Ongoing Investigation of Lubricant Transport within a Powder Metal Compact during Pre-Sinter

Robert Powell[1], Craig Stringer[1], and Stephen L. Feldbauer[2]

[1] The Pennsylvania State University, DuBois Campus, DuBois PA 15801
[2] Abbott Furnace Company, St. Marys, PA 15857

Abstract

Lubricants are a necessary component to the production of parts from powdered metal to assist in the compaction and removal of the part from the compaction die. The added lubricant is then thermally diminished during the initial stage of the sintering process to help achieve desirable microstructure and physical properties of the part. From previous observations, mechanisms were identified in which the lubricant exits the compacted component during the thermal de-lubrication segment. This paper continues previous research that applied metrics to the exit of the lubricant by way of mathematically modeling lubricant pressure and flow using a form of the Rideal-Washburn equation. The data analysis from the previous research is expanded to include additional lubricant data. Several conditions are considered for study including density, lubricant type, temperature, and atmosphere.

Introduction

Due to the high compaction pressure in a conventional press and sinter powder metal process, a lubricant is needed to make this process a production process by reducing die wear and high press forces especially during ejection.[1] The addition of a lubricant for compaction adds an additional step in the sintering process to remove the lubricant so to maintain the desired composition of the part produced.[1] This project is an extension of previous research studying the exit of a lubricant from a powder metal compact which was done in two phases. The first phase was simply observations of the delubrication process and data collection from the process. The second phase consisted of composing a mathematical model and applying metrics to the process of delubrication. Previous research was conducted by analyzing compacted samples of FC-0208 powder which contained 0.75% mass Ethylene Bis Stearamide lubricant having densities of 6.2, 6.8, and 7.0 g/cc. Intentions for this project were to improve our testing apparatus and to reinforce our previous model with additional data. Improvements to the apparatus were conducted by improving the cell that contains the sample. Previously, a clear glass beaker with a foil top was used as the sample containment cell. A new sample containment cell, seen in the apparatus in Figures 1 and 2, was fabricated from steel and has a clear window for viewing the sample and plumbing for atmosphere gas. Additional data was compiled from further study of Ethylene Bis Stearamide and study of an additional multi-component lubricant.

Figure 1: Apparatus

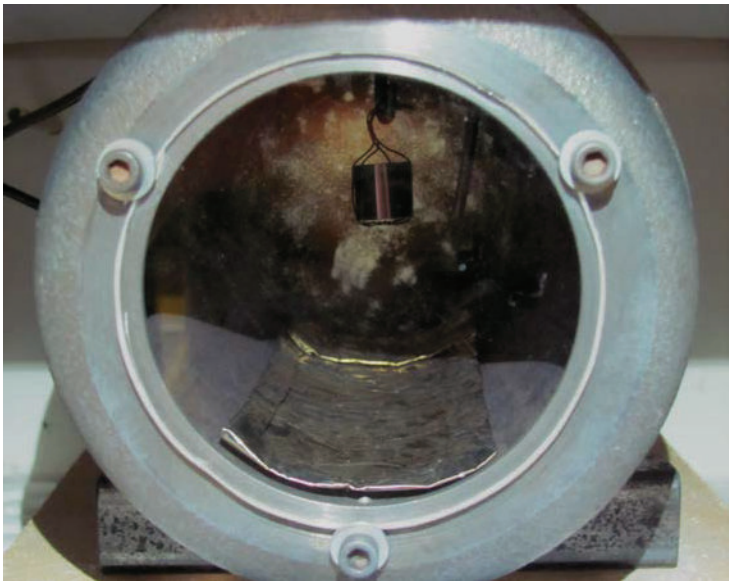

Figure 2: Atmosphere containment cell

Previous research displays the thermal molecular decomposition of the complex hydrocarbon Ethylene Bis Stearamide.[5] A key physical observation from previous observations is the liquid state of Ethylene Bis Stearamide at temperatures above the vaporization temperature. Ethylene Bis Stearamide is commonly referred to as EBS or Lonza Acrawax© C, its trade name, and has a molecular formula of $C_{38}H_{76}N_2O_2$. Figure 3 displays a sample exposed to a temperature of about 572°F (300°C); the liquid flow of the lubricant at a temperature above the vaporization point can be seen.

