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SPATIAL DISTRIBUTION OF ALUMINUM PARTICLE COMBUSTION IN ROCKET PROPELLANT PLUMES AT ATMOSPHERIC PRESSURE

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ABSTRACT

The aluminum combustion data gathered from propellant plumes provides insight into the behavior of aluminum particles leading to more complete combustion models. Fifteen plume particulate samples were obtained from the charge centerline at various elevations above the burning surface on six, twelve and twenty inch diameter propellant charges. Each sample was gathered on a linearly actuated copper coupon that remained in the propellant plume for five seconds. Once removed the next higher sample was inserted. The sample coupons were imaged with a scanning electron microscope and analyzed for aluminum content via differential scanning calorimetry in an ultra high purity nitrogen atmosphere. A temperature profile consisting of a 10°C per minute ramp beginning at 300°C and ending at 800°C was used to ensure the total melting of elemental aluminum in each sample. The amount of energy required to melt the sample aluminum is compared to a set of known energy values yielding the actual sample aluminum mass. The DSC analysis technique can successfully determine the aluminum content present in each sample and the results indicate an increase in burned aluminum as the distance from the burning surface increases.

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INTRODUCTION

The spatial distribution of aluminum particle combustion in the plume of rocket propellant charges accounts for much of the heat flux incident on objects in and around the burning region. Combustion modeling and experimentation is used to understand the distribution and resultant impact on vital components and payloads in accident conditions. The aluminum content data gathered from propellant plumes burning at atmospheric pressure provides insight into the behavior of aluminum particles leading to more complete combustion models for accident conditions. This paper documents a study to determine the composition of samples taken from the plumes of three different propellant fires.

Fifteen plume particulate samples were obtained from the charge centerline at various elevations above the burning surface for 6", 12", and 20" diameter propellant charges burning in air at atmospheric pressure. Each sample was gathered on a linearly actuated copper coupon that remained in the propellant plume for five seconds. Once removed the next higher sample was inserted. The sample coupons were imaged with a scanning electron microscope and analyzed for aluminum content via differential scanning calorimetry in an ultra high purity nitrogen atmosphere. The amount of energy required to melt the sample aluminum is compared to a set of known energy values yielding the actual sample aluminum mass. The DSC analysis technique can successfully determine the aluminum content present in each sample and the results indicate an increase in burned aluminum as the distance from the burning surface increases.

EXPERIMENT DETAILS

Recently NASA JPL funded a battery of aluminized upward rocket propellant burns that were performed at Sandia National Laboratories. Test procedures and equipment setup are documented in this paper. Figure 1 shows the test setup. The actuators were bolted to a tower next to the burn table and covered with insulation. Plume particle samples were obtained using five copper coupons for each propellant burn. The coupons were made of 3" x 3" x 1/8" thick copper plate and attached to the ends of pneumatic linear actuators located 6", 11", 19", 34", and 64" above burn table. These elevations located the coupons at 2", 7", 15", 30", and 60" above the initial propellant burning surface.

