

Development of a Lubricant System for Improved Performance of Premixes

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Abstract

Particulate lubricants are commonly admixed in powder metallurgy (PM) premixes to provide lubricity during the compaction process by decreasing interparticle friction, as well as decreasing the friction between the particles and the die. Friction reduction between the final compact and the die allows for easier part ejection, which leads to improved life of the tooling. In this study, four total admixed lubricants are tested to determine their effect on the properties of the premixed powder, green compacts, and final sintered parts. Two of the lubricants, EBS wax and Kenolube, are commonly used in the PM industry, while two other lubricants are experimental lubricants. All four lubricants are compared over a range of compaction pressures and at two different die temperatures.

Introduction

PM lubricants typically consist of metallic stearates, synthetic amide waxes, and composite hybrids of stearates and waxes admixed into premixes at 0.5 – 1.5 mass percent [1-3]. The main functions of these lubricants are to improve apparent density and flow characteristics of the powder, evenly distribute forces for uniform green density during compaction, and reduce necessary ejection forces to improve tooling life. Ideal lubricants should provide these advantages while maintaining high compressibility of the powder, providing excellent green strength achieved in the powder compacts, and burning out cleanly during sintering.

Following pressing, the lubricant is removed from a PM part during the sintering process. The de-lubrication zone of a sintering furnace removes the lubricant by heating the part using a controlled rate and atmosphere to temperatures between 500 and 600° C. The steps that lead to lubricant removal are melting, vaporization, vapor diffusion through the pore network to the part surface, and vapor removal by the gas flow within the furnace. Improper de-lubrication can lead to defects such as blistering, sooting, microporosity, and carbon segregation [1]. Because of the importance of de-lubrication, it is another important factor to consider when selecting a PM lubricant.

As applications for PM parts become more demanding, high density parts become desirable, while maintaining a low cost of production. High density has been achieved in PM using processes such as double press-double sinter (DPDS), forging, die wall lubrication, warm compaction, and warm die compaction [3-5]. While effective, DPDS, forging, and die wall lubrication techniques require alterations to the normal compaction process, often leading to increased processing steps and high associated costs. Warm compaction involves heating of the powder and the die, while warm die compaction requires heating of the die only [5].

Another route used to improve compressibility of PM premixes is through overall reduction of the lubricant using advanced binders and lubricants that can be used at 0.4% or below [5]. Lowering the total amount of lubricant present in a premix will result in an increase in the pore free density, the density that could be attained if no porosity remained after pressing. Due to the low specific gravity of lubricant particles compared to metallic powders, it is commonly accepted that each additional 0.1% of lubricant addition to a premix will reduce the pore free density by 0.05 g/cm³ [3,4].

While these advanced lubricant systems are currently available for specific applications, this paper will focus on lubricants that can be admixed into premixes and pressed at room temperature or using warm die compaction, which requires heating of the die only, normally to a temperature between 50 and 100° C. When using lubricants designed for warm die compaction, this approach can effectively lower the necessary ejection forces and increase the achievable green density, often resulting in improved properties overall [5]. All lubricants in this study will be added to premixes at 0.75% by mass, showing the advantages and disadvantages of each lubricant system in a common PM premix.

Experimental Procedure

Four regular premixes of similar composition were prepared for this study using commercially available Ancorsteel 85HP base iron powder from Hoeganaes Corporation. All four premixes contained 2.0 mass % Inco 123 Ni powder, 0.60 mass % Asbury type 3203H graphite, and 0.75 mass % lubricant content. The nominal compositions and identifications for each premix are shown in Table 1. Commonly used lubricants, EBS Wax and Kenolube, were used as a reference for the testing, as well as experimental lubricants, Ancorlube W and Ancorlube X from Hoeganaes.

Table 1: Nominal compositions and identifications of lubricants studied (mass %).

	EBS Wax	Kenolube	Ancorlube W	Ancorlube X
0.85% Mo Prealloyed Base Iron	Balance	Balance	Balance	Balance
Nickel	2.0	2.0	2.0	2.0
Graphite	0.60	0.60	0.60	0.60
Lubricant	0.75	0.75	0.75	0.75

All laboratory procedures were carried out in accordance with the appropriate MPIF standards [6]. The apparent density and flowability of each powder premix was tested using a Hall Apparatus according to MPIF Standards 3 and 4. The green density and ejection characteristics were observed on rectangular bars with 32 x 12.7 x 12.7 mm dimensions according to MPIF Standard 15. Using a hydraulic compaction press, the initial ejection pressure (strip) and pressure applied as the bar is exiting the die (slide) was measured over time. Figure 1 shows a schematic of a standard pressure vs. time graph, including the points at which strip and slide pressures are recorded [7]. Green density and ejection data were recorded at compaction pressures of 414, 552, 690, and 827 MPa (30, 40, 50, 60 tsi) at both room temperature and with a die heated to 75° C (170° F).