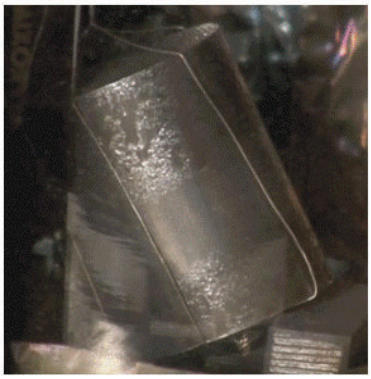

Figure 3: Previous research image;
note liquid state at 572°F (300°C)

From previous research it was observed that density affects the time required to remove the lubricant from a powder metal compact. The density of a green compact determines the average pore size within the compact. Liquid flow within a pore can be considered as capillary flow which is mathematically applicable to the Rideal-Washburn equation and Poiseulle's Law seen in Equations 1 and 2. [2-4]

Equation1: Rideal-Washburn Equation:

$$L = \sqrt{\left(\frac{r\gamma cos\theta t}{2\eta}\right)}$$

L = length traveled to exit part
r = pore radius
γ = surface tension or surface energy
θ = contact angle
t = time
η = viscosity

Equation 2: Poiseulle's Law:

$$\Delta P = \frac{8\mu LQ}{\pi r^4}$$

ΔP = the pressure drop
L = the length of pipe
μ = the dynamic viscosity
Q = the volumetric flow rate
r = the radius
d = the diameter
π = the mathematical constant Pi

Plots of our previous research, seen in Figures 4 and 5, show that our mathematical model resembled the observed data. Figure 5 shows that additional data points would be useful to reinforce the previous research mathematical model. The observed data is nearly linear; this could be due to the data point relationship to the model or possibly that our proposed model is incoherent.

