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Expansion Behavior and Temperature Mapping of Thermites in Burn Tubes as a Function of Fill Length

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Abstract: The reaction of loosely-packed aluminum/copper oxide (Al/CuO) thermites in a 12 cm long acrylic burn tube was investigated as a function of the fill length from 2 to 10 cm. The velocity of the luminous front was measured both in the filled and unfilled region, and approached 1000 m s^{-1} in the unfilled region, independent of the fill length. This value is approximately a factor of two higher than the fastest velocity measured in the filled region, 606 m s^{-1}, for the 10 cm filled tube. The velocity increase in the unfilled region ist likely due to the increased open porosity, which can support faster flow velocities for a given pressure gradient relative to that in the porous material. A high-speed color imaging pyrometer was used to thermally map the evolution of the flame in both regions. Near the luminous front in the filled section, the temperature was observed to rapidly increase in a 1–2 cm zone to a maximum value near 3200 K, and an average value near 3000 K was sustained in the wake well after the front passes and exits the tube. In partially-filled tubes, even for the lowest fill length of 2 cm, the intermediate and/or product species could be seen to expand forward and completely fill the tube with a sustained temperature of ca. 3000 K. This temperature did not decay during the expansion, suggesting that the material continues to react as it expands. The results raise several questions about what a burn tube experiment is really measuring; such as what fraction of the material has burned when the luminous front passes and what role open cracks or microstructures play in promoting transport. These questions are critically important towards developing a predictive capability for confined, loosely packed deflagrations.

Keywords: Al-CuO thermite · Imaging pyrometry · Energetic materials · Burn tube

1 Introduction

Thermites are mixtures of a metallic fuel (i.e. Al, Si, Ta) with a metal oxide (i.e. CuO, Bi_2O_3, MoO_3, Fe_2O_3) oxidizer. Nanocomposite thermites are similar formulations in composition, but utilize at least one constituent having nanometric dimensions. The underlying idea behind using fine particle sizes has been to improve the performance by reducing the mass transport length scale between the constituents, thus decreasing the kinetic time scale of the oxidation/reduction reaction.

Thermodynamically speaking, certain formulations are very attractive, as they can have higher mass and/or volumetric energy densities than many common energetic materials. [1] However, in order to further improve the kinetics, the reaction mechanism needs to be understood. One common technique to evaluate the mechanism has been to loosely pack the material into a channel or burn tube, and then ignite the material on one end and observe what parameters can impact the self-propagating flame velocity. Several researchers have observed that gas production is more important than temperature in governing the flame velocity, and have suggested that convection of gases is important to the energy transport. [2–5] There has also been some discussion of particle advection being important to consider in certain configurations. [6–8] Pantoya et al. [9] have shown that the velocity scales inversely with packing density in a confined geometry. In recent work, Parimi et al. [10] have demonstrated the flame velocity can be dramatically improved through the use of large, ordered microstructures in unconfined experiments using porous silicon as a fuel. All of these examples demonstrate that the energy transport is an important process to consider, and may be more important than the kinetic reaction time scale in governing the flame velocity.

The formation and propagation of a flame in a thermite is a highly complicated and multi-scale process. This involves an ignition event, followed by kinetics as the fuel and oxidizer undergo oxidation and reduction. The intermediate and/or product gases and particles are transported, resulting in a rapid heating environment, which serves to propagate the energy and begin the process over again. Experimentally probing the kinetics or energy transport is

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challenging, especially in-situ and with an appropriate spatial and temporal resolution. Most investigations of thermites involve loosely packed systems, from approximately 5–15% of the theoretical maximum packing fraction. While working with less material is beneficial from a safety and cost perspective, so much open porosity can really complicate the analysis of the results. For example, stoichiometric Al/CuO yields 1225 $cm^3 g^{-1}$ thermite of gases according to a CHEETAH [11] equilibrium calculation. At 14% packing density in a 3 mm ID tube (this work), 1 cm of material contains about 50 mg of thermite. If fully reacted, this amount of material would be able to fill a 62 cm long tube. Thus, in loosely packed systems it's not clear how these gases, along with entrained particles, could flow through the porous networks and, more importantly, how this could affect our interpretation of a "flame" position. Understanding this is important to resolve critical questions, such as what fraction of material has reacted when the luminous front passes, and are such rapid deflagration velocities indicative of fast kinetics, or are hot gases and particles simply being expanded at high velocity through the highly porous material and complicating our interpretation of the flame position?