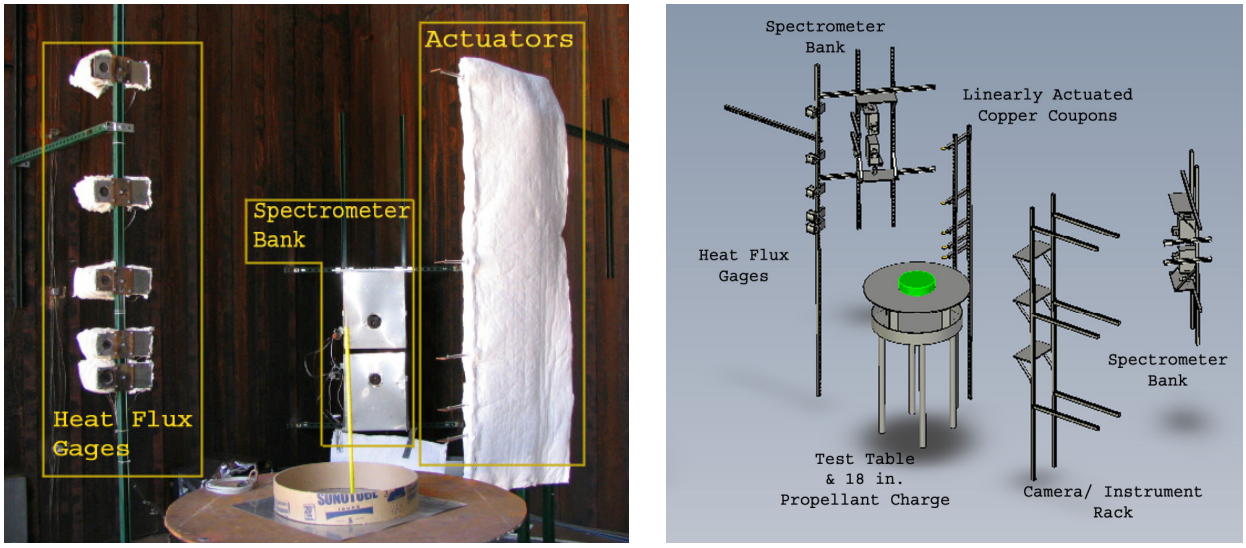

Figure 1: Experimental test setup

Each coupon was extended to the center of the plume when the actuators were activated, held in the flame for five seconds, and then retracted from the flame. Starting from the bottom (closest to the propellant), they were activated every five seconds, beginning forty seconds after ignition of the propellant. Samples were collected from the burning of 6", 12" and 20" diameter propellant charges. Figure 2 shows the coupon and attached substrate following the burn completion. The following is an overview of the analysis of those samples specifically exploring the quantities of elemental aluminum and aluminum oxide present.

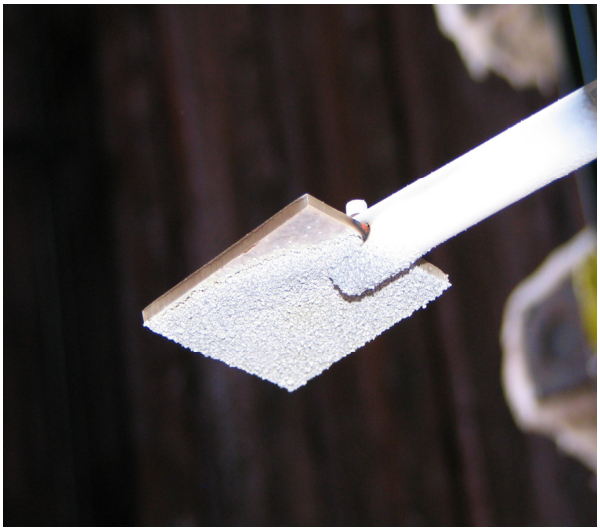

Figure 2: Copper coupon after sample collection

SCANNING ELECTRON MICROSCOPE ANALYSIS

Initially the plume particle samples were analyzed using a scanning electron microscope (SEM) and energy dispersive spectrometer (EDS). Figure 3 is an example of the information obtained. A photograph of the sample surface is shown on the left along with the quantities of identified elements relative to established counts for aluminum. The analysis revealed the surface condition of each sample but results detailing the amount of elemental aluminum and oxygen present in each sample were not useful for determining the total aluminum and aluminum oxide contents. The inability to determine aluminum and aluminum oxide content is attributed primarily to the unknown depth into each sample that the SEM detects the selected elements. This analysis technique provides better understanding of the surface condition of each sample and allows for easier visual comparison of different samples. There are small quantities of copper, carbon and phosphorus present but should not adversely affect the results of subsequent analysis. Figure 4 shows the wide variation in surface condition of four samples.

