Characterization of Surface Compound Formation on PM400

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Caterpillar uses turbocharger systems in its land-based engines to optimize efficiency. In this system, wastegates are used to regulate boost pressure. The wastegate assembly utilizes two bushings made of PM400, a dry lubrication system with a low temperature and high temperature lubricant. This study aims to characterize how these solid lubricants sustain through their part life and how they are enabled at different operating conditions. It was seen through heat treatment experiments that the oxides in the system coarsen, leading to a change in area fraction. In addition, XRD and SEM showed that compositional changes were far and few throughout the heat treatment. It was hypothesized through literature that the mechanism of solid lubricants is shear stress on silver and fluorides which is affected by the temperature and conditions in the system[2].

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CATERPILLAR

Project Background

CAT is a worldwide leader in land-based engines used for power generation and other applications. Turbochargers are utilized to regulate boost pressure in the engine with the wastegate assembly being the mechanism that opens to lower pressure. PM400 bushings are used in the wastegate assembly to support the inconel shaft. PM400 is a powder metal that is a mix of chrome oxide, silver, and calcium fluoride or barium fluoride all held together by a nickel alloy [1]. Silver is the low-temperature solid lubricant and fluorides are the high-temperature lubricant with chrome oxide being the hardener in the material. PM400 was developed by NASA to be used in rocketry, usually a duration of 15 minutes. PM400 is now used in CAT engines for months at a time. This project will explore how the material reacts to long duration heat, and how the lubricants are activated.

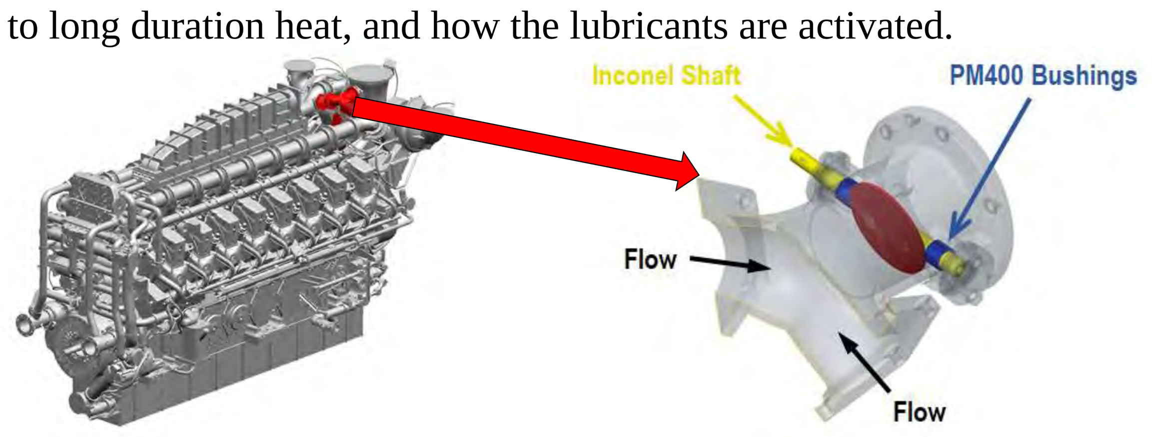

Figure 1. *Caterpillar G3616 A4, a natural gas fueled engine with the wastegate valve highlighted in red. The wastegate assembly is magnified on the right with the components marked.*

Figure 2. *The PM400 bushing : top view (left side) and isometric view (right side).*

Experimental Procedure

Samples: Five stock bushing samples and four field samples were cut into 8 specimen each as per the cutting diagram seen below. The samples were massed, and dimensions were recorded to describe the initial conditions.