In this work we examine the effect of fill length on the propagation velocity of the luminous front in loosely packed Al/CuO thermites reacted in an acrylic burn tube. We compare and contrast the velocity in the filled vs. unfilled region. Furthermore, we adapt a recent advancement in color-camera pyrometry [12] to spatially and temporally resolve the temperature field during the deflagration, and again examine this in the filled vs. unfilled regime to further characterize the luminous region. Whiles some authors have previously used emissivity to predict an average temperature during thermite reactions [13–15], these techniques do not provide much, if any, spatial resolution of the temperature field.

2 Experimental

2.1 Sample Preparation and Velocity Measurements

The thermite formulation chosen for this work was aluminum/copper oxide (Al/CuO). Instead of using nanometric Al, a larger size of 3.5 μm was used (H-2, Valimet corp.). Past work has shown that the dimensions of the fuel are less important than the dimensions of the oxidizer [4], and this was verified in a recent article by Sullivan et al. [16], where it was found that micrometer-sized Al reacted as well, if not better than nano-sized Al, so long as the oxidizer was kept nanometric. Nano-Al has a much higher percentage of Al_2O_3 from the passivation shell, is aggregated in morphology, and may have adsorbed organic species present on the surface. All of these factors may complicate the analysis, and so we chose the larger, spherical μ-Al for this study. The CuO was purchased from Sigma Aldrich, and had a primary particle size < 50 nm, as specified by the supplier.

To mix the materials, stoichiometric quantities of the fuel and oxidizer (2Al:3CuO molar ratio) were weighed and added to an ESD conductive polypropylene container (59 mL size, LA container, Inc.). The total mass of the powder was 1 g. The container was sealed and mixed using a resonant acoustic mixer (LabRAM, Resodyn Corp.) at 100 g acceleration for a total of 2 min. It was found that mixing for longer did not further increase the reactivity. It should be noted that our samples were mixed dry, but we do not advocate this due to the ESD sensitivity. Acoustic mixing using liquid processing agents has recently been investigated by Nellums et al. [17]

The mixed thermite powder was loaded into a 3 mm ID × 6 mm OD × 12 cm long acrylic tube, and the tube was tapped constantly during loading. This resulted in a packing density of approximately 0.71 $g\,cm^{-3}$, or 14% of the theoretical maximum packing density of 5.1 $g\,cm^{-3}$ for this formulation. The filled tube was inserted into a secondary thicker acrylic tube, which was bolted onto a heavy base plate. This second tube holds the burn tube in place during the reaction, and provides additional confinement for increased safety. In addition to filled tubes, we examined the effect of tube fill length. The tube was filled to various lengths from 2 to 10 cm, and by changing this length we could compare the reaction event within the material, as well as the expansion event as energy flowed down the open tube. An image of filled tubes with two different fill lengths (10 cm and 4 cm) is shown in Figure 1. To ignite the material, a nichrome wire was bent and inserted ca.1 cm into one end of the tube and connected to a DC power supply.

Upon ignition, the resulting propagation event was imaged using a high-speed camera (Vision Research Phantom 12.1) with a 55 mm Nikkor lens. Test images were taken to determine the effect of the curved tube on images. A slight fish-eye effect was noticed in the vertical direction. This effect was not in the horizontal direction and had not effect on the velocity measurements. Data collection was manually triggered for each shot. The camera's 12.8 cm by 0.3 cm field of view required 1200 by 32 pixels, setting the maximum frame rate at 140,000 frames per second. The exposure time and lens f-stop were varied to avoid either overexposing or underexposing the image. Distance – time plots (*x*-t) of the luminous front, commonly referred to as the flame front, were plotted to obtain velocity measurements. The best practice for measuring velocity

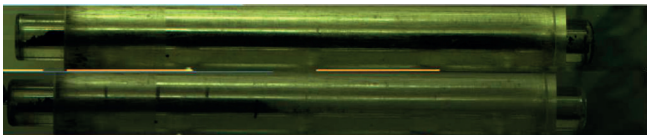

Figure 1. Burn tubes, 10 cm fill length (top), and 4 cm fill length (bottom).

is to take data from a large distance during the steady wave propagation. However, since the fill length was varied, and since the flame can accelerate early in such experiments [5, 18], data could not always be averaged over large distances and thus was taken from shorter distances, accordingly. This will present some uncertainty in the reported velocities, particularly for low fill lengths.