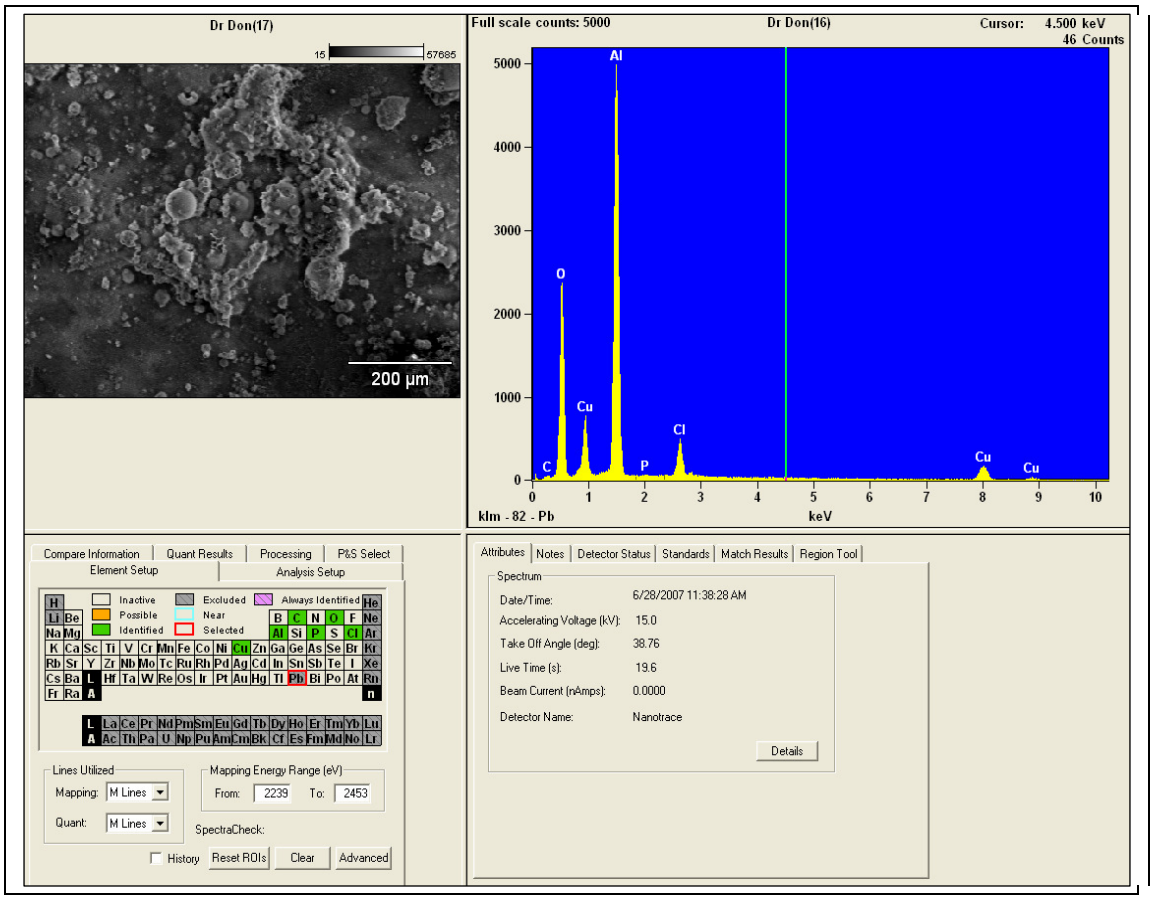

Figure 3: SEM / EDS details

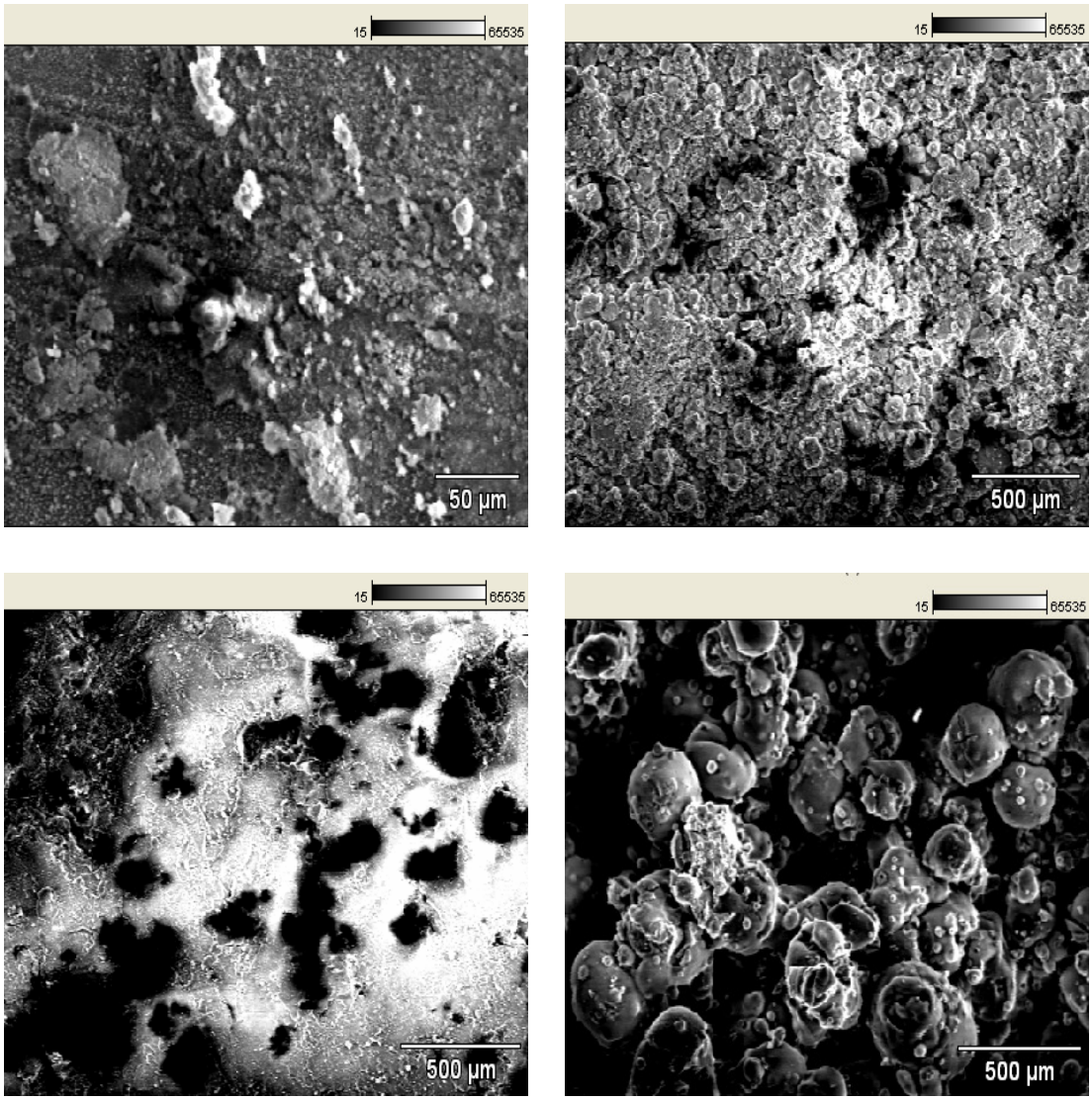

Figure 4: SEM surface sample comparisons

After the SEM images were taken, samples were scraped from the surface of each copper coupon and weighed. Each sample was analyzed using differential scanning calorimetry in ultra high purity nitrogen atmosphere to determine the quantities of aluminum and aluminum oxide in each sample.

DIFFERENTIAL SCANNING CALORIMETRY ANALYSIS

Differential scanning calorimetry is a technique where a sample is heated in an electric furnace and compared with a known standard while the number and magnitude of sample phase changes are observed. The

phase changes are then correlated with the energy flow into and out of the furnace at corresponding temperatures and element and compound details can be calculated. The plume samples are a heterogeneous mixture of aluminum and aluminum oxide and are separated by melting the aluminum component in a process similar to that performed by White et. al. [1]. Aluminum phase changes are identified and compared to phase change data taken using 99.99% pure aluminum control samples. The melting temperature of aluminum is much lower than that of aluminum oxide therefore the sample is heated ensuring all of the pure aluminum in the sample melts. The amount of aluminum in the sample is calculated by tracking the energy required to melt the pure aluminum and then correlated to the melting energy of aluminum control samples. Performed on the Netzsch STA 409PC Luxx DSC/TGA, each sample is held for a 5 minute initial phase at 300°C allowing the instrument to stabilize. The secondary phase consists of a 10^{o} C/min temperature rise from 300 to 800^{o} C which contains the melting point of aluminum at 660^{o} C. Figure 5 shows an endotherm describing the energy flow required to keep the furnace on the established temperature profile while the sample undergoes phase changes. The temperature profile is shown in solid blue and the system response (mW) to phase changes is dotted pink.

