Quantification of Heat Flux from a Reacting Thermite Spray

by

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Abstract

Characterizing the combustion behaviors of energetic materials requires diagnostic tools that are often not readily or commercially available. For example, a jet of thermite spray provides a high temperature and pressure reaction that can also be highly corrosive and promote undesirable conditions for the survivability of any sensor. Developing a diagnostic to quantify heat flux from a thermite spray is the objective of this study. Quick response sensors such as thin film heat flux sensors can not survive the harsh conditions of the spray, but more rugged sensors lack the response time for the resolution desired. A sensor that will allow for adequate response time while surviving the entire test duration was constructed. The sensor outputs interior temperatures of the probes at known locations and utilizes an inverse heat conduction code to calculate heat flux values. The details of this device are discussed and illustrated. Temperature and heat flux measurements of various thermite spray conditions are reported. Results indicate that this newly developed energetic material heat flux sensor provides quantitative data with good repeatability.

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Nomenclature

a_1	Gaussian curve fit coefficient (W/m^2)
a_2	Gaussian curve fit coefficient (m)
a	T(t) curve fit intercept (K)
b	slope of linear curve fit (K/m)
$ Fo$	Fourier number, dimensionless time
k	thermal conductivity ($\text{W}/(\text{m}^*\text{K})$)
m_{fuel}	mass of aluminum (fuel)
m_{ox}	mass of copper (II) oxide (oxidizer)
Q''	energy density (J/m^2)
q_i''	heat flux, current time (W/m^2)
$q_s''(t)$	surface heat flux (W/m^2)
$\bar{t}$	mean time (s)
t_i	current time step (s)
$\bar{T}$	mean temperature (K)
T_b	interior boundary temperature (K)
T_i	current time step temperature (K)
T_n^P	current temperature, current node (K)
T_{n-1}^P	current temperature, west node (K)
T_{n+1}^P	current temperature, east node (K)

T_n^{P+1}	future temperature, current node (K)
$T_{n,new}$	curve fit new temperature value (K)
$T_s(t)$	surface temperature (K)
u_{q_s}	heat flux uncertainty (W/m ²)
u_T	interior temperature uncertainty (K)
u_{T_s}	surface temperature uncertainty (K)
u_x	temperature position uncertainty (m)
Δt_i	current time step size (s)
Δx	spatial step size (m)
α	thermal diffusivity (m ² /s)
ϕ	equivalence ratio

Chapter 1

Introduction

1.1 Thermite Background

The use of metal and metal oxide powders to produce an exothermic reaction (more commonly known as a thermite reaction) has been around for a very long time. The first use of thermite involved a welding application for railroads. In addition, there were some other limited applications for thermite such as the use of aluminum and iron (III) oxide to create a composite pipe lining (Mu, et al. 1997), but the uses were rather limited.

However, with the recent development of nano scale thermites, a door to a new realm of possibilities has been opened. These new possibilities have spawned a wide range of research opportunities. There has been research related to various preparation techniques such as arrested reactive milling (Stamatis, et al. 2009; Umbrajkar, et al. 2006), creation of multilayer foils (Blobaum, et al. 2003) and both pressed and loose mixed powders. A study performed by Puszynski, et al. 2006 examined the possibility of mixing thermites in water in order to allow larger scale thermite production. The approach was to use various organic and inorganic protective coatings on aluminum powders so that oxidation of the aluminum from the water could be minimized during powder mixing. A process to produce large batches of nano scale thermite could be of interest since some of the most significant research has involved both pressed and loose powders.

Although many of the thermite powders can be synthesized, the commercial availability of these powders has greatly aided in the ability to do extensive research with

these materials. Well controlled commercial manufacturing processes allow for consistency with powder properties, and have often provided a range of sizes for various materials.