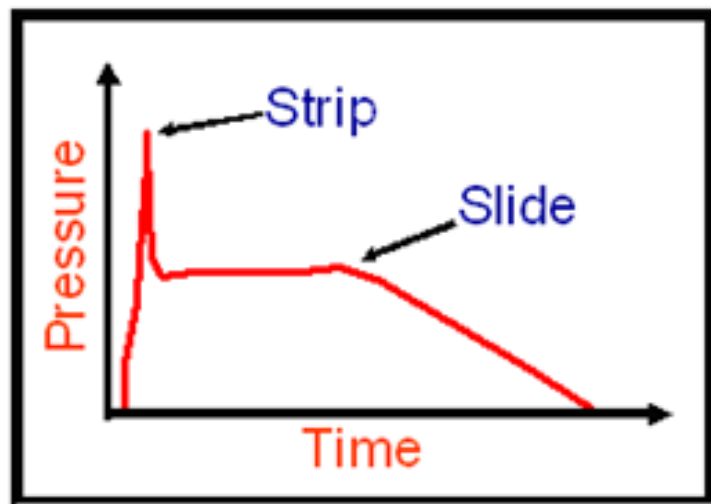
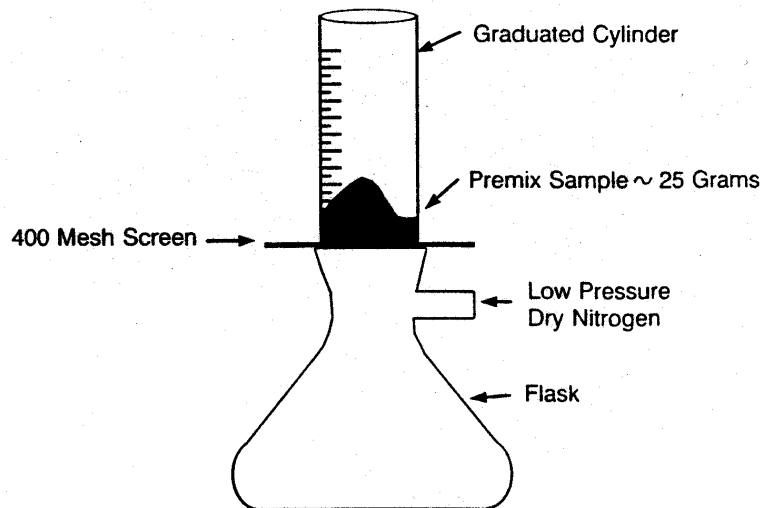


Figure 1: Schematic of ejection pressure measurements [7].

Each powder premix was also tested for dust resistance using an elutriation test. Using the set-up shown in the schematic in Figure 2, nitrogen was forced through a small characteristic sample of each premix. A chemical analysis was performed on powder before and after the elutriation in order to determine any change in the percentage of premix additions. In this study, the carbon content was measured before and after to determine dust resistance and bonding capacity of the graphite for the four tested premixes. The percent bonded graphite for each lubricant type is also a good indication of the dust resistance of other elements in the premix as well.



$$\text{Dust Resistance} = \frac{\text{Chemical Analysis After}}{\text{Chemical Analysis Before}} \times 100 = \%$$

Figure 2: Schematic of the elutriation set-up for testing dust resistance [7].

Mechanical properties were measured using transverse rupture (TR), dogbone tensile, and unnotched Charpy impact bars compacted over the same range of compaction pressures (414, 552, 690, and 827 MPa) and die temperatures (room temperature and 75° C) as described above. These samples were sintered at 1120° C for 25 minutes in a mixed atmosphere of 90 volume % nitrogen and 10 volume % hydrogen. Prior to mechanical testing, green and sintered density, apparent hardness, and dimensional change were determined on the TR samples using MPIF Standards 42, 43, and 44, respectively. The mechanical properties were evaluated on sets of five bars for each premix and density combination. TR strength was determined using MPIF Standard 41, tensile properties were found in accordance with MPIF Standard 10, and impact energy was measured using MPIF Standard 40. Sintered carbon values were also measured using a Leco 200 carbon-sulfur combustion gas analyzer with reference standards run before and after test samples.