The solid to liquid phase change of aluminum can be seen as the large dip in the endotherm in Figure 5 and isolated in Figure 6. The equation of a line, in blue, approximating the path of the endotherm, assuming aluminum heat of fusion does not occur, is used to connect the onset and offset of the actual aluminum melting endotherm. The area conscribed by the endotherm and linear approximation is calculated using the trapezoid method and represents the energy required to melt the aluminum in the sample.

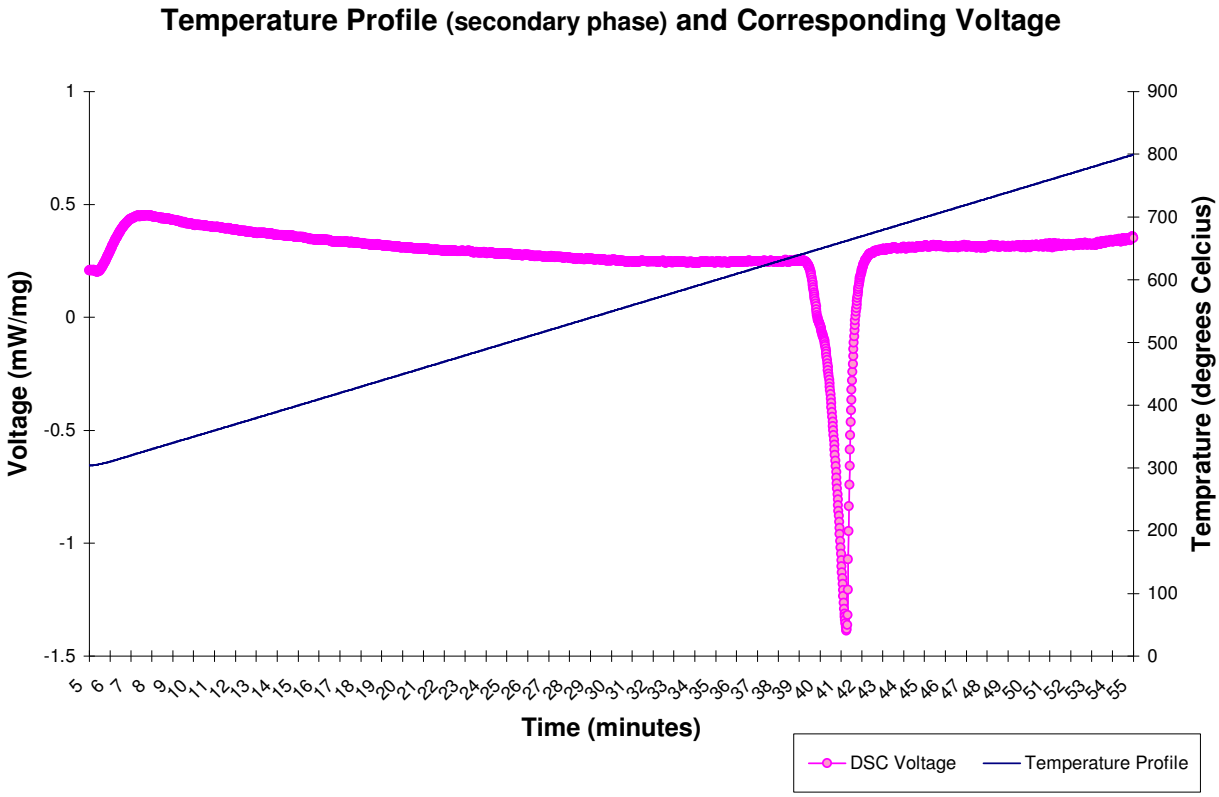

Figure 5: DSC voltage representing heat flow into and out of sample and furnace temperature profile