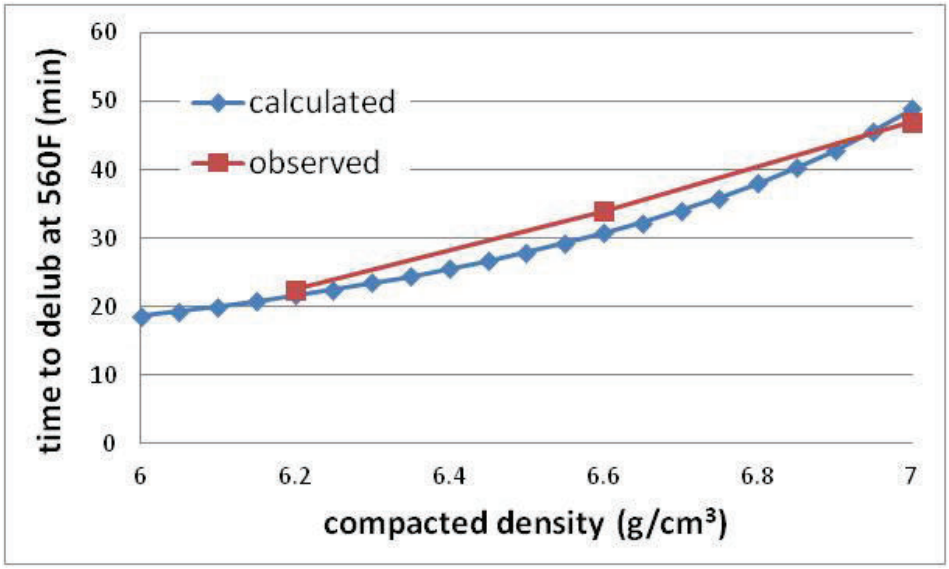

Figure 4: EBS lubricant data: calculated versus observed data

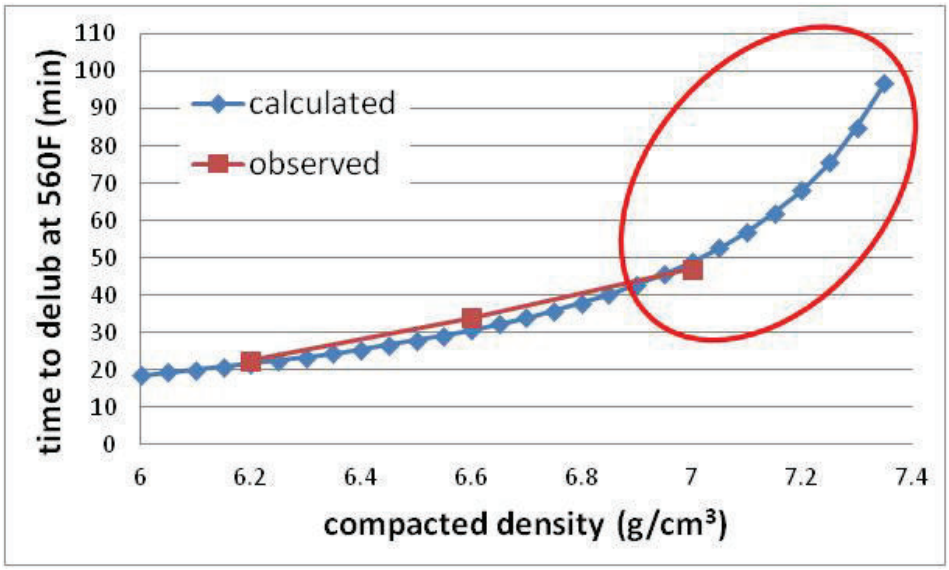

Figure 5: EBS lubricant data: calculated versus observed data, extended plot

Continued research was conducted in the same manner as previous research by using FC-0208 powder, but sets of samples were composed with 0.75% mass EBS lubricant and about 0.25% of an undisclosed, multi component lubricant mixture. EBS samples had densities of 6.2, 6.5, 6.8,

and 7.1 g/cc, while multi component lubricant mixture samples had densities of 6.2, 6.5, 6.8, 7.1, and 7.4 g/cc.

Experimental Procedure

The apparatus was improved by fabricating an improved cell from steel having a glass window thus providing means of improved data collection and observation of delubrication. This improved cell provided improved atmosphere control, more efficient switching of samples, and more ergonomic design for plumbing and scale connection. Figures 1 and 2 show the testing apparatus and new atmosphere cell.

Continuing research was conducted with samples having 6.2, 6.5, 6.8, and 7.1 g/cc densities from FC-0208 powder containing 0.75% mass EBS lubricant and with samples having 6.2, 6.5, 6.8, 7.1 and 7.4 g/cc densities from FC-0208 powder containing about 0.25% mass undisclosed multi-component lubricant. Geometry and mass of the new samples is nearly the same as the previous samples, which is a cylindrical geometry having a diameter of 0.550 inches and a mass of 15.0 grams. A volume to mass ratio was maintained from the previous research samples to the continued research samples.

Experimentation consisted of placing a sample into a Nitrogen atmosphere having a temperature of about 572°F (300°C). The change in mass of the sample over time was recorded. Observations were made through the clear glass cover of the cell.

The final step of the research is constructing the mathematical model of delubrication for both sets of samples as was done in previous research. The mathematical model is based on the Rideal-Washburn equation and Poiseulle's Law seen in Equations 1 and 2 above.

Results

EBS Lubricant Data

For the samples composed from FC-0208 powder containing 0.75% EBS lubricant, it was observed that the lubricant exited every sample in liquid form. For the 6.8 and 7.1 g/cc density samples the liquid lubricant was visibly dripping, while the 6.2 and 6.5 g/cc density samples presented a surface that was simply wet in appearance. The difference in appearance may be attributed to the larger cross sectional pore area of the lower density samples providing an increased rate of evaporation. Plots of the mass loss in respect to time for the EBS lubricant are provided below in Figures 6 and 7. This data is new data conducted in the updated apparatus. Figure 6 displays the change in mass over a time of 40 minutes, while figure 7 displays the change in mass up to the completion of delubrication. Delubrication completion occurs at the inflection point where the graphical plot transitions from mass loss due to lubricant exit, to mass change due to oxidation and de-oxidation, which may be cyclic and somewhat unpredictable. Note that the time for completion of delubrication increases with the density of the samples.