The burn-out characteristics of each of the four lubricants was characterized in two ways. First, photographs were taken of 1.25 cm tall sintered bars immediately upon exiting the sintering furnace in order to document any discoloration or sooting present on the sintered bars. Second, weight loss of each lubricant was measured using thermogravimetric analysis (TGA) on a Netzsch STA 449 C. The total weight loss was measured in air over a temperature range from room temperature to 800° C in order to simulate lubricant burn-out during sintering.

Results

Powder Properties

The basic powder properties of the four premixes are shown in Table 2 below, labeled with the corresponding lubricant contained in the premix. As seen in the table, the different lubricants, added in the same amount to each premix, had an effect on overall premix behavior. The apparent density of the four premixes ranged from 3.03 to 3.27 g/cm³, with Kenolube giving a far higher value than any of the other three lubricants. The four premixes had flow values between 30 and 38 seconds for a 50 gram sample. EBS wax gave the slowest flow, while Kenolube was faster than both Ancorlube premixes. The lubricant added had little effect on the total carbon or sintered carbon in this study, but did have an effect on overall dust resistance of the premix. It was found that the EBS wax had the best dust resistance, maintaining over 60% of the added carbon, while both Kenolube and Ancorlube W dusted off approximately 60%. Ancorlube X had a middling dust resistance, losing approximately half the added carbon.

Table 2: Powder properties of the four premixes containing the lubricants of study.

Lubricant	Apparent Density (g/cm³)	Flow (s/50g)	Total Carbon (%)	Sintered Carbon (%)	Dust Resistance (% Carbon)
EBS Wax	3.10	38	1.22	0.56	62.3
Kenolube	3.27	30	1.19	0.56	43.9
Ancorlube W	3.03	34	1.18	0.57	40.0
Ancorlube X	3.10	34	1.21	0.55	52.4

Green Properties

The compressibility curves over a range of compaction pressures for bars pressed at room temperature and 75° C are shown in Figures 3 and 4, respectively. Kenolube was found to have the lowest overall compressibility at both die temperatures. Both Ancorlube W and Ancorlube X were found to be similar to EBS wax at room temperature, but had increased compressibility when pressing with a heated die. Figures 5 and 6 show the green strength values for each lubricant under the same pressing conditions. At room temperature, Ancorlube X and Kenolube outperformed the other two lubricants. With an elevated die temperature, Kenolube had a far higher green strength than the three other lubricants.

The ejection characteristics for each premix at a compaction pressure of 690 MPa can be seen in Figure 7. This pressure was representative of the lubricant behavior over the range of compaction pressures tested. For each lubricant type, the first two bars in the graph denote the necessary stripping and sliding pressures to eject the part at room temperature. The last two bars show the stripping and sliding pressures from a die at 75° C. In both scenarios, the EBS wax had the worst performance, requiring higher pressures than any other lubricant to eject the sample. Kenolube was consistent at both die temperatures, while the Ancorlube samples excelled at the increased die temperature. At this temperature, both Ancorlube X and Ancorlube W had sliding pressures similar to Kenolube, but required a stripping pressure of slightly more than 20 MPa, compared to almost 25 MPa for Kenolube and almost 30 MPa for EBS wax.

