High Density Sinter-Hardened Materials with High Performance Lubricant

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ABSTRACT

Heat-treating is a secondary process that uses heating and rapid cooling to develop a martensitic microstructure. While heat treatment is utilized to achieve higher mechanical properties and improve overall part performance, the addition of secondary operations such as quenching increase overall fabrication costs compared to single operation sintering. Sinter hardening is an operation that combines both conventional sintering and secondary heat treatment into a single process by using accelerated cooling in the sintering furnace. Prealloyed material systems that yield high hardenability, such as those alloyed with chromium and molybdenum, are capable of developing small percentages of martensite during conventional sintering. By utilizing accelerated cooling during the sintering process, these materials are capable of achieving microstructures and mechanical properties equal to or greater than heat-treated components. However, one of the drawbacks to using prealloyed base iron is that they have lower compressibility compared to standard iron powder. Because of this, specially designed lubricants are an important development to achieve the highest densities possible. A new lubricant system has been developed which provides improved density and ejection performance. The mechanical properties of prealloyed chromium materials with the high performance lubricant will be compared to other sinter hardened materials and lubricant systems.

INTRODUCTION

Most ferrous materials in the as sintered condition consist of the microstructure phases ferrite and pearlite [1]. During heat treatment, the microstructure phase known as martensite is formed. Martensite is a highly strained body-centered tetragonal form of ferrite. The phase forms during rapid cooling which restricts carbon atoms from diffusing out of the crystal lattice. [2]. This causes the lattice to distort and form a supersaturated solution of carbon, know as martensite. While heat treating is a proven method to improve strength and performance, it is also a costly and challenging operation that requires extra production time, precise temperature control during both the heating and cooling process, and consistent atmosphere control to ensure component consistency [3]. Failure to adequately control the temperature and atmosphere during heat treatment can cause distortion and cracking in components.

Sinter hardening is a single step process capable of developing martensite by using accelerated cooling in the sintering furnace. This process decreases production time, reduces distortion, and improves dimensional control due to less severe heating and cooling rates compared to the quench hardening process [4]. By controlling the cooling rate and alloy composition, the sinter hardening process is capable of being manipulated to achieve the desired microstructure and mechanical properties [5].

Before high cooling rates were available on typical sintering furnaces, sinter hardening could only be accomplished using highly alloyed materials with high carbon levels [6]. Molybdenum, nickel, and more recently chromium are common alloying elements used in prealloyed materials. Copper is also a common additive that can be admixed to prealloyed irons for additional hardenability. Molybdenum, nickel and copper were the most common alloying elements used in PM materials because they are less sensitive to oxidation than chromium. [5]. However, due to developments in conventional sintering atmospheres, prealloyed chromium has become increasingly popular as an alloy addition in PM materials due to its lower cost, exceptional hardenability and resistance to temper softening [7]. Chromium is also proven to provide better dimensional stability in sinter hardened systems compared to traditional prealloyed materials. This is because chromium is capable of achieving high hardenability without high graphite levels or additional copper, thus reducing distortion [8].

However, by prealloying iron with elements which increase hardenability, the particle hardness of the alloy also increases. The increase in particle hardness reduces the particles' ability to distort during compaction, which leads to lower compressibility of prealloyed materials compared to typical iron powder (Figure 1). The higher the alloy content of the prealloyed particles, the lower the compressibility of the material. It should also be noted that different alloying elements affect the compressibility more than others. Molybdenum has only a small effect on compressibility when prealloyed, while nickel and chromium reduce the compressibility significantly.

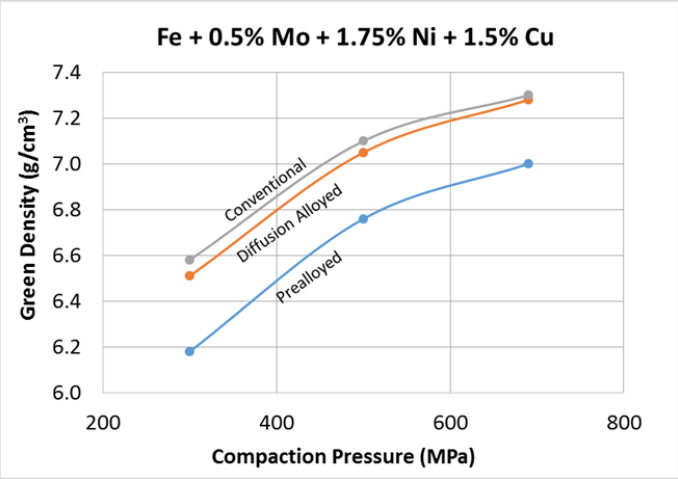


Figure 1: Compressibility curves for ferrous powders produced using different alloying methods with the same nominal chemical composition [9].