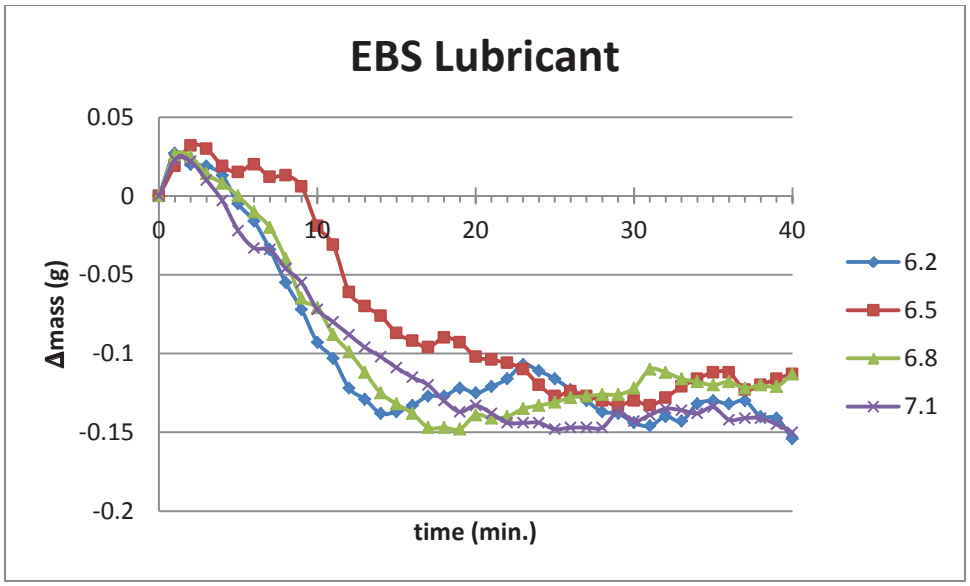

Figure 6: EBS lubricant: mass loss over time, (new data)

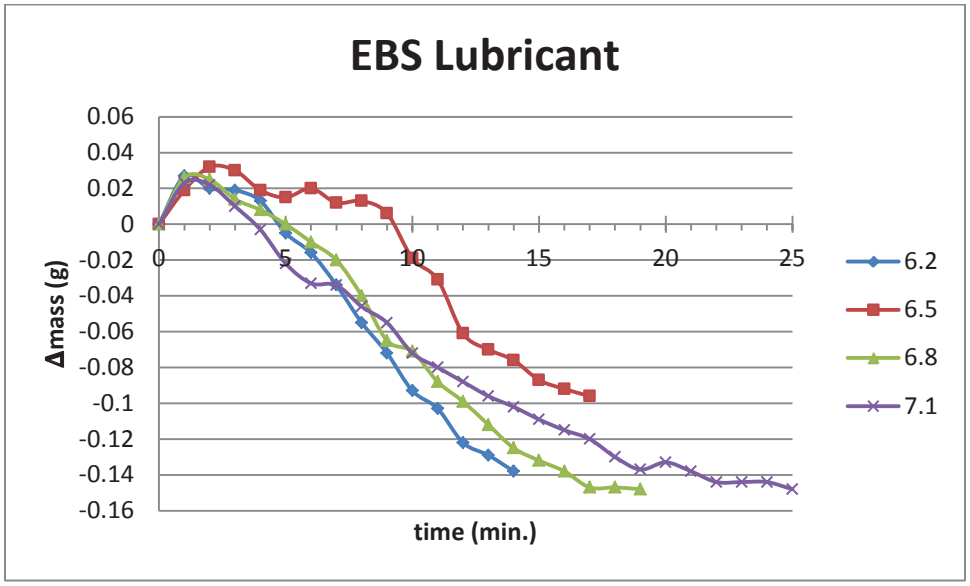

Figure 7: EBS lubricant: mass loss over time cut off at delubrication completion; (new data)