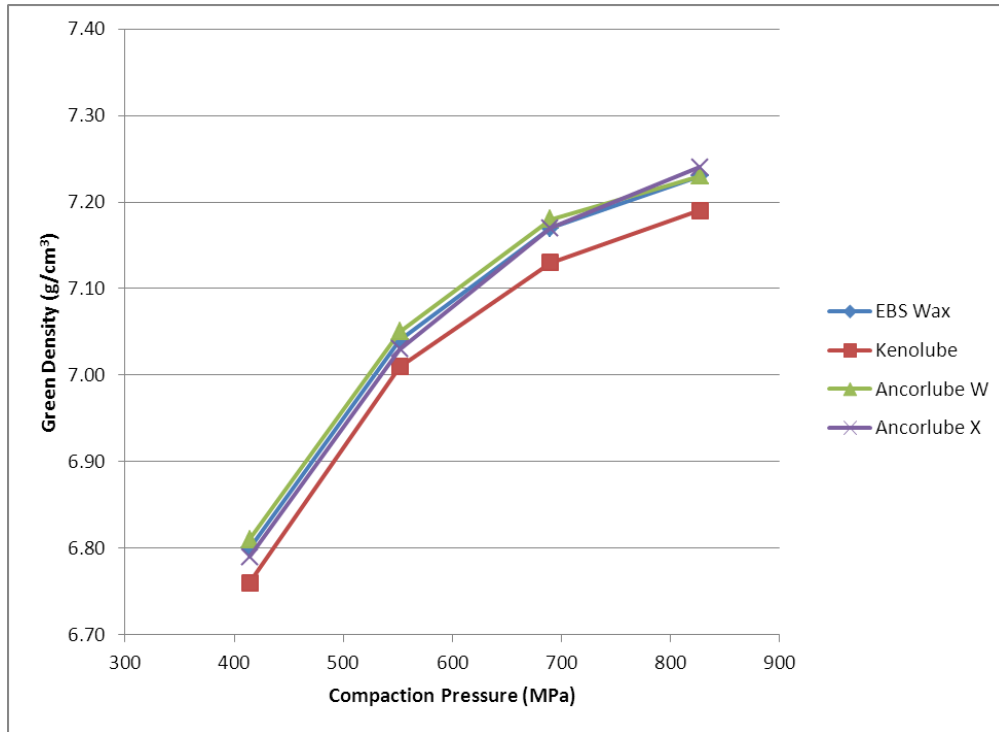


Figure 3: Green density vs. compaction pressure with a room temperature die.

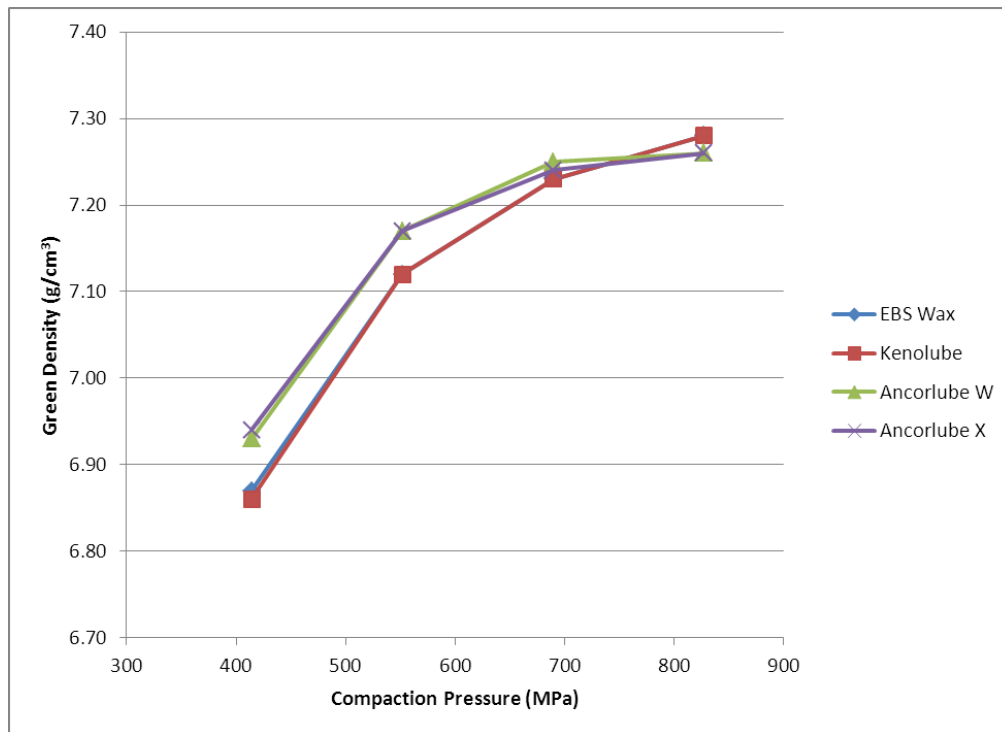


Figure 4: Green density vs. compaction pressure with a die at 75° C.