While sinter hardening materials result in martensitic microstructures with increased strength properties, high density is also an important property for achieving high performance components. Porosity reduces the physical properties of PM components compared to wrought steel. Densification and a components overall strength have a linear correlation, where the higher the component density, the higher the strength. Figure 2 shows the correlation of density compared to selected mechanical properties. Tensile and fatigue strength properties increase linearly as density increases, while elongation and impact energy increase exponentially [10]. Due to prealloyed materials having lower compressibility than standard iron, other considerations need to be taken to achieve the highest density possible.

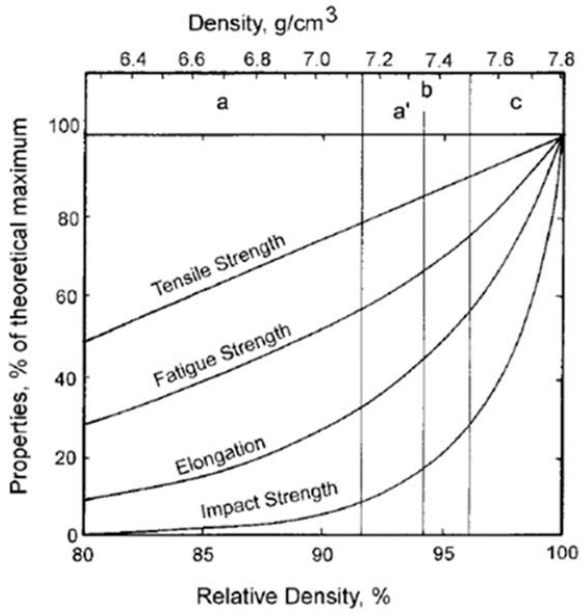


Figure 2: Density vs Physical properties [10]

Though lubrication is necessary in the PM compaction process to reduce die and particle friction and eject components out of the die, lubricant content adversely effects a components achievable density. Increased

lubricity at the die wall is proportional to the lubricant content added to the mix [11]. By utilizing lubricants with increased lubricity, it is possible to reduce lubricant content while maintaining similar ejection forces, thus increasing densification. Warm die compaction is a compaction technique that consists of heating the tooling of the component to assist in softening lubricant. By softening the lubricant, it is allowed to travel to the die surface faster, thus getting out of the way of the iron particles during compaction and increasing densification. This technique also allows for the lowering of total lubricant content required leading to increased densification.

This paper will evaluate a specially designed lubricant in combination with warm die compaction, to achieve optimum density levels for prealloyed iron commonly used in sinter hardened applications. It will evaluate the effect lubricant content and density level have on surface appearance and strength properties of different sinter-hardened prealloyed material systems.

EXPERIMENTAL PROCEDURE

Three commercially available prealloyed base irons commonly used in sinter hardening applications were chosen for this study. The alloys and compositions are listed in Table 1.

Table 1: Prealloyed Powder Composition (w/o)

Base	Mo	Mn	Ni	Cr
Astaloy® 85 Mo	0.85	0.1	-	-
Astaloy® CrM	0.50	0.1	-	3.0
Astaloy® MoNi	1.20	0.42	1.35	-

Mixes were manufactured from the three alloys according to MPIF designations. Various lubricant levels were targeted in each mix comparing a newly designed lubricant system verse a standard industry lubricant. Table 2 shows the material systems used in the evaluation.

Table 2: Material Systems Evaluation (w/o)

Base	Cu	Ni	Graphite	Lubricant	MPIF Code
Astaloy® 85Mo	2%	2%	0.8%	0.4% Intralube® HD	FLNC-4408
Astaloy® 85Mo	2%	2%	0.8%	0.5% Intralube® HD	
Astaloy® 85Mo	2%	2%	0.8%	0.6% Intralube® HD	
Astaloy® 85Mo	2%	2%	0.8%	0.75% Amide Wax	
Astaloy® CrM	-	-	0.6%	0.4% Intralube® HD	FL-5305
Astaloy® CrM	-	-	0.6%	0.5% Intralube® HD	
Astaloy® CrM	-	-	0.6%	0.6% Intralube® HD	
Astaloy® CrM	-	-	0.6%	0.75% Amide Wax	
Astaloy® MoNi	2%	-	0.9%	0.4% Intralube® HD	FLC2-4808
Astaloy® MoNi	2%	-	0.9%	0.5% Intralube® HD	
Astaloy® MoNi	2%	-	0.9%	0.6% Intralube® HD	
Astaloy® MoNi	2%	-	0.9%	0.75% Amide Wax	