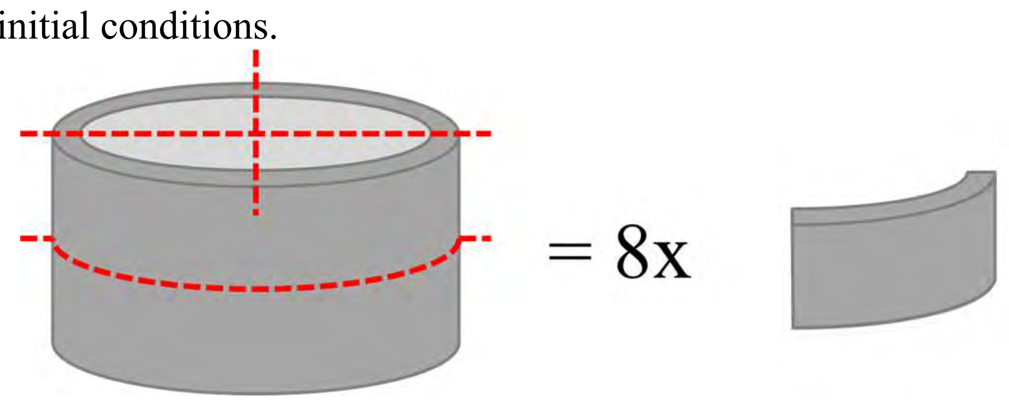

Figure 3. *Example bushing with red dotted lines indicating where sectioning occurred. This shows how 8 specimen we pulled from the sample.*

Heat Treatment: Three sample sets were heat treated for 12, 24, 48, 96, 250, and 500 hours in the furnace at the temperatures of 500°C, 600°C, and 650°C. The treated samples were rapidly air-cooled with a bellow to freeze the microstructure at that stage.

Figure 4. *A FB1400 Thermo Scientific Thermolyne furnace used to heat treat samples at different temperatures for 9 weeks.*

Analysis: Each specimen was analyzed via D8 Bruker XRD before being mounted and polished. The mounted specimen were polished with 120, 240, 400, 600, and 800 grit paper. They were consequently analyzed via optical microscopy (Olympus BX41) and SEM (Phenom Pro) and EDS.