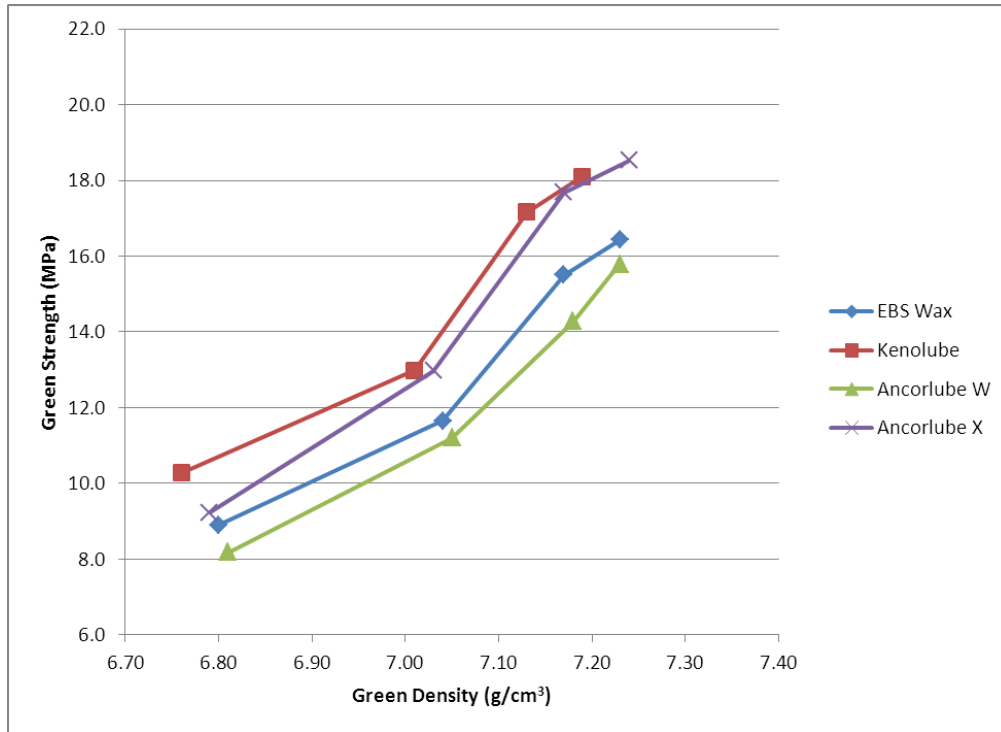


Figure 5: Green strength vs. green density with a room temperature die.

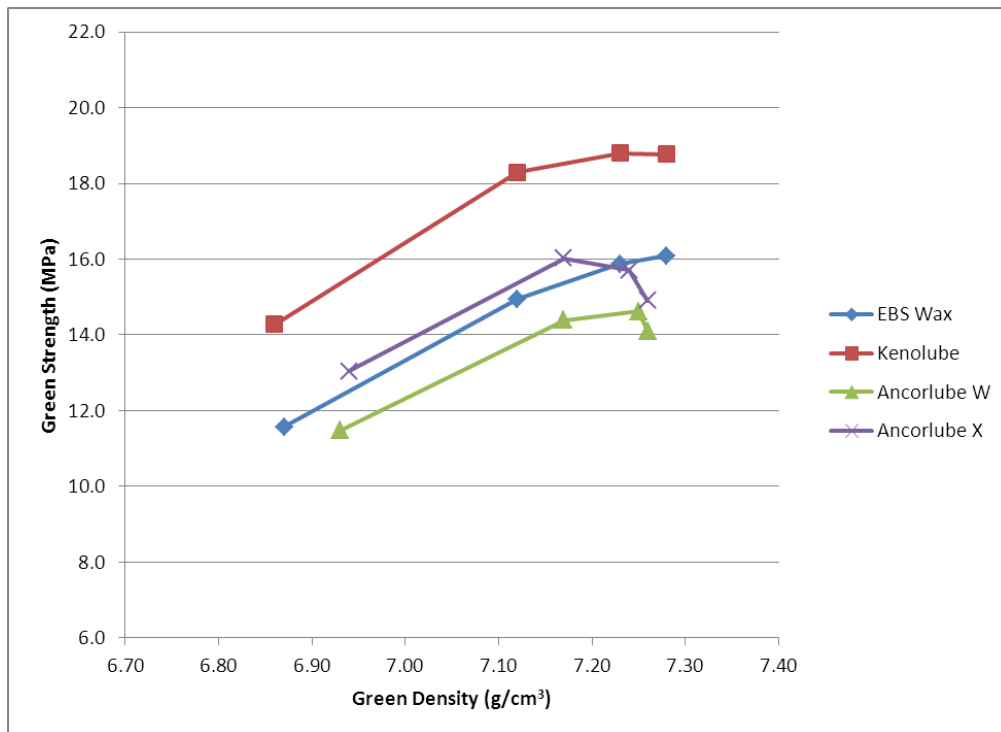


Figure 6: Green strength vs. green density with a die at 75°C.