2.2 Imaging Pyrometry

Temperature measurement in flames and energetic materials is a challenging problem based on the physical nature of the problem. Common methods such as thermocouples are intrusive and disturb the phenomena under study. Traditionally optical pyrometry, which relies on thermal radiation, has been used to measure the temperature of flames. [19] The most straightforward, but most tedious, method utilizes the flame brightness. The flame brightness is compared with a calibrated blackbody source to determine the flame temperature. The pyrometer setup and configuration must be identical between the calibration measurement and experiment. Additional, this method requires knowledge of the emissivity, which is normally not known.

A second and more common method is the ratio method. [12, 20] In this technique the object's radiation is measured in multiple discrete bands. The ratio of the radiation in the discrete bands is a function of the temperature of the object. This method has the added benefit that the emissivity does not need to be known. The only assumption on the emissivity made is that it must be wavelength independent, or at least a known scaling with wavelength. This is the most commonly used method to measure the temperature of thermite flames, hydrocarbon flames, and post detonated explosives.

The implementation of the ratio method is typically done using multi-mode fiber optics coupled to wavelength-filtered high-speed diodes. In the temperature range of flames, visible region, silicon diodes are sufficient for detectors. The optical fibers in these pyrometers collect all the radiation within the fibers field of view, effectively performing a spatial integration of the radiation. Since the radiation intensity is proportional to the fourth power of temperature, high temperature regions contribute the most to the signal and lower temperature regions are neglected.

We have used a high-speed color camera to spatially resolve the temperature of the flame as it transverses the tube. The technique has been described in much more detail elsewhere [12], but a brief description is as follows. Color imaging is done with a silicon complementary metal-oxide semiconductor (CMOS) device that is sensitive to light with a wavelength between 300 and 1100 nm. The sensor is a two dimensional grid of pixels. Above each pixel is a single color filter, alternately arranged in a Bayer pattern. The information from the Bayer pattern can be reconstructed to obtain a full color image. In a full color image each pixel has three color values representing the light from each color filter in the Bayer pattern. The three color filters used for digital cameras span the visible regions between 400 and 650 nm. The color filter transmission is normally published by the camera manufacturer, and can be measured using a standard light source and monochromator. The measured and published transmission data are generally in good agreement. The filter transmission is broadband with approximately 100 nm bandwidths. While the filter is not ideal for temperature measurements, it provides enough sensitivity in the temperature range of typical energetic material flames to perform either two or three color pyrometry at each pixel group. In nonhomogeneous flames with large temperatures gradients over small areas the image reconstruction using the Bayer pattern may introduce uncertainties in the temperature measurement. Measurements done at two different spatial resolution determined that the uncertainty due to the image reconstruction is generally less than 50 K.

In our analysis we assume that the light behaves as a gray body. That is to say, the emissivity is independent of wavelength. A more robust measurement may be performed if detailed knowledge of the temperature and wavelength dependence of the emissivity is known. The wide bandwidth and spectral overlap of the color filters help mitigate the uncertainty introduced due to the gray body assumption.