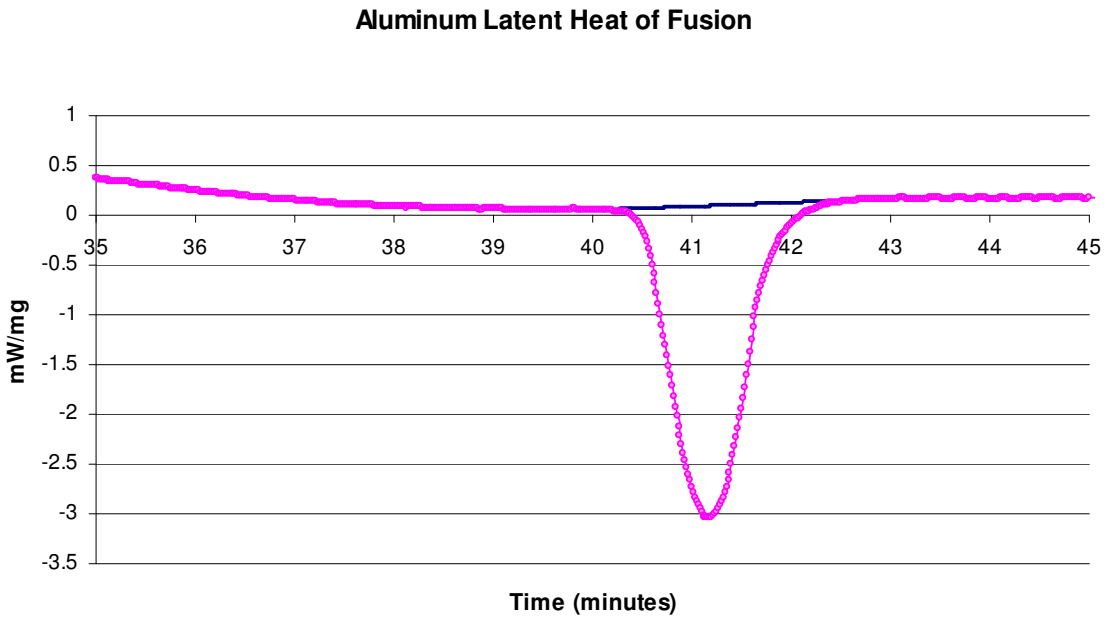

Figure 6: Aluminum heat of fusion endotherm with linear approximation

The calculated area is compared to a calibration curve of sample mass versus area which indicates the amount of aluminum present in each sample. Six separate 99.99% aluminum wire samples of various weights were tested to provide the aluminum calibration curve shown in Figure 7. It was calculated that 1 mg of aluminum requires approximately 190 μV-s to melt. This value can vary by the standard deviation of 20.87 μV-s therefore; the amount of unburned aluminum in each sample can vary by approximately 11%.