Multi-Component Lubricant Data

An additional lubricant system was studied for this body of research; this lubricant is a mixture of three different composition lubricants. Two of the three lubricant compositions had melting and flash point temperatures below that of EBS lubricant. One of the three lubricants in the mixture has a melting and flash point temperatures above that of EBS lubricant. Samples were composed from FC-0208 powder containing about 0.25% of the multi-component lubricant. The observation of liquid lubricant at the surface was clearly made with 7.4 g/cc densities only.

Delubrication plots for densities below 7.4 g/cc provided more inconsistent data in addition to the absence of visible liquid lubricant. Plots of the mass loss in respect to time for the EBS lubricant are provided below in Figures 8 and 9. Figure 8 displays the change in mass over a time of 40 minutes, while figure 9 displays the change in mass up to the completion of delubrication. Delubrication completion occurs at the inflection point where the graphical plot transitions from mass loss due to lubricant exit to mass change due to oxidation and de-oxidation, which may be cyclic. Note that the time for completion of delubrication increases with the density of the samples.

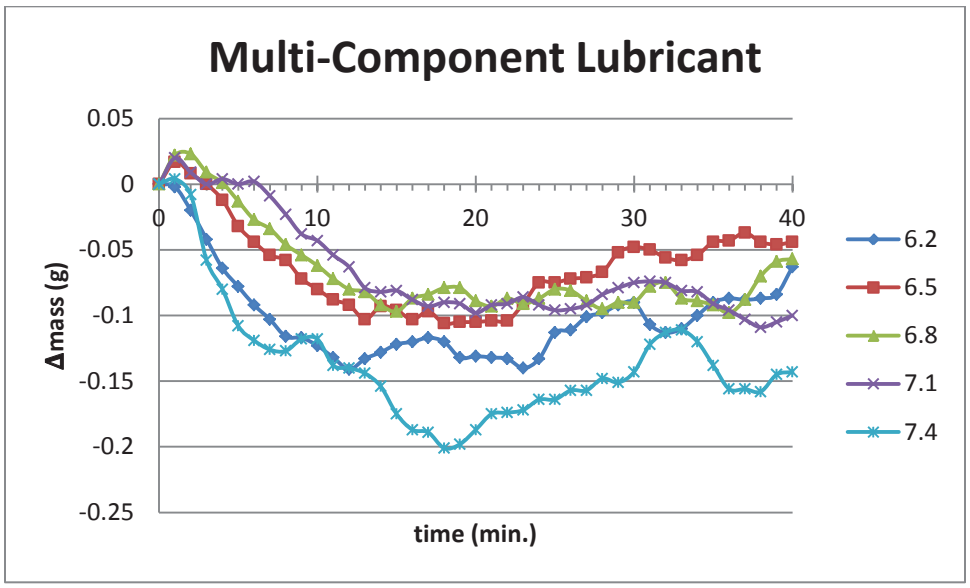

Figure 8: Multi-Component lubricant: mass loss over time, (new data)