The ratio of the color values (e.g. green and red) is compared with an analytical curve, which is produced using the Planck equation, $L(\lambda,T)$, and the filter transmission, $\chi_G(\lambda)$,

$$\frac{\text{green}}{\text{red}} = \frac{\Psi_G \int L(\lambda,T)\chi_G(\lambda)\mathrm{d}\lambda}{\Psi_f \int L(\lambda,T)\chi_f(\lambda)\mathrm{d}\lambda}$$

where Ψ_G is the channel internal gain. Since the internal gain is not known it is wrapped in a calibration factor that is measured by imaging a standard blackbody source. The calibration curve and data for the camera used in these ex-

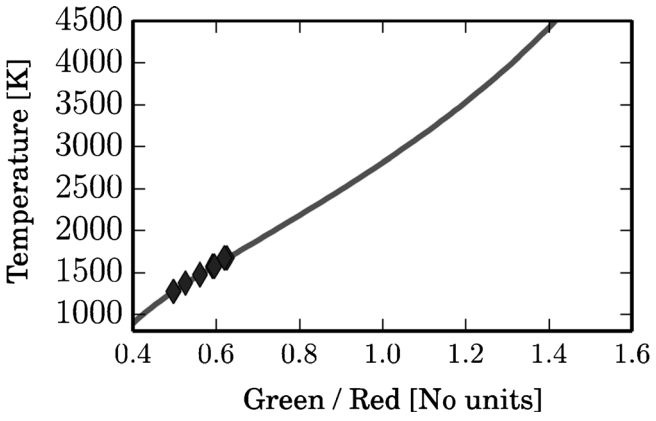

Figure 2. Analytical calibration curve (solid line) and data (diamonds) for the imaging pyrometer.

periments is shown in Figure 2. Calibration data were collected up to 1700 K. All three color ratios were in excellent agreement with the analytical curve after taking into account the calibration factor. Calibration data were also collected with a polycarbonate window between the blackbody source and camera. The polycarbonate window had no noticeable effect on the measurements.

3 Results and Discussion

3.1 Velocity of Luminous Front

The *x-t* plot for all fills is shown in Figure 3. Since the ignition time for each flame is not precisely known, the time has been normalized at the frame before the flame exits the tube. The flame position in the last frame varies by 0.3 cm. The *x-t* plot represents the velocity (inverse of the slope) at all points along the tube in both the filled thermite region (referred to here as the combustion wave) and free air region (referred to as the pressure or expansion wave). Table 1 shows the average velocity of the luminous front in both regions, and as a function of the fill length. In general, the combustion wave velocity was linear in the first few cm, then accelerates before approaching another linear velocity region. For any fill length, the wave velocity is seen to significantly increase once the end of the thermite is reached and the wave expands into the free air regime. This expansion increases the observed velocity, which, interestingly, approaches an asymptotic value of close to 1000 $m\,s^{-1}$ in all cases.

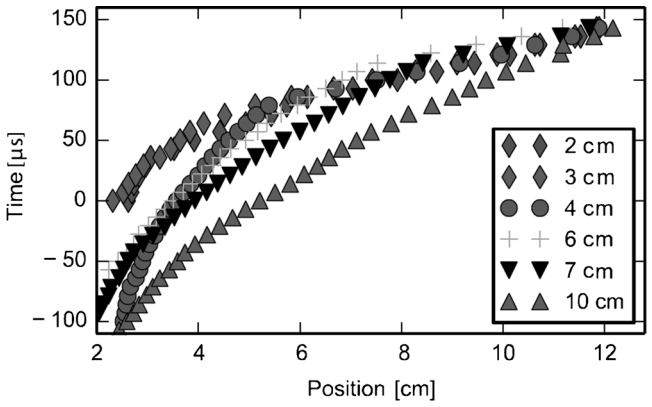

Table 1. Combustion and pressure wave velocity for all fill factors.

Fill length	Combustion wave velocity [$m\,s^{-1}$]	Pressure wave velocity [$m\,s^{-1}$]
2 cm	126	891
3 cm	273	1063
4 cm	252	979
6 cm	425	1053
7 cm	431	988
10 cm	606	*

Figure 3. *x-t* diagram of the luminous front for all fill lengths.

