C

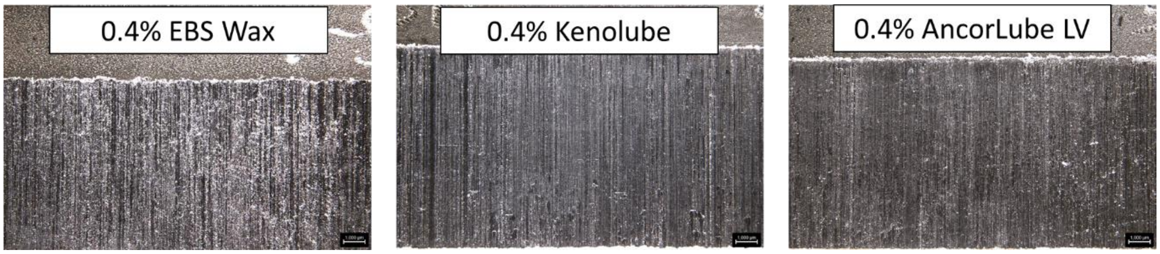


Figure 8: Stereoscope images of rectangular sample surfaces made from F-0000 premixes with 0.4 wt.% lubricant compacted at 760 MPa at 70 °C

With lubricating properties of the advanced lubricant confirmed on a lab-scale, an effort was made to complete a larger-scale compaction study to ensure compatibility with production-sized compacting presses along with process stability over a long period of time. To do this, a standard VVT die was utilized as well as a typical FC-0205 composition, commonly used in VVT parts. Using a 0.6 wt.% AncorLube LV addition and a rate of 10 strokes per minute, a compaction study was completed with part weight measured regularly up to 1,400 parts. Overall, good stability was observed with the advanced lubricant premix, with a total scatter of only 0.61 grams over the entire run. No issues with powder flow, die filling, or part ejection were observed during this study, and a reasonable part temperature was maintained.

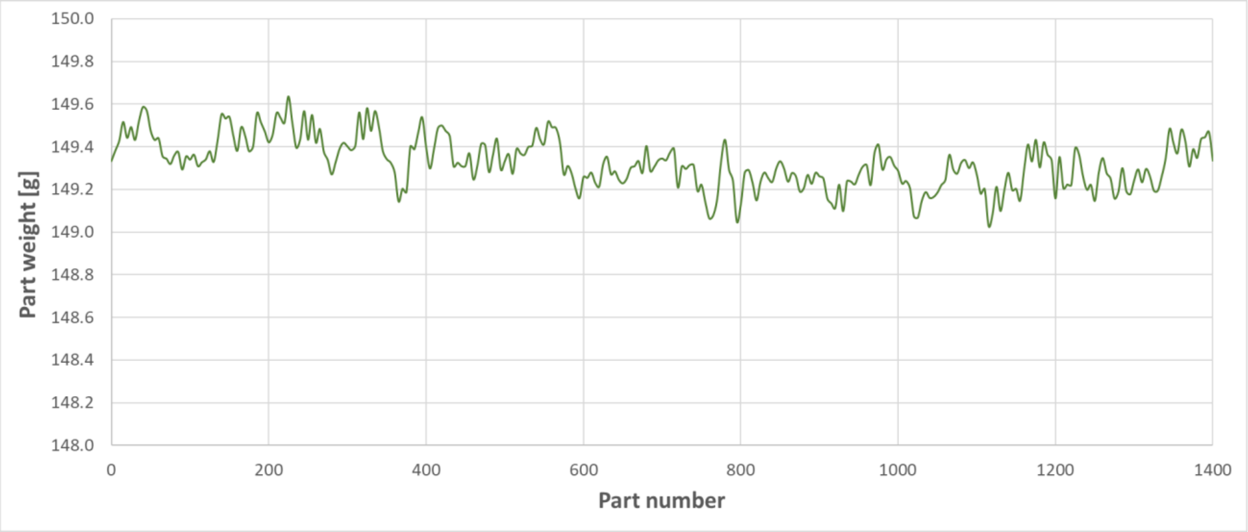


Figure 9: Compaction study of VVT stator components press at a rate of 10 strokes per minute with an FC-0205 composition with 0.6 wt.% admixed AncorLube LV

The final aspect studied with the various lubricants was their respective burn-off behavior during sintering. Due to the fact that the Kenolube is the only one of the three that contains a metallic stearate component, it would be expected to be the most likely of the three to present staining on the final parts after sintering. The experimental results support this finding, as shown in Figure 10. The slugs produced using the EBS and AncorLube LV premixes had a very clean appearance with no visible sooting on the surface. In contrast, the sample with Kenolube was found to have a high degree of surface staining, likely due to the presence of metallic stearates in the lubricant.


EBS Wax Kenolube AncorLube LV

Figure 10: Surface condition of sintered slugs made from FC-0208 premix with 0.6% admixed lubricant

In order to verify this result, further burn-off testing was completed using TGA analysis to measure weight loss of each lubricant type over a temperature range from room temperature to ~800 °C to mimic burn-off that would occur in a standard sintering furnace. The results of the TGA analysis, shown in Figure 11, align well with expectations and previous results. The figure shows the percentage mass loss of pure lubricant when heated up to 800 °C. Most of the mass loss occurs between 300 and 500 °C, as the lubricants begin to break down and decompose. The EBS wax and AncorLube LV experience the steepest slope, showing the fastest breakdown, with most of the weight removal complete by 500 °C. Beyond 600 °C, only a negligible amount of lubricant remains (~1% or less).