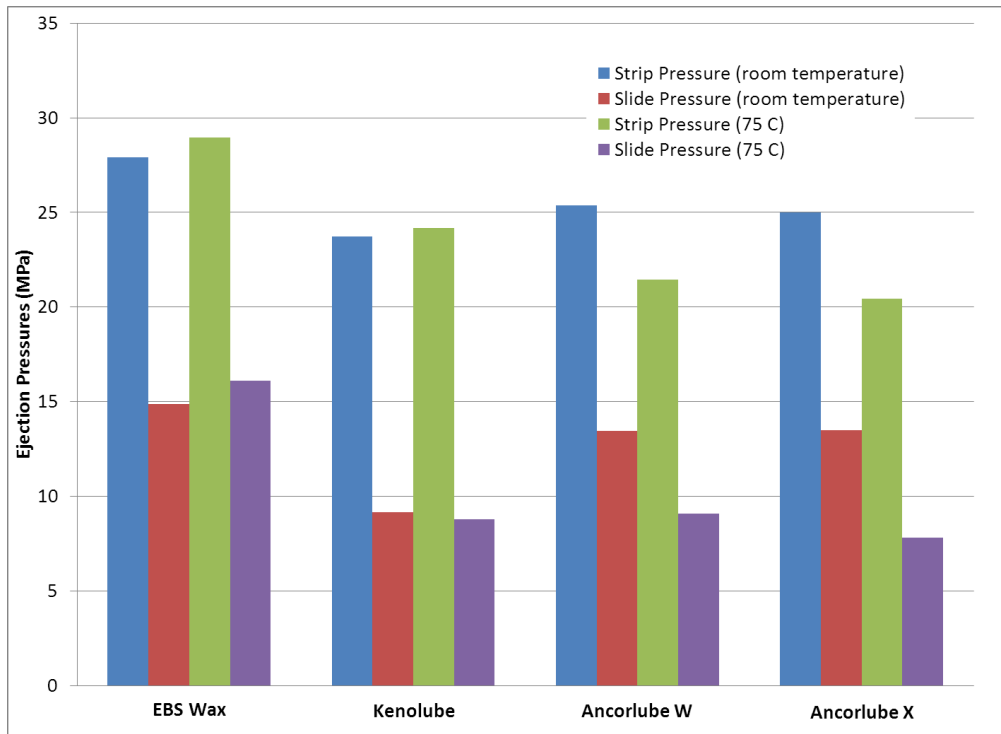


Figure 7: Ejection pressures at two die temperatures and 690 MPa compacting pressure.

Mechanical Properties

Table 3 displays some of the important sintered properties for the premixes of study, after being pressed at 75° C over a range of compaction pressures. Similar trends were observed when these same properties were measured after room temperature compaction. There is little difference that results from the various lubricant additions when pressed at either room temperature or 75° C. For the most part, it was found in this work that TR strength increases as a function of sintered density, regardless of lubricant type. Ancorlube X was found to have a slightly lower TR strength, but the difference was minimal. A similar relationship was observed while measuring ultimate tensile strength (UTS). Both properties were found to increase linearly with increasing sintered density, while lubricant type and compaction temperature had little, if any, effect. At approximately 7.15 g/cm³ density, properties such as UTS and impact stopped increasing with sintered density and began to level off for all premixes.

Table 3: Sintered properties of the premixes studied compacted at 75 °C.

Lubricant	Compaction Pressure (MPa)	Sintered Density (g/cm³)	TR Strength (MPa)	Apparent Hardness (HRA)	UTS (MPa)	Impact Toughness (J)
EBS Wax	414	6.89	990	50	492	9
	552	7.12	1197	53	563	16
	690	7.24	1297	55	585	20
	827	7.27	1362	57	584	20
Kenolube	414	6.88	997	51	477	11
	552	7.12	1181	53	555	16
	690	7.23	1260	55	577	20
	827	7.28	1306	57	592	20
Ancorlube W	414	6.95	1030	52	508	11
	552	7.19	1225	54	548	18
	690	7.28	1278	56	570	20
	827	7.31	1301	57	564	19
Ancorlube X	414	6.94	1006	52	446	9
	552	7.18	1174	55	527	16
	690	7.28	1258	56	565	20
	827	7.31	1290	58	594	20

Burn-out Study

In order to determine the burn-out characteristics of each lubricant, a 1.25 cm tall bars compacted from each of the four premixes were sintered together in the furnace and the surface finish was immediately photographed, as seen in Figure 8. A sample premix containing zinc stearate lubricant was also included in this portion of the study, because it is well known that zinc stearate can commonly leave sooting on sintered parts.