Motivation for thermite research with nano scale thermite powders deals a great deal with the enhanced material properties found at these smaller length scales. Research performed by Bockmon, et al. 2005 showed that there was a significant increase in combustion velocity of loose Al + MoO₃ powder as the aluminum particle diameter was decreased until a certain diameter threshold was reached. Once the threshold size of 80 nm was reached, the velocity became independent of particle size. A comparison of the combustion velocity of nano and micron scale Al + MoO₃ powders as a function of powder density found that the micron scale thermite combustion velocity increased as the density of the composite was increased while the nano scale composite exhibited the opposite relationship (Pantoya and Granier 2005). Another comparison involving the activation energies of micron and nano scale Al and Ni compacted pellets showed that the nano scale pellets had approximately an order of magnitude lower activation energy than their micron scale counterparts (Hunt and Pantoya 2005). It was concluded that the reactions with nano scale aluminum particles involved a solid-solid phase mechanism while the micron scale aluminum particle compositions required liquid aluminum to react.

For reactions that involve micron scale or larger particles, diffusion is the primary mechanism that controls the reaction rate. An example of diffusion would be CuO into Al₂O₃. In this case, it has been shown that various interface reaction layers consisting of various compositions occur as a result of different high temperatures (Fujimura and

Tanara 1998). Between the CuO and Al₂O₃ it was observed that Cu₂O and CuAlO₂ also formed at various stages of heating.

When particle sizes are reduced to the nano scale regime and high heat rates are applied, classical diffusion theory is no longer able to explain the observed characteristics of these reactions. One possible explanation proposed involves a different reaction mechanism. Under the melt dispersion mechanism theory, the aluminum core melts before any fractures in the aluminum shell occur. The volume change from the molten aluminum creates pressures of 1 to 2 GPa which in turn cause a spallation of the alumina shell. The unbalanced pressure between the aluminum core and the exposed surface creates an unloading wave with high tensile pressures resulting in dispersion of atomic scale liquid clusters (Levitas, et al. 2007). Experiments with Al + MoO₃ were conducted in which the diameter and alumina shell thickness were varied (Levitas, et al. 2008). The results of these tests matched the predictions of the melt dispersion theory.

In addition to the enhanced properties of nano scale thermites, several compositions of micron and nano scale thermites were tested to determine the sensitivity to impact ignition (Hunt, et al. 2009). Results showed that nano scale thermites were only an order of magnitude more sensitive to impact ignition. Given that these materials are an order of magnitude more sensitive unlike the three orders of magnitude sensitivity as seen by other ignition methods, these materials could be used for various applications without a great concern for accidental ignition due to impact.

1.2 Thermite Sprays

As the understanding of nano thermites continues to grow, so does the potential applications. One potential application deals with the use of a thermite spray. A thermite

spray can be achieved with the use of a nozzle to produce a stream of thermite.

Currently, there is no literature on thermites being used as a spray. Depending on the characteristics of the spray, potential uses could include small surface coatings, cutting applications, or as a primer to a secondary explosive. Some of the metals already commonly seen in thermite reactions such as copper and nickel are currently used in various surface coatings (Yuan, et al. 2006). In order for a thermite spray to be used in any of these applications, a diagnostic is needed to be able to quantify parameters of the sprays. There are no commercial ‘off-the-shelf’ devices available to provide quantitative information as parameters are varied. Part of this body of work focuses on the design of a diagnostic approach to allow various parameters to be quantified.

1.3 Nozzle Geometry Review

A review of current literature did not yield any publications that directly deal with nozzle configurations and thermites. Instead, nozzles in a variety of applications were evaluated to aid in nozzle design for various tests. For example, in diesel fuel injection, very small convergence angles are used to atomize fuel (Payri, et al. 2009). However, in achieving a thermite spray, a concentrated stream would be the objective. Therefore, the geometries used to atomize particles serve as boundaries where a thermite spray would not work well. It was found that certain high velocity oxygen-fuel thermal spray torches use a convergent nozzle to achieve sonic conditions where the throat of the nozzle is at the exit (Oberkampf and Talpallikar 1996). The under expanded supersonic region is used to promote turbulence and therefore better mixing of products. In the case of a thermite spray, products should not require the aid of external mixing, and sonic conditions may not be achieved. By modifying the entrance convergent section length of a thermal spray

nozzle, particle velocity was found to decrease (Sakaki and Shimizu 2001). A lower particle velocity could indicate an overall lower stream velocity, and result in lower convective heat transfer. However, in the case of a submerged and confined liquid jet discharging into the same fluid at rest, it has been found that for small aspect ratios (convergence length/diameter < 1) the heat transfer coefficients were at their highest (Garimelia and Nenaydykh 1995). Given that the effect of the convergence section length (and thus the aspect ratio of the section) are uncertain, it was determined that this parameter could potentially effect the energy transfer of a thermite spray. The results of this parameter are discussed further in this body of work.