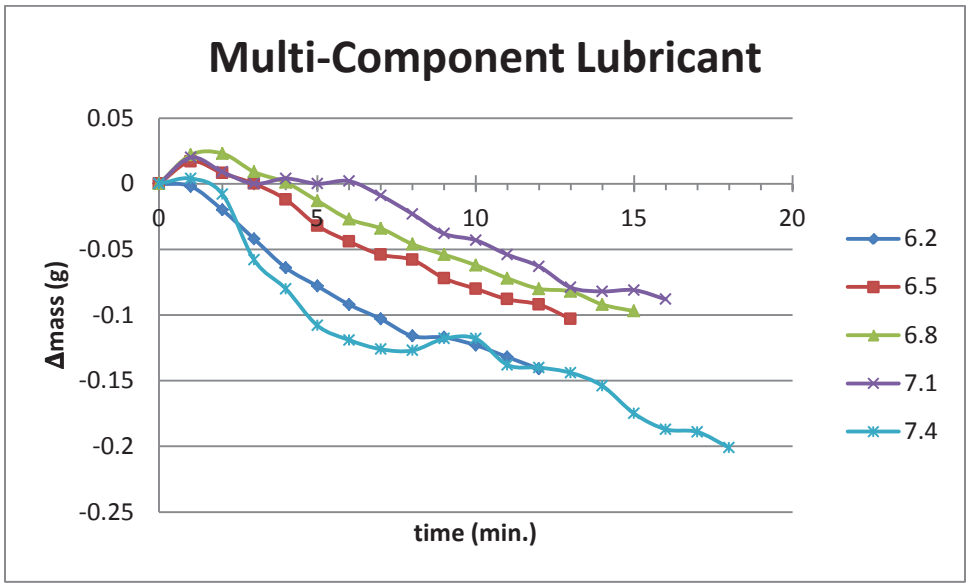

Figure 9: Multi-Component lubricant: mass loss over time cut off at delubrication completion; (new data)

Mathematical Models

An additional consideration that was added to the mathematical model was heat transfer. The last lubricant to be thermally diminished will be the lubricant initially located in the geometric center of the sample, and it will also be the last of the lubricant to be exposed to critical temperatures. Heat transfer to the inner-most lubricant occurs primarily through conduction. Due to powder metal compacts being a porous media, the actual thermal conductivity of the compact will differ from that of the compositional particle material. The thermal conductivity constant will decrease as the density of the compact sample decreases.[6] The time for heat transfer to occur will create a time lag in the delubrication process; this time lag will increase as sample compact density decreases. A time ($t_{T,C}$) for the sample center to reach temperature was estimated by applying the estimated thermal conductivity for each density to a transient heat transfer equation for an internal temperature of 500˚F (260˚C). Effects of constituents other than iron particles were neglected. This temperature falls in between the melting temperature of EBS lubricant and the steady state temperature of the sample. The heat equation utilized is a one term approximate solution (shown in Equation 3 below) derived from the Fourier and Biot numbers which are products of the particular sample geometry, composition, and exposed atmosphere. Mathematical models for EBS lubricant and the multi-component lubricant include the $t_{T,C}$ factor. The nature of the time lag increasing as density decreases, along with the particular sample densities chosen, may also validate the somewhat linear relationship of the previous research delubrication times. Estimated thermal conductivities and delubrication $t_{T,C}$ factors are listed in Figure 10 below.

Equation 3: Infinite Cylinder **(**one term approximation) [6]

$$\theta_o^* = C_1 \exp(-\zeta_1^2 \, Fo)$$

Heat Variables		
Density (g/cc)	Estimated Thermal Conductivity (k_{eff})(W/m·K)	$t_{T,C}$ (min)
6.2	33.80	2.9
6.5	39.46	2.5
6.8	45.50	2.1
7.1	51.97	1.9
7.4	58.91	1.6

Figure 10: Estimated heat variables

The addition of heat transfer to our previous mathematical model equation leads to a new mathematical delubrication model shown below as Equation 4. This equation is derived from Rideal-Washburn equation, Poiseulle's Law, and heat transfer from Equation 3.

Equation 4: Mathematical Model

$$t = \frac{2\eta L^2}{r_h \gamma_{lv} cos\theta} + t_{T,C}$$

L = length traveled to exit part
r = pore radius
γ = surface tension or surface energy
θ = contact angle
t = time
η = viscosity
$t_{T,C} = FoL^2/\alpha$

As seen below in Figure 11, the observed data from the delubrication times of EBS lubricant very closely follow the calculated mathematical model. Data observed for the delubrication of the multi component lubricant do not closely adhere to the calculated mathematical model as seen below in Figure 12. The observation of the absence of liquid at the surface for all densities except 7.4 g/cc and the low melting temperature for two of the three components lead to the hypothesis that the viscosity of the lubricant at the experimental temperature is too low for continuous overall capillary action in the particular pore size, and the delubrication process may be primarily dictated by vaporization. Capillary action does occur for the 7.4 g/cc density multi-component lubricant, as lubricant was observed in liquid form at the surface for all samples tested. Further research of samples having densities of 7.2 and 7.3 g/cc may be beneficial to modeling this particular lubricant as could testing at a lower temperature to increase the viscosity.