Each mix was compacted into cylindrical pucks with a 40 mm (1.57 in) diameter at compaction pressures of 400 MPa, 550 MPa, and 690 MPa (30, 40, 50 tsi). The green density was recorded at each compaction pressure to generate a compressibility curve. Ejection forces were also measured at each compaction pressure. Based on the compressibility data, a maximum height compaction study was conducted to compare the surface appearance of each lubricant system and level. Cylindrical pucks with a 40 mm (1.57

in) diameter were compacted to different heights. Mixes using the new lubricant system were compact using a die heated to 90 ºC (194 °F). Mixes using Amide Wax were compacted in a die at room temperature. The heights were incrementally increased from 15 mm (0.59 in) to 40 mm (1.57 in) at 5 mm (0.20 in) intervals, while maintaining a density level of 7.10 g/cm^3. Pucks over 40 mm (1.57 in) could not be compacted due to die fill limitations on the press. The surface appearance of the specimens was evaluated.

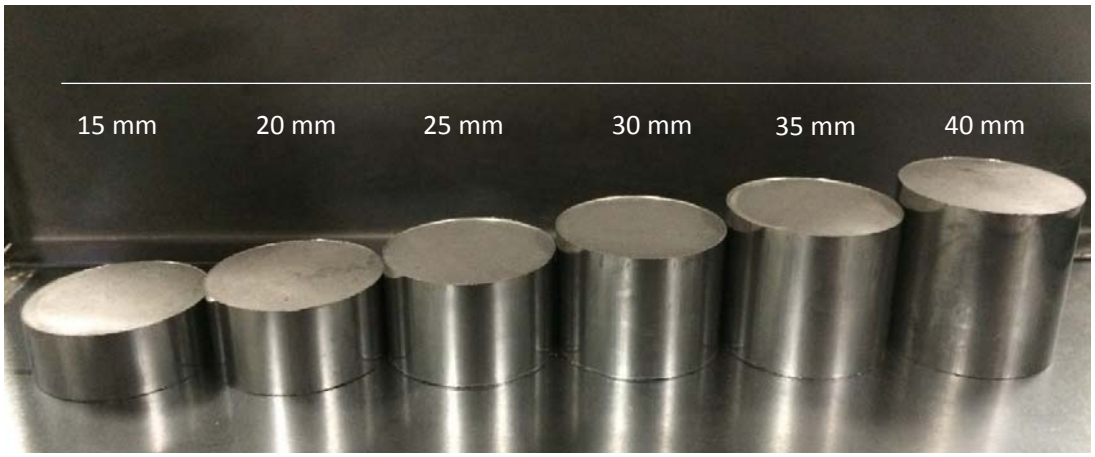
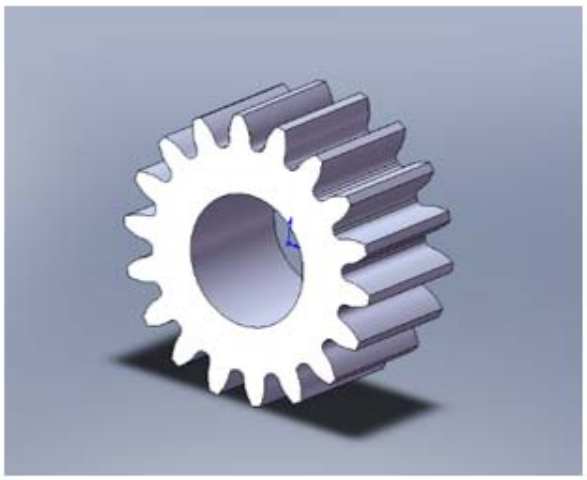


Figure 3: Incremented Height Specimens

Gear specimens (Figure 4) were compacted from the mixes containing 0.5% Intralube® HD and 0.75% amide wax. The Intralube® HD mixes were compacted in a die heated to 90 ºC (194 °F). The amide wax mixes were compacted at room temperature. The gears were compacted at a pressure of 690 MPa to a height of 20 mm (0.79 in). The gears were sinter hardened at 1121 °C (2050 °F) for 20 minutes in a 90/10 N_2/H_2 atmosphere using a cooling rate of 2 °C/sec (4 °F/sec). The gears were then tempered at 204 °C (400 °F) for 1 hour in air.



HB Gear	
No. of Teeth	18
Module	1.56
Pressure Angle	20 °
Major Dia.	31.5 mm
Minor Dia.	24.5 mm
Root Radius	0.69 mm
Tooth Thickness	2.4 mm

Figure 4: Gear Specimen

Five gears from each mix and density were evaluated for gear tooth crush strength. The remaining gears were evaluated for hardness, sintered density, microstructure, microhardness according to industry standards.