Chapter 2

Experimental Setup

2.1 Setup Overview

A schematic for the overall setup is shown in figure 1. Two different ignition sources were used throughout testing. The first source was electrical resistance heating. A variac transformer set at 15V provided power to a 0.020 inch diameter nichrome wire with a resistance of 30.4 Ω /m (nichrome 2009). A sharp bend is produced inside the spray apparatus to create a localized increase in wire resistance. This localized point in turn becomes a 'hot spot' which allows the ignition location of the powder to be more controlled. The other source included a 12V DC power source and a model rocket igniter within the spray apparatus. An infrared camera (FLIR Phoenix 9803 high speed MWIR camera) was positioned such that the face plate of the sensor could be viewed. This allows the infrared camera to capture qualitative information regarding the temperature distribution on the face plate of the sensor. A Phantom 7.1 high speed camera (Vision Research, Inc.) was positioned so that a side view of the thermite spray could be captured. The sensor was directly connected to an InstruNet 100B external data acquisition system (DAQ). The DAQ has signal conditioning amplifiers and analog filters built in to allow the direct connection of thermocouples. The DAQ computer is connected to a universal power supply (UPS). The UPS provides battery power to the computer allowing it to be disconnected from the wall power outlet. The battery source ensures noise from the commercial power grid or other equipment is not carried over into the computer through an external power source.

Figure 1. Schematic of the test setup overview

The setup for these experiments was designed to allow for the measurement of surface temperature and heat flux from a reacting thermite spray impinging a surface orthogonally. Many potential applications for a thermite spray would most likely involve a similar setup, and therefore, data acquired in th

is form could be more directly utilized. The distance from the tip of the nozzle on the spray apparatus to the front face of the sensor is 1.05 inches (figures 2 and 3).

Figure 2. View of sensor plate

Figure 3. View of nozzle

The centerline of the nozzle is in direct alignment with probe 5 of the sensor (figure 8). This alignment allows for the determination of symmetry of the spray as well as a mapping of energy transfer into the plate.

2.2 Spray Apparatus

The spray apparatus consists primarily of four components. The design allows parts that are damaged or otherwise modified during a test to be easily interchangeable with a new component. Due to the conditions of the test, two components are specifically designed for replacement. The first component is the nozzle. A Cross sectional view of the nozzle as well as the actual part is shown in figure 4 and figure 5, respectively.

**Figure 4. Cross sectional view of
spray nozzle**

**Figure 5. Inside view of thermite
spray nozzle**

It should be noted that the holes for the ignition wires are not shown in figure 3. The center of the ignition wire holes are 0.150 inch from the centerline of the nozzle. This position allowed the ignition wires to be very close, but not touch the acrylic liner (figures 6 and 7). By placing the wires very close to the liner, the disturbance of the wires on the thermite flow could be kept to a minimum. This disturbance is unavoidable with the available equipment and setup design. The ignition wire holes are drilled to 0.021 inch. With a 0.020 inch diameter ignition wire, only a 0.0005 inch gap exists between the wire and wall of the hole. This small tolerance allowed the wire to be inserted without requiring excessive force to be used while minimizing the opportunity for any particulates to be jettisoned out of the ignition holes. Avoiding the use of

excessive force to insert the wires (i.e. the need for pliers to pull the wire through) helps to prevent accidental damage to the wire which could lead to undesired 'hot spots' during ignition.

Before the conic section of the nozzle, it can be seen in figure 4 that there are two square cut-in regions of slightly larger diameter. The first square cut-in region just before the conic section mates with an acrylic cup. This diameter and the outer diameter of the acrylic cup have a light press fit. The second square cut region before the conic section allows for the seating of an o-ring. The press fit with the acrylic cup and the O-ring serve as the primary seals for this system. The acrylic cup is a disposable component used to prevent damage and wear to the body and end plug components. This acrylic cup can be seen mated to the nozzle in figure 6.