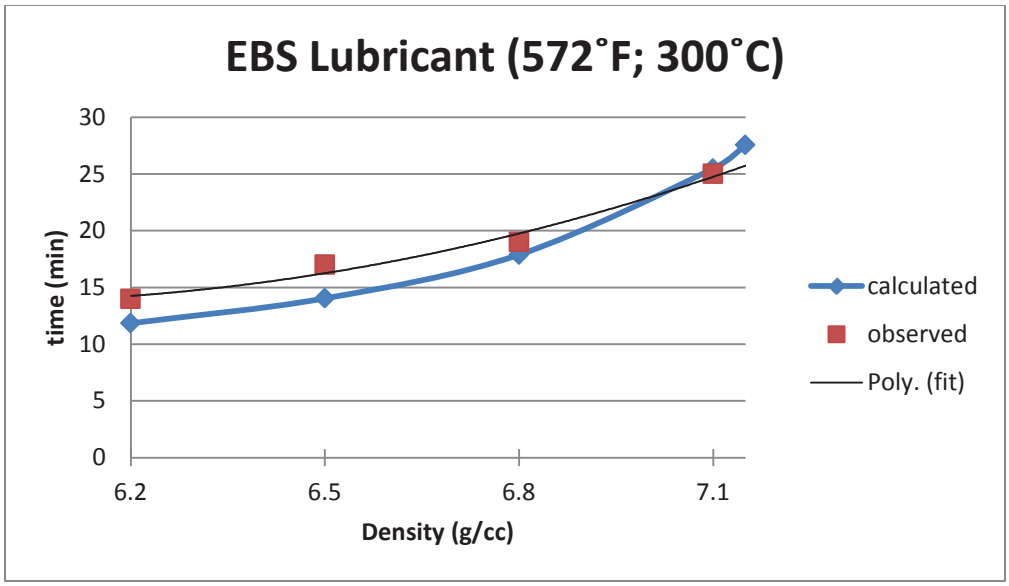

Figure 11: EBS lubricant: observed versus calculated data, (new data)

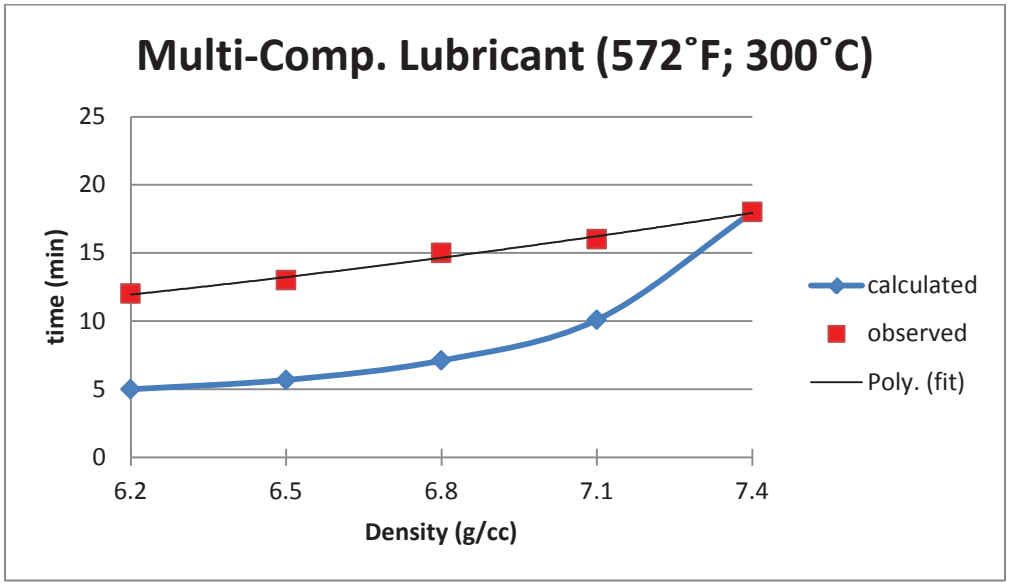

Figure 12: Multi-Component lubricant: observed versus calculated data, (new data)