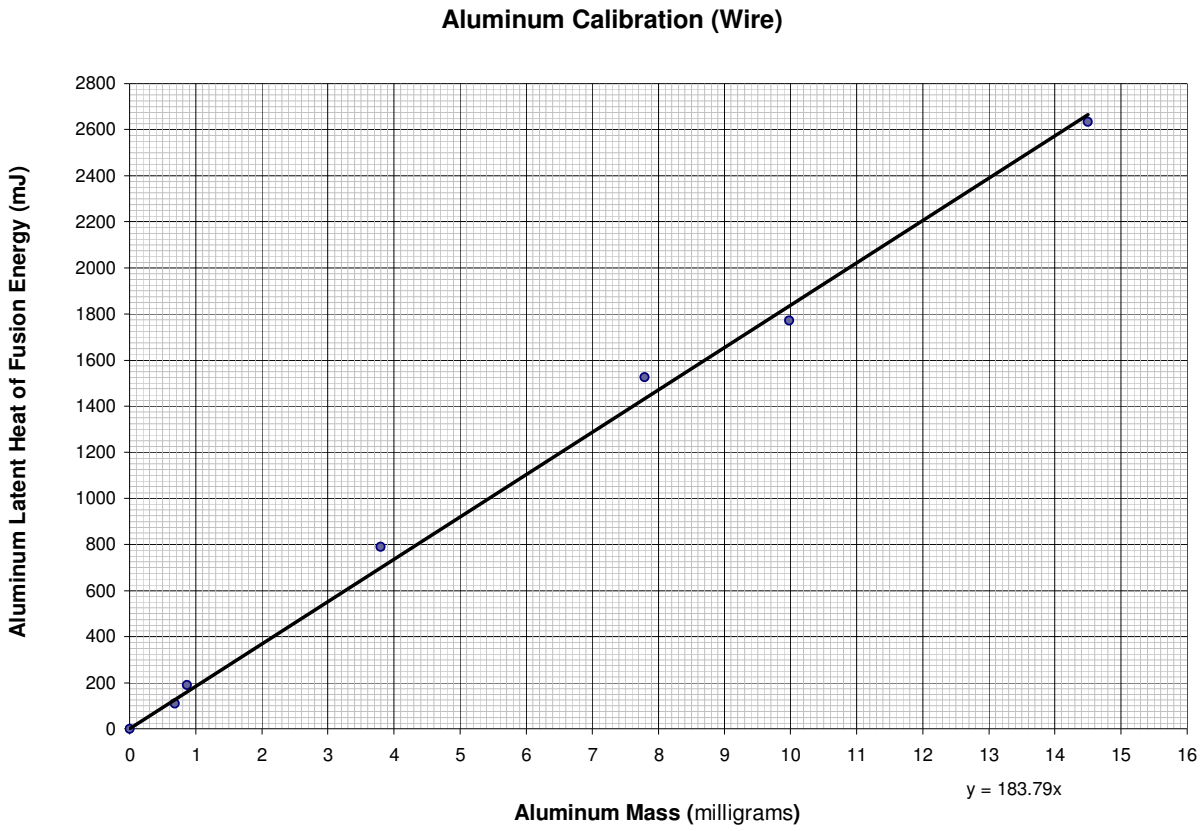

Figure 7: Aluminum calibration

RESULTS AND CONCLUSIONS

The results are shown in Figure 8. Overall the general data trend indicates a decrease in aluminum content (and corresponding increase in aluminum oxide content) as the distance from the burning surface increases. This result is expected from previous atmospheric combustion of propellants of this. It indicates that combustion of the aluminum is slow compared to the binder and continues for a number of diameters down from the charge.

A possible exception appears to be near the surface of the propellant. The results indicate that the coupon 2" above the burning surface yielded a lower percentage of aluminum particle deposition whereas the percentage of

aluminum deposited on the coupon at 7" has increased for both the 6" and 20" diameter charges and most like occurs in the 12" charge but is unconfirmed due to pneumatic actuator malfunction. There are several theories that may explain why this phenomenon is occurring. One is that the uncertainties in the experiment are greater than the data variance shown in Figure 8 due to potential bias errors in the experiment. The potential sources of bias include an assumption that the forty seconds from ignition to insertion of the first coupon allows the burning propellant to reach a quasi-steady state where the rate which molten aluminum is convected away from the burning surface is constant. Secondly, minor variations in the location of the coupon center relative to the vertical centerline of the propellant charge may expose the coupon to different radial environments. If it exists, this variation in location would be consistent for each charge size and may explain the indicated increase in unburned aluminum between the 2" and 7" elevations.