Figure 6. Acrylic cup mated with the spray nozzle

To aid in visibility, a ‘spent’ cup is used to ‘add contrast’ to the internal volume of the cup.

The nozzle and liner mate with the apparatus body. The apparatus body serves to house the acrylic cup. A second O-ring can be seen in figure 7, which serves as a secondary seal for the nozzle. At the opposite end of the body, the end plug is inserted. When the body and plug are mated, the bottom of the acrylic liner rests against the top of the plug. On the plug, two O-rings can be found. These serve two purposes. First, they provide ‘grip’ when the plug is inserted into the body, and second, they function as backup seals in the event of a leak, such as a fracture of the acrylic cup.

Figure 7. Apparatus body and end plug shown with fully assembled apparatus

2.3 Sensor Design

As mentioned, a commercially viable solution for acquiring the data of interest could not be found. As a result, the entire sensor apparatus had to be designed and built ‘in house’. The concept used for this design is similar to a design used to measure the effect of aluminum on heat flux from a simulated rocket propellant flame (White, et al. 2007). Thermocouples are used to take measurements of interior temperatures along a rod. These temperature histories can then be used to back calculate the surface temperature and heat flux. These calculations are based on a one dimensional transient

model. The arrangement of temperature probes (rods with thermocouples attached) can be seen in figure 8.

Figure 8. Arrangement of probes shown on the face plate

The spacing between the centerline of each probe is 0.3 inch. The probe diameters are 0.125 inch. The probe diameter was selected to allow for a sufficiently large area of the plate to be sampled such that oscillations that might be present in the heat flux could be damped, but also small enough that a point source heat flux could still be considered a reasonable assumption. Between the edge of each probe and the front face plate is a gap of approximately 0.0005 inch. The thickness of the stainless steel immediately surrounding the probes is 0.010 to 0.015 inch thick. This gap and plate thickness provide a radial contact resistance (calculated with air as median at STP) of 126.6 to 189.8 K/W. Past the 0.010 to 0.015 inch thickness depth, the diameter is expanded to 0.1875 (3/16) inch. Figure 9 shows a cross sectional view of a probe and the face plate for clarity.

Figure 9. Cross section of probe and face plate

The face plate was milled smooth and the probes were positioned so that each probe end is flush with the plate surface. This minimizes fluid flow disturbances from uneven heights, surface roughness and the presence of the probes themselves. The faceplate and probes are both constructed of 316 stainless steel.

With the use of thermocouples, the true temperature profile will never be recorded, as the very presence of the thermocouple disturbs the temperature field. Even very similar setups have been shown to have completely different effects on the relative error recorded. For example, a semi-infinite plate with a thermocouple embedded beneath the surface causes a very different temperature disturbance depending on whether the thermal conductivity of the thermocouple is greater than or less than that of the surrounding material. (Beck and Hurwicz 1960; Beck 1962). It has been shown in a previous study that as the radius of the thermocouple is decreased, the disturbance of the

thermocouple decreases dramatically as well (Keltner and Beck 1983). Given this relationship, type E thermocouples with a radius of 0.003 inch were purchased (Omega Engineering, Inc.). This size was the smallest thermocouple size that could be readily purchased while still having an insulation sheath to prevent undesired temperature junctions (thermocouple wire contact before the probe). Type E thermocouples were selected due to their highest change in voltage output per degree change in temperature of $68 \mu\text{V}/^\circ\text{C}$ (Thermocouple 2009; Lipták 1993). This voltage change is a result of the Seebeck effect. The Seebeck effect is solely a function of the temperature difference between the two junctions of the dissimilar, homogeneous conductors. It is independent of all other variables, including cross section, temperature distribution and temperature gradients, as long as the compositions of the two conductors remain unchanged (Pollock 1982; Pollock 1990). All probes have thermocouples located at the same spacing from the tip of 0.035, 0.505 and 2.000 inches, respectively. A fixture aided in cutting small v-notches 0.007 inch deep and 0.014 inch wide on opposite sides of the rods. Figure 10 shows this fixture along with a visual reference to aid in giving a notch size perspective.