Results

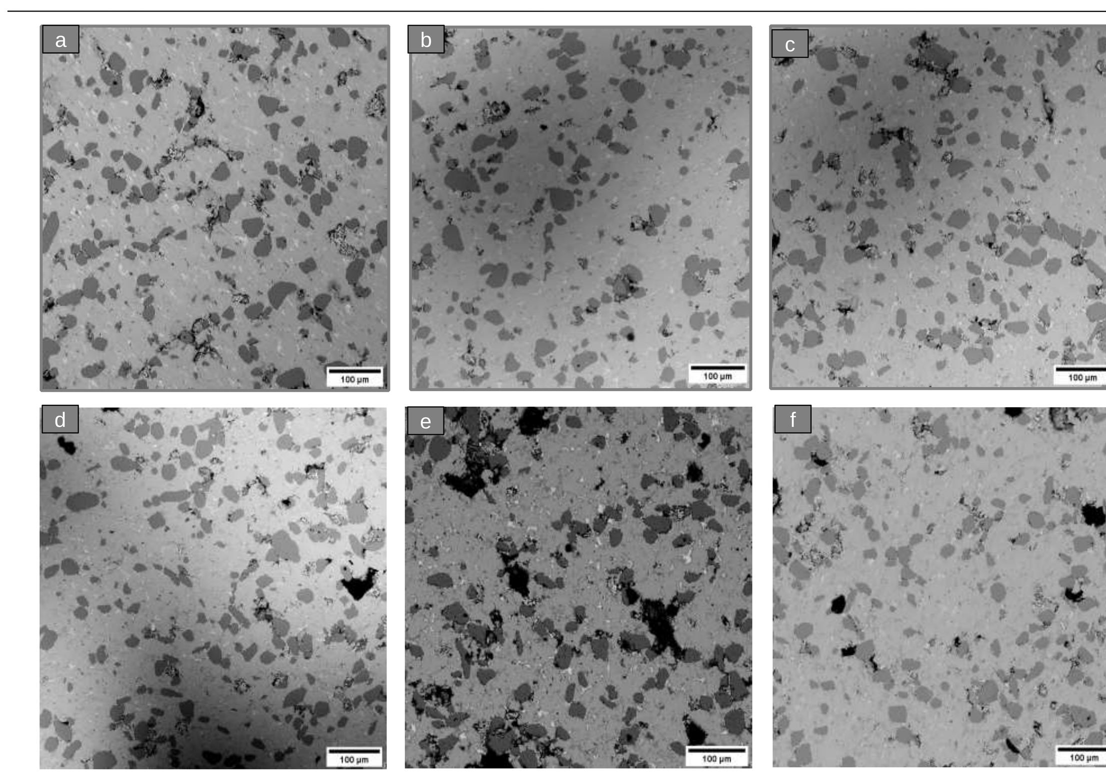

Figure 5. *SEM imaging of samples (a) 34.1, (b) 34.2, (c) 34.3, (d) 34.4, (e) 34.6, and (f) field sample B1. Samples 34.1-34.6 were heat-treated at 500°C for 12, 24, 48, 96, and 500 hours, respectively.*

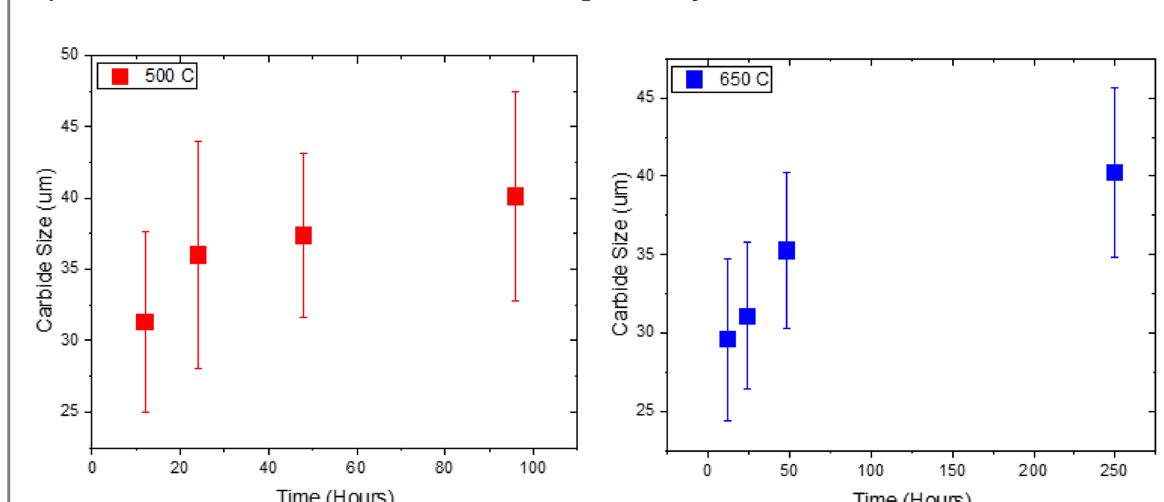

Figure 6. *The charts show the average oxide size across different heat treatment times for a given temperature (500 C on left and 650 C on right). This graph represents the precipitation of the oxides on the system.*

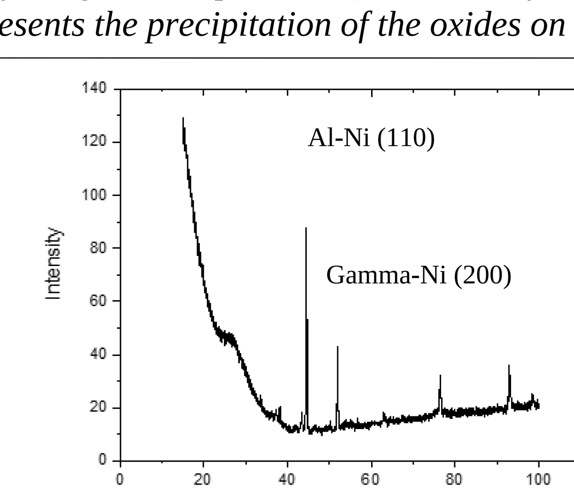

Figure 7. *A representative XRD spectra is seen for the PM400 samples. This spectra was constant with all samples including the heat treated ones. The major peaks are indexed as seen.*