RESULTS

40 mm Puck Specimens

Figure 5 shows the compressibility at 400, 550 and 690 MPa (30, 40, 50 tsi) comparing amide wax to three different lubricant levels of Intralube® HD for each material system. The compressibility curves show the mixes with the lowest lubricant content of 0.4% Intralube® HD result in the highest densities compared to the mixes with higher lubricant contents. The FLNC-4408 prealloyed material achieves higher densities than the other two prealloyed materials, capable of reaching 7.3 g/cm^3 green density at 690 MPa with a lubricant content of 0.4% Intralube® HD. The FL-5305 and FLC2-4808 mixes achieve similar densities of 7.17 g/cm^3 at 690 MPa.

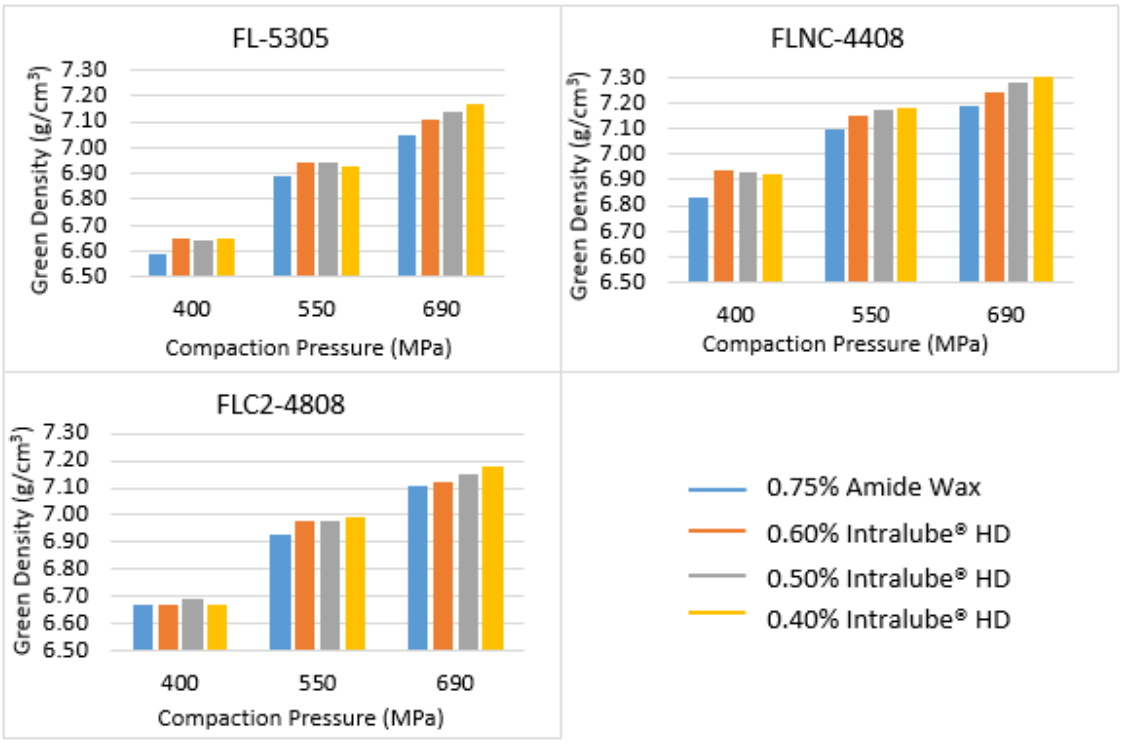


Figure 5: Compressibility

Figure 6 shows the ejection properties at 400, 550, 690 MPa (30, 40, 50 tsi) comparing amide wax to three different lubricant levels of Intralube® HD for each material system. The stripping pressure of each prealloyed material system with various lubricant contents are shown. The results show that the advanced lubricant mixes with 0.6% lubricant content has lower peak ejection forces compared to the mixes with lower lubricant contents. The mixes with 0.5% and 0.4% Intralube® HD lubricant levels have similar ejection forces as the 0.75% amide wax mixes. All three prealloyed material systems have similar ejection forces at each lubricant content.