Figure 10. Fixture used to mill v-notch on opposite sides of rods

In order to solder thermocouples to 316 stainless steel, the notches first had to be tinned. Tinning is the process in which the surface to be soldered is coated in a thin layer of solder. In order to solder to the metal, the oxide layer must first be dissolved, and the solder must be used to quickly replace the dissolved oxide layer (Budnikov 1970).

Without dissolving this oxide layer, capillary action can not be achieved between the stainless steel and the solder. As a result, the solder will not bond to the stainless. Upon tinning the surface, the thermocouple wires are placed in the notches and the notches are filled with solder. Excess solder around the thermocouples is carefully removed to avoid increasing the thermal inertia at the thermocouple location. The thermal conductivity of

this 96% Sn 4% Ag solder is 33 W/(m*K) (Chen and Liang 2006) while the stainless steel has a value of 13.4 W/(m*K) (Incropera et al. 2007; Mills 1992; Cengel 2003). Given a thermal conductivity ratio of 2.5 along with the small volume of the solder, 'lumped capacitance' assumption is made with respect to the solder. The resulting temperature junction is shown in figure 11.

Figure 11. Completed temperature junction

The law of intermediate metals states that a third metal can be added between to other metals and the net emf produced by the third metal will be zero as long as the junction temperature between metal one and metal three is the same as the junction temperature of metal three and metal two (Manual on the use of thermocouples in

temperature measurement, 1974). In this case, the law can easily be extended given that isotherms should be orthogonal to the length of the rod.

A calibration curve was created for each probe by using an Imperial IV Laboratory Oven over a temperature range of 25°C to 180°C. The control temperature was measured with a 0.1°C resolution Omega digital thermometer (model number HH11A). This thermometer uses a type K thermocouple wire which allowed the temperature readings to be taken relatively close to the probes. The calibration setup is shown in figure 12.

Figure 12. Probe calibration setup

The calibrations showed a maximum 3.5 degree variation between the control temperature and the probes, and less than a degree deviation between thermocouples attached to the same probe. This 3.5 degree variation could be a result of placement in the oven and the slight variation of temperature within the oven. The calibration curves can be seen in figure 13.

Figure 13. Calibration curves of all thermocouples

The casing for the probes was machined out of aluminum. The void between the probes and the casing was filled with Cotronics 740 series alumina silica insulation foam which has a thermal conductivity of 0.1442 W/m-K (Cotronics Corp.). This insulation aids in improving the one dimensional conduction assumption.

Chapter 3

Testing Procedures

3.1 Thermite Powder

Thermite reactions can use several different metals and metal oxides for fuels and oxidizers. In this setup, aluminum was used for the fuel and copper (II) oxide was used as an oxidizer. The main motivator for this choice of thermite is due to the high gas production of this reaction. Given that the primary mode of heat transfer in a thermite spray is convection, higher gas production should lead to higher internal pressures which in turn should produce higher stream velocities. Data regarding pressures for this type of setup is not available, but several researchers have studied confined burn pressures by using modified versions of a Bockmon tube. One modified version of the Bockmon tube utilized an exploding bridge wire to ignite the sealed end of the tube. Pressure transducers were located at 1 cm intervals along the tube. Peak pressures of an aluminum and copper (II) oxide reaction showed to be on the order of 10 MPa (Malchi, et al. 2008).

All tests performed utilized the same fuel and oxidizer. The aluminum nano powder was commercially produced by Novacentrix, formerly known as Nanotechnologies, Inc. The particles are spherical shape with a mean 80nm diameter and an 80% active aluminum content by mass. The aluminum powder contains an alumina passivation shell. This passivation shell accounts for the other 20% of the aluminum particle mass. The copper (II) oxide used was commercially produced by Alfa Aesar. The particles are spherical shape and have a mean particle diameter between 30 and 50nm.