The asymptotic nature of the luminous front velocity indicates that there is a maximum flow velocity achievable for this particular formulation and geometry of approximately 1000 $m\,s^{-1}$. More importantly, we note that this flow velocity is significantly higher than even the fastest propagation velocity measured in the filled region of 606 $m\,s^{-1}$. Thus, it's possible that the measured propagation velocity in a thermite may not necessarily be controlled by the reaction kinetics, but instead may be indicative of pressure-driven flow of the hot, multi-phase, fluid through the material. We have recently calculated average flow velocities in porous thermites assuming Poiseuille's flow, where we treat the porosity of the thermite as pipes of known radius *R* [21]. The flow velocity (*v*) was written as:

$$v = \frac{R^2}{8^*\mu}\,\frac{\Delta P}{L}$$

where *R* is the radius of the pores or "pipes", μ is the fluid viscosity, and $\Delta P/L$ is the pressure gradient. In the referenced work, we were interested in calculating the *average* flow velocity, but for a propagating flame it's more appropriate to discuss the higher velocities; at least to the point where some critical flux of energy is transported to ignite adjacent material and propagate the flame. For a fluid with some known viscosity, while the flow velocity is sensitive to the pressure gradient, it is much more sensitive to pore size. Thus, the larger pores, or even cracks in the sample, may be most important to consider in predicting the resultant flow velocity. The observation that the propagation velocity increases at the end of the thermite is likely an artefact of the effective porosity increasing from that in a packed bed (86% porosity) to that in the open tube (100% porosity), thus facilitating faster flow.

3.2 Temperature Mapping of Full Tube

Temperature results for the highest (10 cm) filled tube are shown in Figure 4. Color images of the flame are shown at the top, and the corresponding temperature maps are shown in the middle. The bottom plot in each case is the average temperature taken from the cross-section of the luminous region (i.e. integrated in the vertical direction), at each point along the *x* axis.

In general, the first 2 cm of the tube results in poor images and temperature maps since this is near the ignition point, and the flow could be perturbed by the nichrome wire. It can be seen that the flame reaches an average temperature of close to 3000 K, and this value is sustained throughout the tube for the duration of the propagation, and well after the flame has passed any fixed position. Upon exit of the tube at $x = 12$ cm, a sudden temperature drop of approximately 600 K is observed due to the free expansion into air. Near the leading edge of the flame, there seems to be a small 1–2 cm zone with a slight increase in temperature on the order of 200 K. This zone

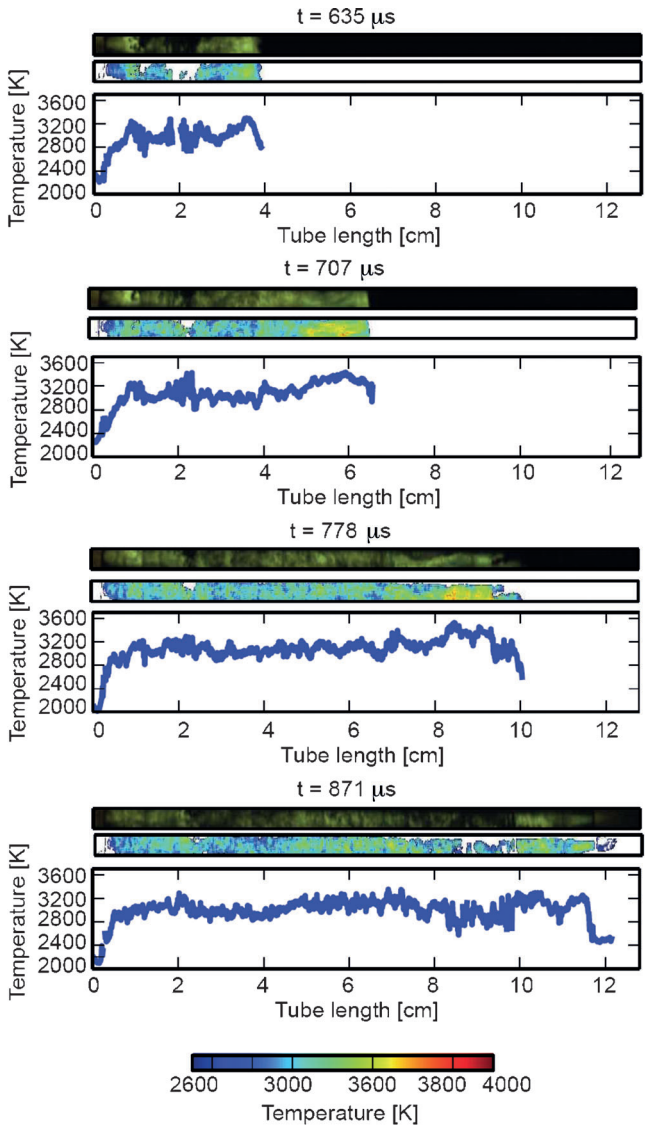

Figure 4. Flame image, temperature map, and average temperature for the 10 cm fill length at different times during the flame propagation event. There is a slight increase in the temperature towards the leading edge of the flame, with a thickness of 1–2 cm. Upon exiting the tube the temperature decreases by 600 K. The color scale for the temperature maps is at the bottom of the figure.