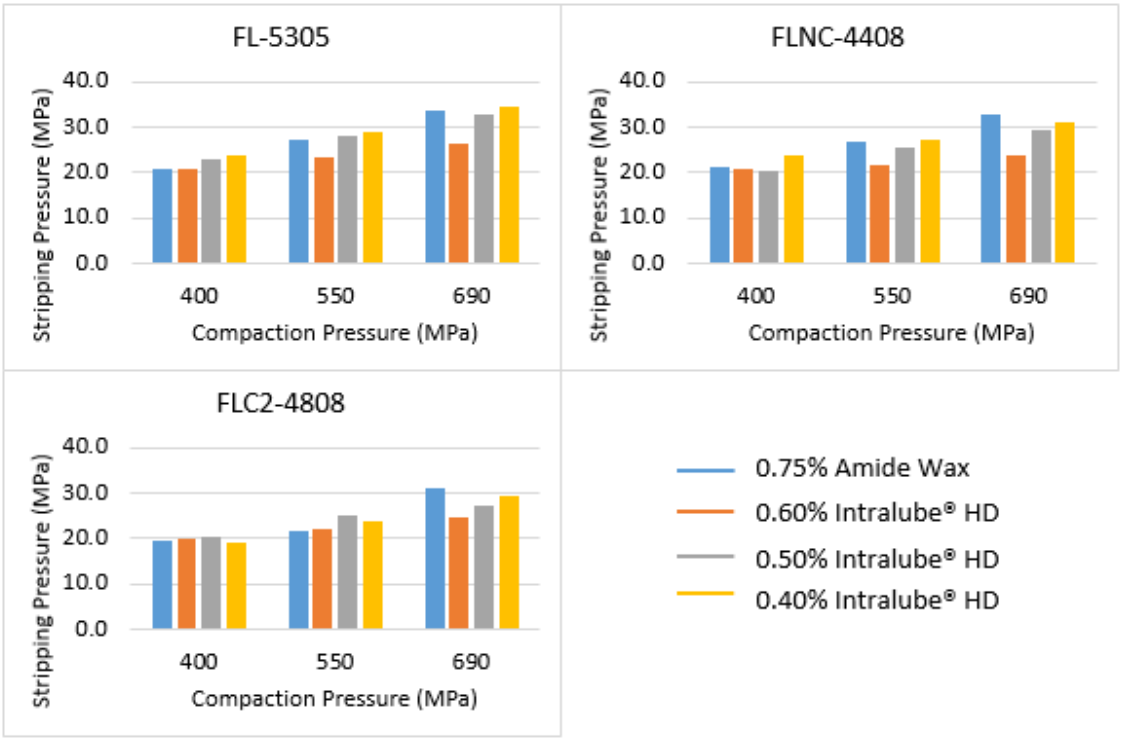


Figure 6: Stripping Pressure

Figure 7 shows the surface appearance of FLNC-4408 of the 40 mm pucks from the maximum height trial. The surface appearance of the mixes with lower additions of the advanced lubricant system are similar to that of the mix with 0.75% Amide Wax.

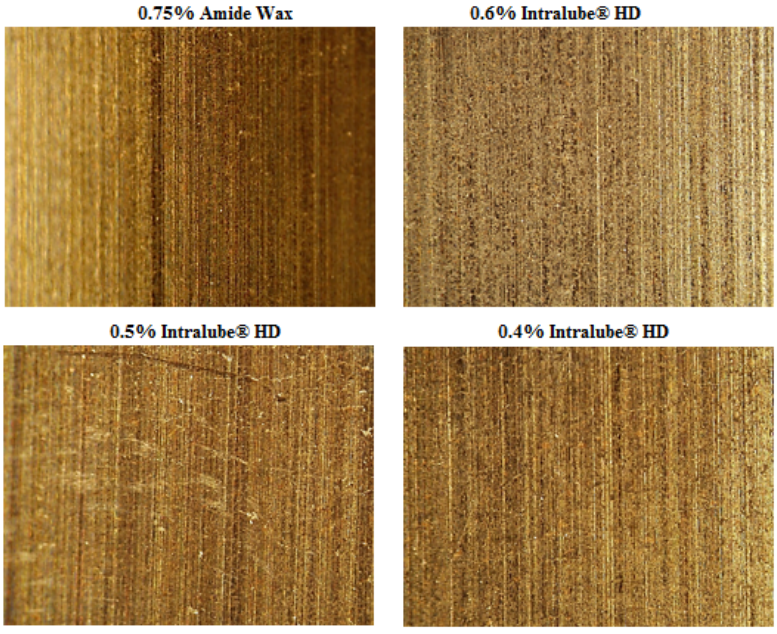


Figure 7: Surface appearance of FLNC-4408 40 mm pucks

Gear Specimens

Figure 8 shows the gear tooth crush strength of the amide wax mixes compared to the 0.5% Intralube® HD mixes. The advanced lubricant mix resulted in higher gear tooth crush strength compared to the mixes with 0.75% amide wax. The FLNC-4408 mix resulted in the highest gear tooth crush strength compared to the other prealloyed mixes.