Following sintering, it was observed that EBS wax gave bars with the cleanest surface. No sooting was observed on the surface of these bars. In contrast, the bars containing Kenolube and zinc stearate had a fair amount of sooting and the poorest surface cleanliness. Ancorlube W had only a middling surface quality with some sooting, while Ancorlube X was relatively clean with only minor sooting.

The burn-out characteristics were further studied using TGA, where weight loss of the lubricant is measured vs. temperature over a range of 100 to 800° C. This relationship, shown in Figure 9, verified the sooting characteristics of the sintered bars containing these lubricants. As seen in the figure, EBS wax and Ancorlube X burn out quickly at the lowest temperature of any of the lubricants, and approximately 0% of the lubricant remains after the temperature exceeds 600° C. This means that 100% of the lubricant was vaporized and is no longer present. Meanwhile, Kenolube, Ancorlube W, and zinc stearate begin to burn off between 400 and 500° C, but level out at about 600° C and reach a minimum value of remaining lubricant between 5 and 15%. This means that after reaching a temperature equivalent to that seen in the de-lubrication zone of a sintering furnace, approximately 5% Kenolube and Ancorlube W remain, while almost 15% zinc stearate remains. This portion of the lubricant is left behind in the form of soot on the sample surface.

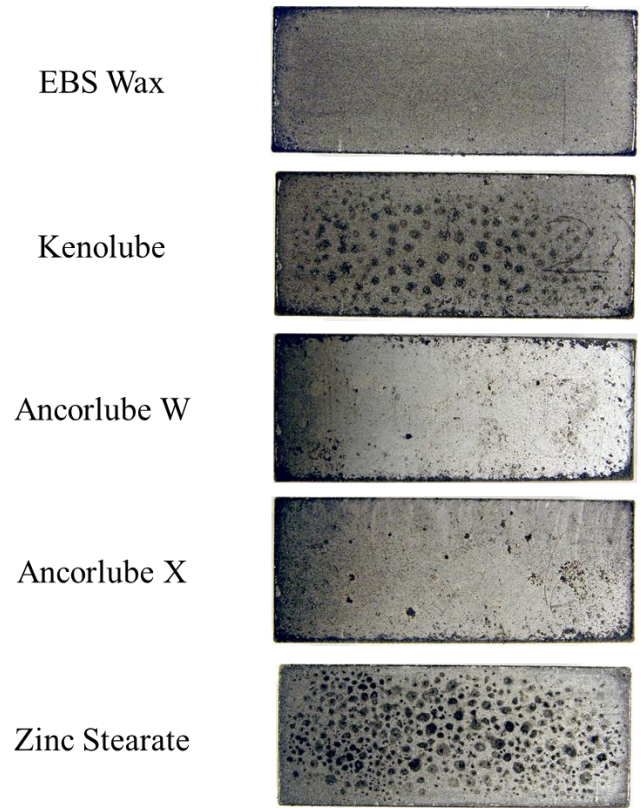


Figure 8: Surface finish on 1.25 cm tall bars after exiting the sintering furnace.