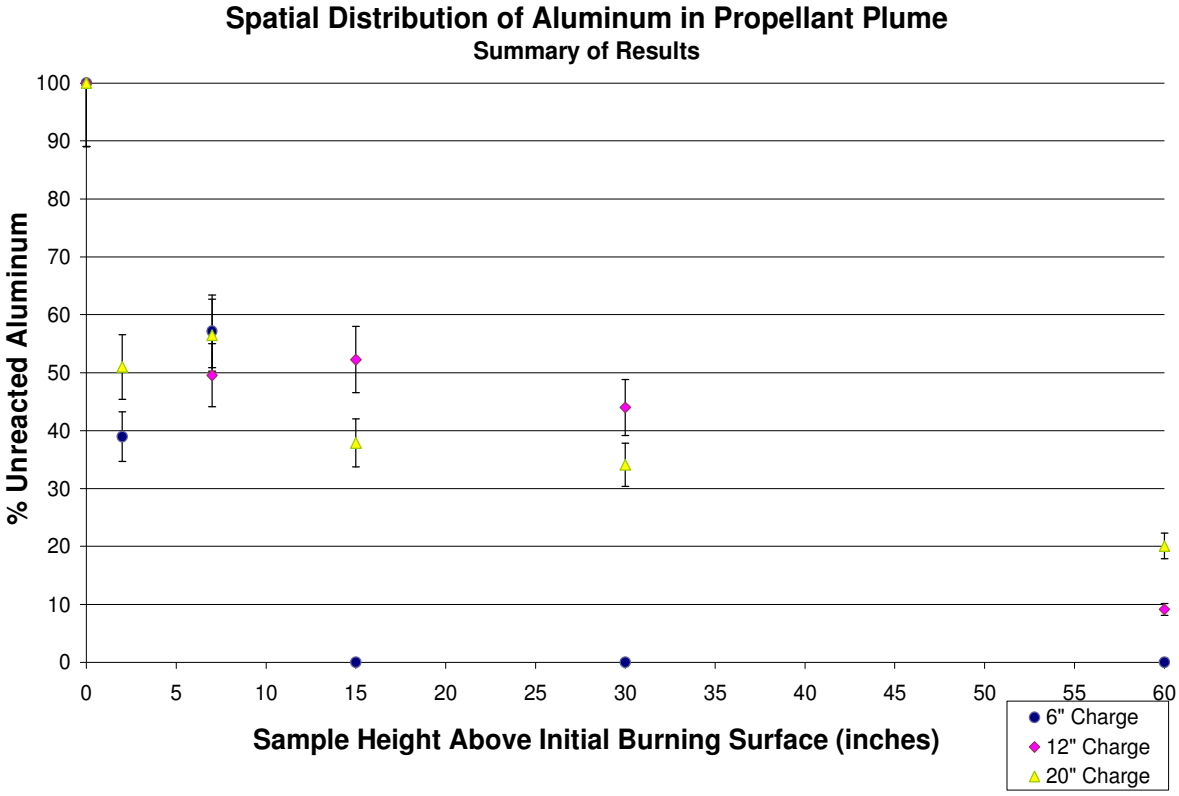

Figure 8: Test matrix results of % unreacted aluminum vs. elevation above burning surface

Possible physical explanations of the increase in aluminum between 2" and 7" elevations are that unburned aluminum particles are being entrained deeper into the burning region, increasing the aluminum particle density at the flame core then impacting the coupon. Also, there is a possibility that some aluminum oxide either impacts the

coupon and does not remain attached or never impacts the coupon but is drawn higher into the plume by the flow field creating the perceived decrease in aluminum oxide (i.e, the aluminum preferentially sticks but the aluminum oxide bounces at some velocities or material temperatures) between the 2" and 7" elevations. Secondly, minor variations in the location of the coupon center relative to the vertical centerline of the propellant charge may expose the coupon to different radial environments. If it exists, this variation in location would be consistent for each charge size and may explain the indicated increase in unburned aluminum between the 2" and 7" elevations. Future experiments will address this phenomenon. Overall the general data trend indicates a decrease in aluminum as the distance from the burning surface increases.

Sample Height (inches above the initial burning surface)	**% Al**	**% Al2O3**
Test Matrix 3: 6" diameter		
Surface	100	0.00
2	40.38	59.62
7	59.24	40.76
15	0.00	100.00
30	0.00	100.00
60	0.00	100.00
Test Matrix 2: 12" diameter		
Surface	100.00	0.00
2	Actuator malfunction	Actuator malfunction
7	49.55	50.45
15	52.25	47.75
30	43.98	56.02
60	1.03	98.97
Test Matrix 1: 20" diameter		
Surface	100.00	0.00
2	50.99	49.01
7	56.46	43.54
15	37.87	62.13
30	34.09	65.91
60	20.06	79.94

Table 1: Percentage of aluminum and aluminum oxide present on coupons at indicated heights and charge diameter

REFERENCES

1. White, R., et al., "Thermal Analysis of Aluminum Particle Combustion in a Simulated Propellant Flame", submitted to *Journal of Propulsion and Power*, 2006.