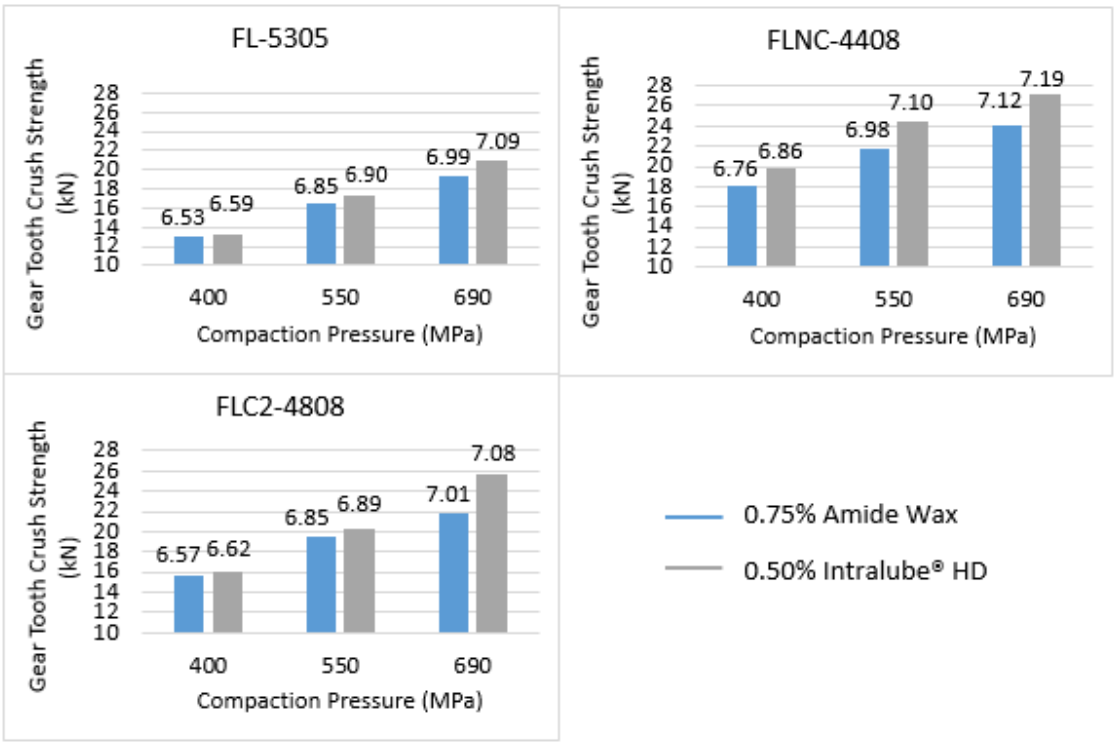


Figure 8: Gear tooth crush strength at gears compacted to 690 MPa vs sintered densities

The microstructures of the Amide wax mixes vs the 0.5% Intralube® HD mixes are shown in Figures 9-14. FLNC-4408 material system has a microstructure of bainite, martensite and nickel rich austenite. FLC2-4808 and FL-5305 microstructures were fully martensitic. The martensite of the FL-5305 material has fine, lathe martensite needles. The FLC2-4808 martensite shows a coarser, plate martensite.