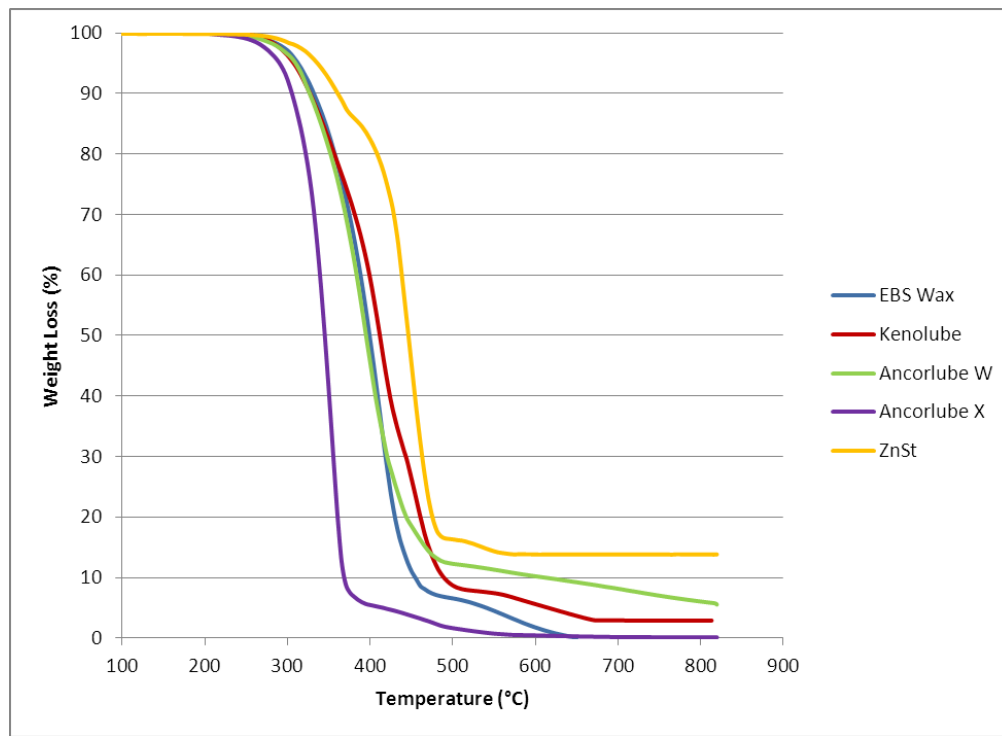


Figure 9: TGA results for the four studied lubricants and zinc stearate.

Conclusions

Four different lubricants were studied and tested for premix powder properties, green properties, sintered properties, and burn-out. The conclusions from this study are as follows:

- Each lubricant had its own characteristic powder properties including apparent density, flow, and dust resistance. Total carbon and sintered carbon were unaffected by lubricant type.
- EBS wax had the worst compressibility of any lubricant studied, and both Ancorlube samples were found to possess superior compressibility at 75° C compaction temperature.
- Kenolube had the highest green strength of any lubricant tested.
- EBS wax had the highest necessary ejection forces of the four premixes.
- Sintered properties such as TRS, UTS, and impact toughness were linearly dependent on sintered density, and did not appear effected by lubricant.
- EBS wax displayed the cleanest burn-out of the lubricants in this study, while Kenolube and zinc stearate had the most sooting. Ancorlube X was a relatively clean-burning alternative to the other lubricants.

Acknowledgements

The authors would like to thank Chen Wing Hong for his contributions to this work.

References

1. D. Saha and D. Apelian, "Control Strategy for the De-lubrication of P/M Compacts", *International Journal of Powder Metallurgy*, Vol. 38, No. 3, pp. 71-79, 2002.
2. H. Rodrigues, S. Madill, M. Folliard, T. Liu, "Optimizing Compacting Lubricant Selection – A Comparison Study of Various Commercially Available Lubricants", *Proceedings of the 2008 World Congress on Powder Metallurgy & Particulate Materials*, Washington, D.C., June 8–12, 2008.
3. S. St-Laurent, Y. Thomas, L. Azzi, "High Performance Lubricants for Demanding PM Applications", *Advances in Powder Metallurgy and Particulate Materials*, Vol. 1, Part 3, pp. 1-13, 2006.
4. D. Milligan, P. Hofecker, I. Howe, S. Miller, O. Litstrom, "Performance Characteristics of a Newly Developed PM Lubricant used in Combination with Elevated Die Temperatures", *Proceedings of the 2008 World Congress on Powder Metallurgy & Particulate Materials*, Washington, D.C., June 8–12, 2008.
5. F. Hanejko, "Single Press/Single Sinter Solutions to High Density", *Powder Metallurgy*, Vol. 53, No. 2, pp. 100-103, 2010.
6. Standard Test Methods for Metal Powders and Powder Metallurgy Products, published by MPIF, 2012.
7. C. Schade, M. Marucci, F. Hanejko, "Improved Powder Performance Through Binder Treatment of Premixes", *Proceedings of the 2011 International Conference on Powder Metallurgy & Particulate Materials*, San Francisco, California, May 18–21, 2011.