may be indicative of local gas trapping, which would suppress vaporization and cause more energy to be utilized for sensible heating, but this would need more analysis.

The measured flame temperatures are slightly higher than the calculated adiabatic flame temperature of 2843 K using a constant enthalpy and pressure calculation [1], but lower than the value of 3967 K obtained using CHEETAH with constant volume and internal energy. Due to the high porosity in the sample, it's not even clear which equilibrium calculation would be more appropriate to use, as the material is confined but can locally expand. This is important to note, since equilibrium calculations are often used to corroborate flame propagation velocity experiments. The fact that the flame temperature is sustained long after the luminous front passes indicates that either heat losses are slow, or that the reaction continues in the wake behind the luminous front. A two-step reaction mechanism involving condensed-phase reactions followed by heterogeneous burning has previously been suggested for nano-Al/CuO. [22] The measured temperature is very similar to what Bazyn et al. [13] measured for nanometric Al/Fe_2O_3 and Al/MoO_3 thermites ignited by a reflected shock, but is significantly higher than the value of 2250 K reported by Weismiller et al. [15] However, both of these studies used nanometric Al, and so a direct comparison may not necessarily be warranted.

3.3 Partially Filled Tube

Temperature maps from the reaction in partially filled tubes were taken for fill lengths of 2, 3, 4, 6, and 7 cm. Thus, we can directly compare the flame evolution in two regimes: within the thermite and also in the open volume of the tube. A temperature map of the 2 cm, 4 cm, and 6 cm filled are shown in Figure 5, Figure 6, and Figure 7 and at three different instances in time. What can be seen is that the hot temperatures (near 2900 K) are sustained in the forward direction well after the end of the thermite fill, and this behavior was consistent for all samples. If this were an adiabatic expansion process, one would expect the temperature to decay as the material undergoes volumetric expansion into the unfilled regime. The fact that the temperature does not decay indicates that the material is continuing to react as it expands into the unfilled portion of the tube. Since the thermal imaging technique really is capturing thermal radiation from particles (not gases), it follows that entrained particles are expanded along with the gases. This

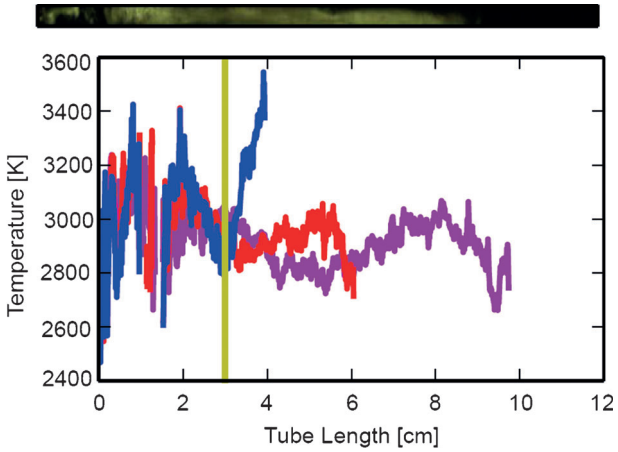

Figure 5. Flame temperature as a function of position for the 2 cm fill length. The image on top is the flame representing magenta curve. The vertical line represents the end of the thermite fill.