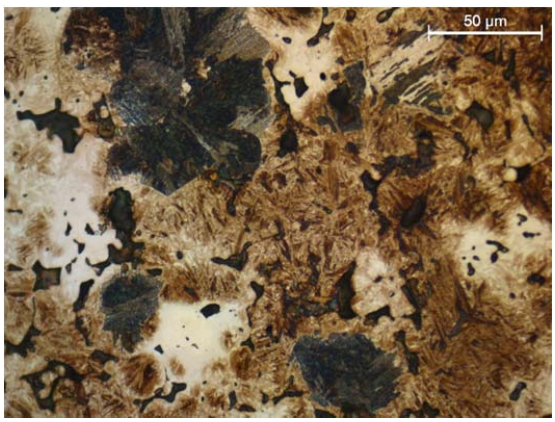


Figure 9: FLNC-4408 with 0.75% Amide Wax

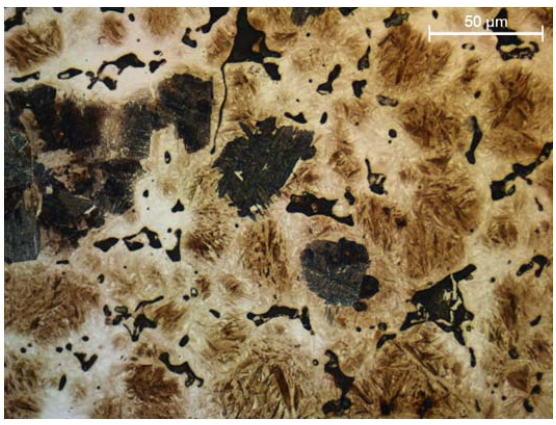


Figure 10: FLNC-4408 with 0.5% Intralube® HD

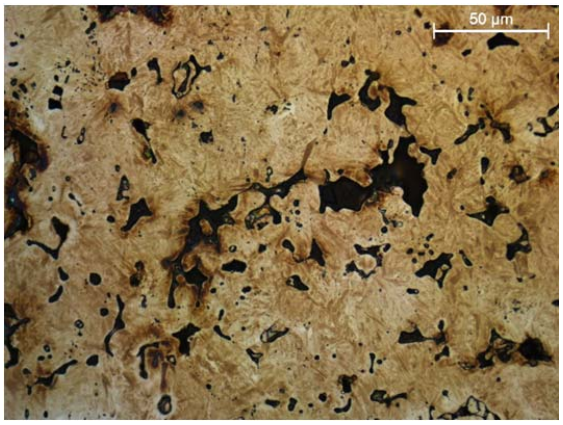


Figure 11: FL-5305 with 0.75% Amide Wax

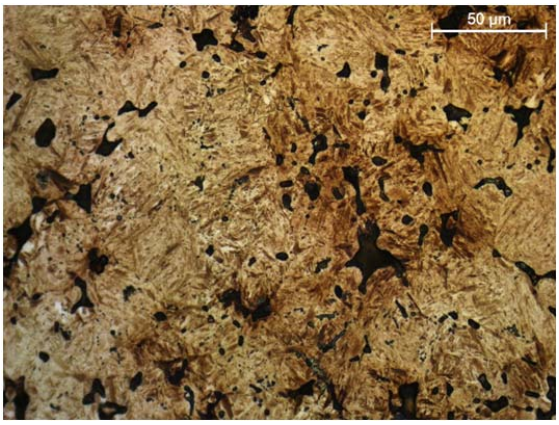


Figure 12: FL-5305with 0.5% Intralube® HD

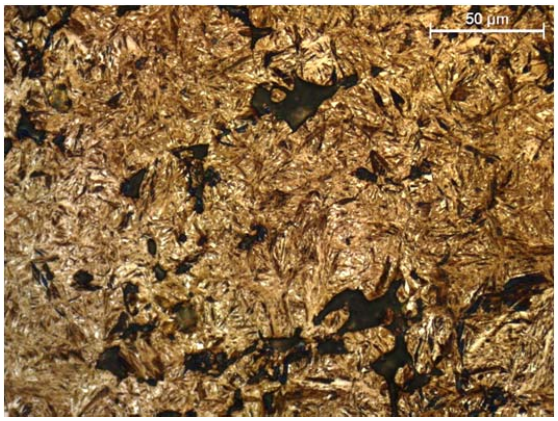


Figure 13: FLC2-4808 with 0.75% Amide Wax

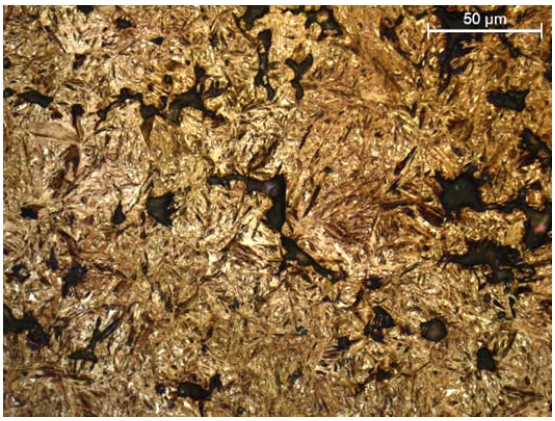


Figure 14: FLC2-4808 with 0.5% Intralube® HD

DISCUSSION

Sinter hardened materials are designed to achieve high hardenability to provide excellent mechanical properties. By utilizing these high strength materials along with an advanced lubricant system, even further improvement can be made to strength values, which will open the way to allow larger, high strength components to be manufactured using powdered metal (PM) materials.

Achieving high density in prealloyed materials is necessary for high performance applications that require high strength. Due to the decrease in compressibility of materials that have been prealloyed, development of advanced lubricant systems that provided increased lubricity is key. Improved lubricity allows for a reduction of lubricant content, which increases a materials achievable density.

Figure 14 shows the MPIF Standard 35 Tensile properties for the three prealloyed materials evaluated in this paper. By increasing the density of these materials, significant improvement in strength is obtained. The FL-5305 material, which has the highest overall tensile strength, shows a 10% increase in strength from 7.0 to 7.2 g/cm^3 sintered density. The FLC2-4808 has a 29% increase in strength and FLNC-4408 a 23% increase.