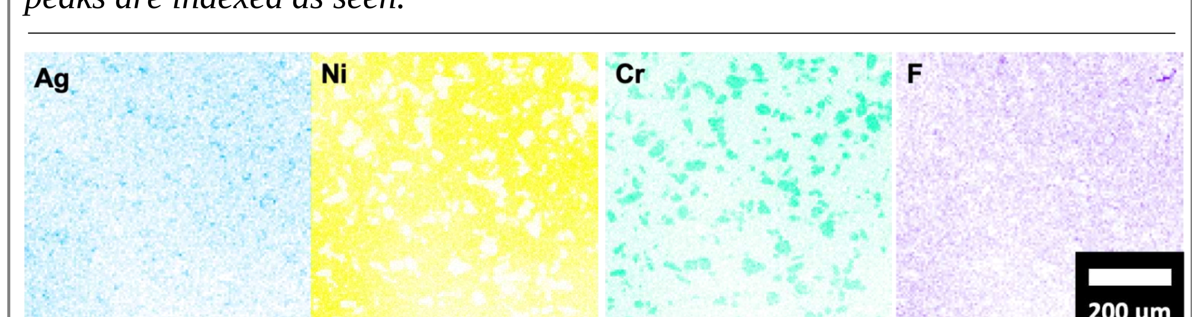

Figure 8. *EDS color maps are seen outlining the silver, nickel, chromium, and fluorine present in the system. The sample shown was heat treated for 12 hours at 500 C.*

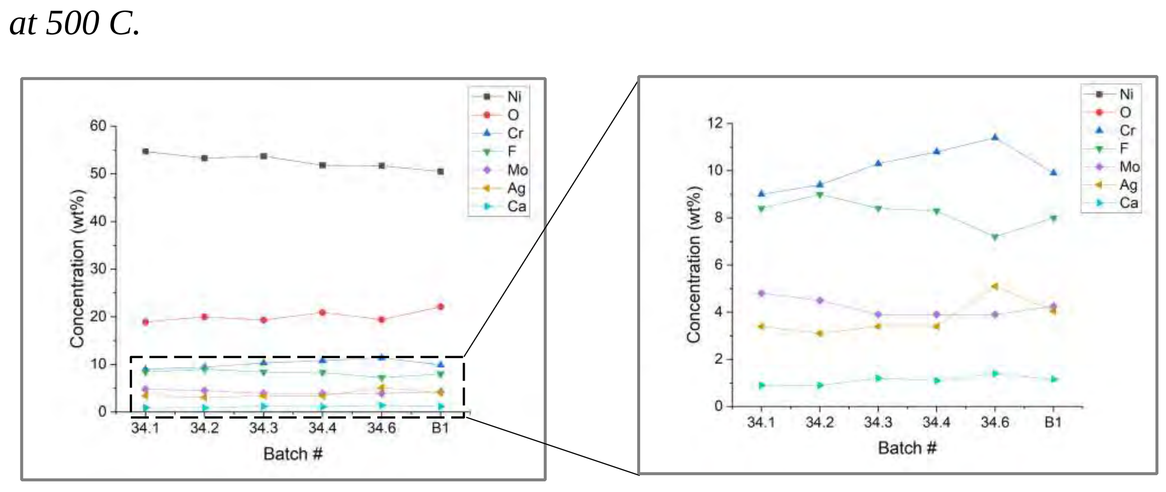

Figure 9. *Experimental EDS data for 500°C tempered PM400 samples 34.1-34.6 and field sample B1 for elements Ni, O, Cr, F, Mo, Ag, and Ca.*

Table 1. *The area percentages are seen for the matrix, hardener, and lubricants compared across measurement methods.*

Batch	34.1	34.2	34.3	34.4	34.1	34.2	34.3	34.4
Type	Area Percentage (%)				EDS Color area percentage			
Ag	3.58	2.19	2.63	1.71	19.7	10.6	12.6	14.0
Ni	79.82	75.55	73.12	70.58	76.2	75.9	80.6	80.3
Oxides	16.37	21.29	22.31	26.88	17.8	16.8	17.7	20.3
Fluorides	0.56	0.97	2.33	1.21	26.2	21	17.5	15.0
Porosity present increasing value					Note: EDS areas overlap			

Discussion

Activation Mechanism

The solid lubricants are activated at different temperatures. The basis for activation is dependent on the temperature and shear stress applied on the solid lubricants. As temperature increases, the shear stress of the lubricants decrease as the soften up and hence are more shearable. In summation, different lubricants soften enough to be sheared at certain temperatures. This is when they are dubbed as activated.