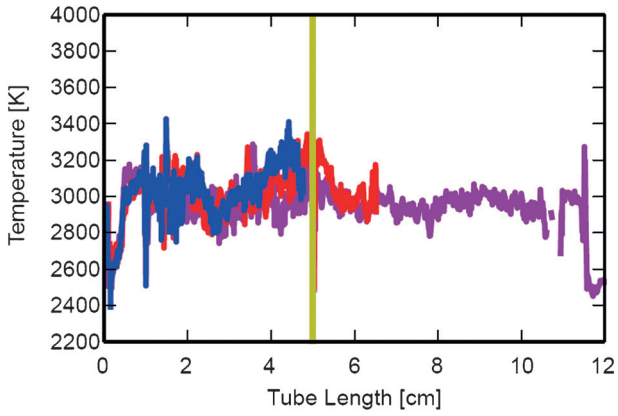

Figure 6. Flame temperature as a function of position for the 4 cm fill length.

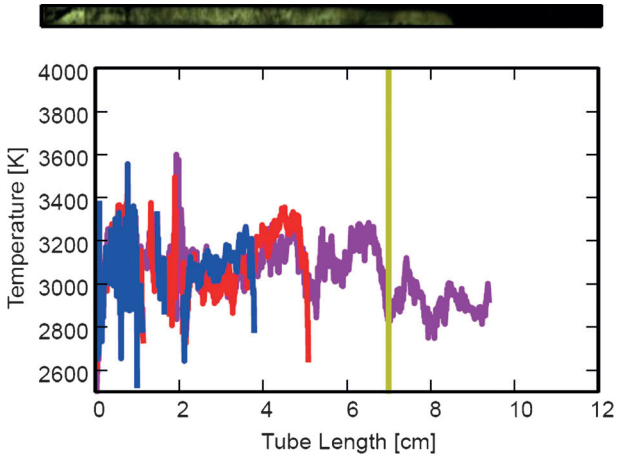

Figure 7. Flame temperature as a function of position for the 6 cm fill length.

is possible for small particles, which have low Stokes numbers and thus can follow the gas streamlines.

Combining the results of the luminous front velocity and the temperature mapping, it's evident that the interpretation of a flame position is fairly subjective and needs much more work if we are to rely on such measurements to draw conclusions about reactivity and combustion mechanisms. While the existence of a luminous front does indicate at least some reaction has occurred, it is not clear what fraction of the material has actually reacted when this wave reaches a position in the material. Given the results of this work, it's possible that hot intermediate and/or product gases from reaction of even the first 1–2 cm of material could be rapidly expanded forward and continue to react well ahead of their initial position. In this case it would not seem appropriate to call this a flame velocity, and instead it would be more indicative of an expansion velocity.

Despite the complexity of interpreting the experimental results, one promising result is that the velocity in the unfilled region does reach a constant value, which is higher than the propagation velocity in the material. Thus, one method to tailor the reactivity would be to utilize organized microstructures designed to promote forward transport of the hot gases and particles through the material. It will continue to be difficult to predict what fraction of the material is reacted during a rapid deflagration, but promoting faster flow is an important first step in enhancing forward energy transport.

4 Conclusion

The velocity and temperature of the luminous front was investigated for loosely packed Al/CuO thermites reacting in acrylic burn tubes. The thermite was filled to various lengths from 2–10 cm, and the filled vs. unfilled regions were compared and contrasted. For any fill length, the propagation velocity approached 1000 m s^{-1} in the unfilled regime, which is notably faster than the fastest velocity of 606 m s^{-1} in the filled region. This increase is likely indicative of the relative porosity, which increases in the unfilled region and thus can support faster pressure-driven flow. This result could be important in that it indicates large pores, cracks, or microstructure could be utilized to promote faster energy transport through the material. The temperature during reaction approached a value of 3000 K within the material, and this temperature was sustained in the wake well after the luminous front passed. In partially filled tubes, a sustained temperature was observed beyond the filled region and as material expanded into the unfilled region. Since the temperature was not observed to decay during this expansion, we suggest that the material continues to react as it expands. Putting this together, we discussed the complexity of interpreting the luminous wave velocity in loosely packed samples, where significant volumetric expansions and sustained reactions can continue and be transported forward through the highly porous network. We suggest that microstructure and other large openings may govern the effective flow velocity, and may be utilized to tailor the energy transport. We also caution against using flame velocity measurements to predict reaction mechanisms or other parameters such as burn time until a much better fundamental understanding of loosely packed burn tubes is available.

Acknowledgments

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