Conclusion

The average experimental temperature for this continuing body of research was somewhat higher due to the more efficient and improved atmosphere containment cell (seen above in Figures 1 and 2). Delubrication times for the EBS lubricant were also lower for this continuing body of research, which may also be attributed to the improved atmosphere containment cell. This continuing body of research indicates that the mathematical model considering a heat transfer lag closely follows observed delubrication data for EBS lubricant.

Further study of the multi-component lubricant is necessary to compose a sufficient model of the delubrication process of this lubricant. The further study may examine sample densities between 7.1 and 7.4 g/cc, a lower experimental temperature, and or more in depth study of the lubricant mixture properties.

A mathematical model of EBS delubrication at varying temperature/viscosities is shown below in Figure 13. The model shown in Figure 13 is valid for an iron based powder part with cylindrical geometry that has a 0.275 in. (6.985 mm) radius that is larger in magnitude than half of the cylinder height. A mathematical model of EBS delubrication from a part having infinite cylinder geometry of varying radii is shown in Figure 14. Atmosphere temperature is constant 580˚F (304˚C) as is the composition of a base iron powder. Both models in Figures 13 and 14 are for testing in a nitrogen atmosphere.

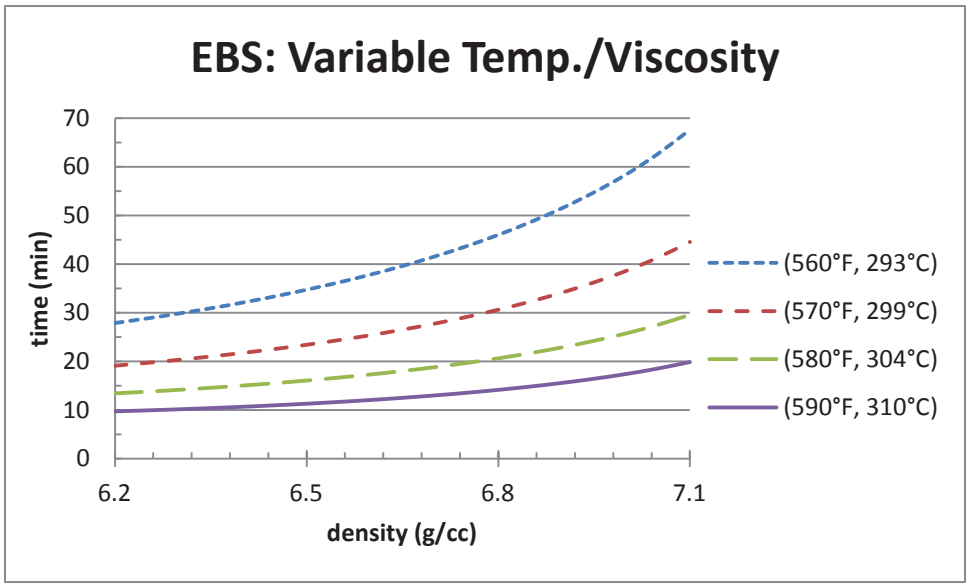

Figure 13: Mathematical Model, EBS lubricant, infinite cylinder 0.275" radius, iron base powder

Future Work

The mathematical model for EBS lubricant appears valid. Adding additional variables to the model will increase its value and versatility. Performance of the multi-component lubricant displayed a critical temperature-viscosity-pore size relationship for modeling delubrication. Further investigation of the multi-component lubricant properties is necessary to develop a working model. This lubricant performs well at high density, which it was designed for, and utilizing data points at 7.2 and 7.3 g/cc densities may aid in composing a mathematical model.

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