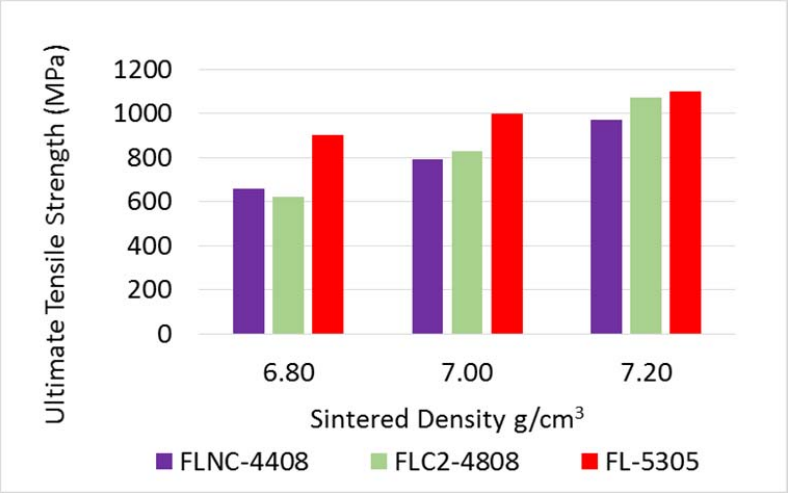


Figure 14: MPIF Standard 35 Tensile Properties (12).

When compacting to high densities, it is also important to be aware of the effect it will have on tool wear and component appearance. Tool breakage and poor surface appearance will increase the overall cost of production. High density material systems need to be capable of being compacted to high density and ejected from the die without detriment to the tool or components themselves.

The stripping pressure shown in Figure 5 shows the advanced lubricant system at 0.5% and 0.4% additions have similar ejection performance to the mixes with 0.75% amide wax. The improved lubricity of the advanced lubricant system reduces the ejection forces during compaction and also allows for taller components to be manufactured without degrading surface appearance and causing tool damage. With the advanced lubricant system, 40 mm tall pucks were capable of being compacted with 0.4% lubricant content while maintaining similar surface appearance to those with higher lubricant contents. In addition, by being able to compact taller components with less lubricant, higher densities are achievable.

CONCLUSION

- The Intralube® HD mixes with 0.6%, 0.5% and 0.4% lubricant content had improved compressibility compared to the 0.75% amide wax mixes.

- The Intralube® HD mixes with 0.6% lubricant content have lower peak ejection forces compared to the mixes with lower lubricant contents. The mixes with 0.5% and 0.4% Intralube® HD lubricant levels have similar ejection forces as the 0.75% amide wax mixes. All three prealloyed material systems have similar ejection forces at each lubricant content.

- The 0.5% Intralube® HD mix had higher overall gear tooth crush strength properties for all three prealloyed mixes compared to the 0.75% Amide Wax mixes. The FLNC-4408 material had the highest gear tooth crush strength at each density compared to the FL-5305 and FLC2-4808 material systems

REFERENCES

[1] W. Brian James, "What is Sinter Hardening", 1998, *International Conference on Powder Metallurgy & Particulate Materials.* Las Vegas, NV. Hoeganaes Corporation, Cinnaminson, NJ.

[2] Pablo G. Caceres-Valencia "Martensite" University of Puerto Rico at Mayagüez http://academic.uprm.edu/pcaceres/Courses/MatEng/MSE8-2.pdf

[3] Global Security, *Steelworker - Basic Heat Treatment*, Global Security Non Resident Training Coarse 14250 1996, Vol 1, Ch 2 http://www.globalsecurity.org/military/library/policy/navy/nrtc/14250_ch2.pdf

[4] Hatami, S., Malakizadi, A., Nyborg, L., & Wallin, D. "Critical aspects of sinter-hardening of prealloyed Cr-Mo steel" (2010), *Journal of Materials Processing Technology*

[5] Ulf Engstrom, "Evaluation of Sinter Hardening of Different PM Materials" (n.d), Höganäs AB, Sweden

[6] Bruce Lindsley " Alloy Development of Sinter-Hardenable Compositions", 2007, Hoeganaes Corporation, Cinnaminson, NJ.

[7] Bo Hu, Alex Klekovkin, Dave Milligan, Ulf Engstrom, Sigurd Berg, Barbara Maroli, " Properties of High Density Cr-Mo Pre-alloyed Materials High Temperature Sintered" (n.d), North American Höganäs, USA, Höganäs AB, Sweden

[8] Roland Warzel III, Amber Neilan " Effect of Composition and Processing on Precision of Sinter Hardening Powder Metal (PM) Steels (2013) North American Höganäs, USA

[9] Höganäs, AB, *Höganäs Handbook for Sintered Components* - Material and Powder Properties pg 2-17, 2004 Höganäs AB, Höganäs Sweden

[10] Höganäs, AB, *Höganäs Handbook for Sintered Components* - Design and Mechanical Properties, 2004 Höganäs AB, Höganäs Sweden.

[11] Sylvain St-Laurent, Yannig Thomas, Lhoucine Azzi, "High Performance Lubricants for Demanding Applications" (n.d), Quebec Metal Powers Limited Tracy, Quebec, Industrial Materials Institute, National Research Council Canada, Boucherville, Quebec

[12] *MPIF Standard 35 Material Standards for PM Structural Parts.* (n.d), MPIF