The Kenolube and zinc stearate experience a similar curve, but the burn-out does not happen as quickly and the mass remaining at completion is higher. Even at 800 °C, approximately 3% of the mass of the Kenolube remains and nearly 14% of the zinc stearate remains in the compact beyond the typical de-lubrication temperature range. This represents the mass of the metallic oxides that will not fully breakdown during a typical sintering process. Because Kenolube only contains a portion of metallic stearate, the mass percentage remaining after sintering is lower than in the zinc stearate lubricant, and therefore, the presence of staining is expected to be less extreme as well, though still present.

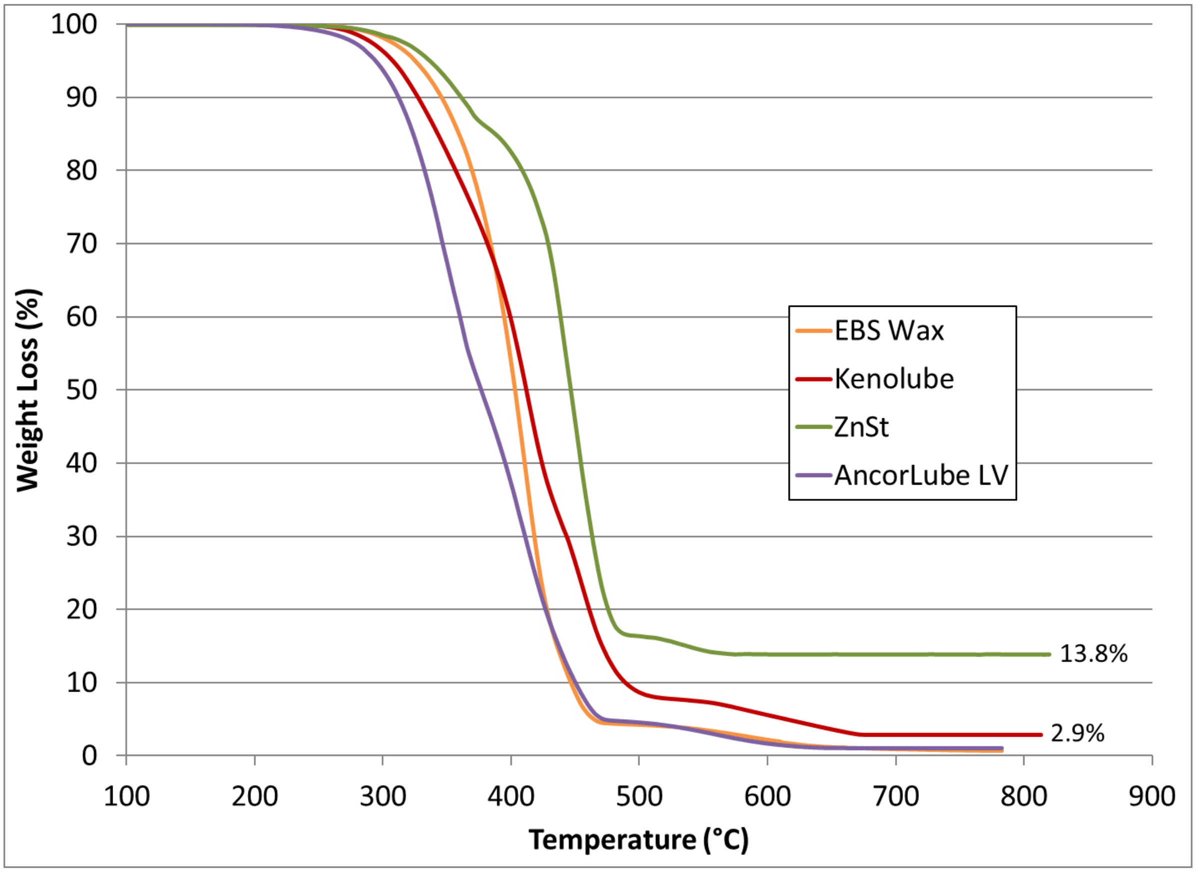


Figure 11: TGA analysis (weight loss %) of four different lubricants heated up to 800 °C

Conclusions

As a result of the experimental work performed during this study, the following observations were made:

- Advanced lubricant options, such as AncorLube LV, have been developed to function well at high M/Q ratios to effectively compact and eject parts with difficult geometries. The necessary ejection forces with such lubricants are significantly lower than with standard admixed lubricants such as EBS was and Kenolube.
- Advanced lubricants are effective at much lower lubricant levels than are typical for PM, allowing parts to be compacted to much higher green density without sacrificing surface finish or tool wear.
- The AncorLube LV has been proven effective when used over a range of premix compositions, lubricant additions, die temperatures, or part geometries.
- AncorLube LV can be used effectively in a production setting, allowing for good part-to-part consistency and speed of operation.
- Avoiding lubricants with metallic stearates can have a dramatic effect in reducing or eliminating surface sooting and staining in final sintered parts. This was evident both from the visual part examination and the TGA analysis of the lubricants themselves.

Acknowledgements

The authors want to thank Edwin Schnaider, Jason Gotta, Nicole Scheer, and Barry Diamond for all their contributions to this manuscript. Without their help, this degree and quality of analysis would not be possible.

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