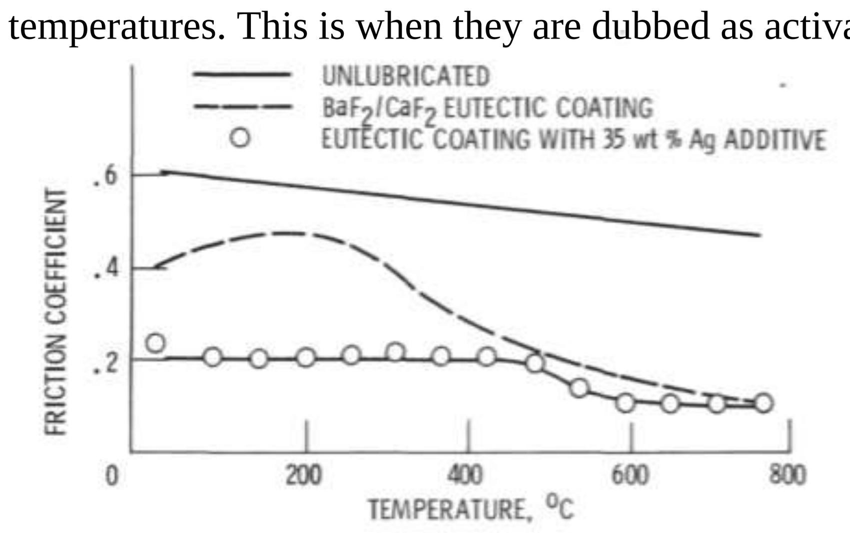

Figure 10. *Friction coefficient vs temperature for surfaces with and without solid lubricant coating [2].*

Microstructural Evolution

SEM and Optical Microscopy: The microstructure of the PM400 bushing evolves throughout heat treatment. Visually, it can be seen that the grains coarsened and got larger at longer heat treatment times. Quantified in Figure 6, that the oxides in the system grew and plateaued their growth after hitting a saturation limit. The growth of the oxides is beneficial in that the matrix has a higher volume fraction of hard particles present. In comparison to the field samples, the treated samples had larger grains and oxide particles. However, a conclusion cannot be drawn as the exact field conditions are not known.

X-Ray Diffraction: As seen in Figure 7, the microstructures XRD results did not change at a high temperature test. This means that the powder metal is made with stable alloys and is not prone to react with the air or each other. Corrosion test were planned to test the materials reactiveness to substances that a PM400 bushing could experience in an engine. However, current circumstances made the experimentation impossible.

EDS and Surface Area Analysis: The silver changes seen in the microstructure are unusual as they are not consistent across the EDS detection method and is not explained with diffusion. However there is a possibility that the silver is not evenly distributed leading to a deficiency with silver in a particular area due to a lack of silver in the first place. This may cause erosion problems in service as silver is the low-temperature lubricant. As seen in Figure 9, the EDS further showed that most of the major elements maintained a consistent composition. The increase noted in chromium is due to the coarsening of oxides through the heat treatment.

Recommendations

We recommend that there be a lower temperature test to get the oxide grain size changes as well as a more sensitive analysis done to accurately observe composition. In addition, we recommend a high temperature corrosion test to observe the stability of the powder metal in a high temperature exhaust environment.

References

[1] Schabes, H. (2019). PS/PM400 Solid Self-Lubricating Composite for Extreme Temperatures. Retrieved November 22, 2019, from https://technology.grc.nasa.gov/featurestory/ps-pm400.
[2] Miyoshi, K. (1996). Solid Lubrication Fundamentals and Applications. Chapter 1: Introduction and Background . Retrieved from https://ntrs.nasa.gov/search.jsp?R=19960045816

Acknowledgements

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