Various masses of powder are measured out on a mass scale accurate to 0.1 mg. The mass of each powder depends on the desired total mass, equivalence ratio and active aluminum content. An equivalence ratio (Φ) is defined as:

$$\Phi = \frac{m_{fuel} / m_{oxidizer}}{\left(m_{fuel} / m_{oxidizer} \right)_{st}} \quad (1)$$

where m_{fuel} is the mass of the fuel, $m_{oxidizer}$ is the oxidizer mass and the subscript st represents the stoichiometric ratio of fuel and mass. The stoichiometric ratio refers to a reaction in which the optimal ratios of fuel and oxidizer are used so that a complete reaction would consume all of the fuel and oxidizer. The global reaction for a stoichiometric reaction between aluminum and copper (II) oxide is shown in equation 2.

A metal container with a pour spout was used in the scale to hold the powder. Once the proper amount of powder is measured, it was transferred to a plastic lab cup. A chem-wipe was used to ensure the metal container was clean of any residues from the previous powder. This process is repeated for the second powder.

Approximately 60 mL of Hexanes was added to the cup. The Hexanes serve as a fluid medium for mixing of the powders. A Misonix Sonicator 3000 ultrasonic probe mixer was used to break up agglomerates and promote homogeneous mixing of particles.

The mixing cycle consists of 10 seconds with the sonicator on and 10 seconds idle, repeated 6 times for a total process time of 120 seconds. The 10 second breaks during mixing allow the particles to cool and reduce the chance of thermal breakdown of particles (Dean 2008).

Upon completion of mixing, the slurry of Hexanes and thermite was poured into a Pyrex glass pan. The Pyrex pan was placed on a hot plate which was set to the 'warm' setting. The pan was allowed to sit until the Hexanes are fully evaporated off. This process takes approximately 15 minutes and can be visually verified. When the thermite is submerged in Hexanes, it has a rather dark tint. As the Hexanes evaporate off, the thermite changes to a light grey color. Once the Hexanes are fully evaporated, a light grey residue with a dry appearance will remain. The Pyrex pan was then removed and a grounded metal wire brush is used to gently scrape the residue out of the Pyrex pan and into the original plastic container. The plastic container was capped and information regarding thermite mixture and date information was marked accordingly.

3.2 Loading Procedure

In order to initiate the reaction, either a model rocket igniter or a nichrome wire was used. The igniter and nichrome wire are shaped accordingly to fit into the nozzle wire ignition holes. The igniter and nichrome wire are shown in figure 14 and an example of a nichrome wire inside a nozzle can be seen in figure 5.

Figure 14. Illustration of ignition sources

Next the acrylic cup and O-ring, spray apparatus body, and end plug are all assembled so that the acrylic cup can be seated against the end plug. Due to mass limitations of the scale, the end plug is then removed and the apparatus body is placed on the scale (figure 15). The scale is allowed to ‘settle’ and the mass is zeroed out. Approximately 250 mg (± 0.5 mg) of Al + CuO thermite is loaded into the acrylic cup (figure 7).

Figure 15. Mass scale used for powder loading

The body is carefully removed from the scale and very lightly tapped on the counter top to level out the thermite within the cup.

Past studies of thermite reactions have shown that loose thermite powder could have advantages in this setup over pressed thermite. When using nano scale aluminum and molybdenum trioxide, it has been observed that loose powders exhibit higher combustion velocities than pressed pellets of the same composition (Moore and Pantoya). By having a higher combustion velocity, a higher pressure could be achieved as the material is jettisoned out of the nozzle orifice. Higher pressures will result in higher stream velocities, which will increase the convective heat transfer coefficient.

Before the nozzle was placed on the apparatus body, the end plug was mated back with the body. The nozzle was then placed on the apparatus body. In order to not disturb the powder, the nozzle must be lowered onto the body at a very slow rate until the acrylic cup was completely seated into the nozzle. The holder for the spray apparatus (figure 16) was placed over the spray apparatus. The holder was then bolted to the test apparatus along with a power screw to ensure the nozzle and acrylic cup remain assembled during the test.

Figure 16. Test Apparatus with spray apparatus section shown

The sensor was then connected to the InstruNet DAQ as shown in figure 17.

Figure 17. InstruNet DAQ connected to the sensor junction box

The junction box shown was designed to avoid errors due to wires being connected to the wrong port. The copper wires have an insulated coating that prevents grounding out between thermocouples. A noise test was performed by performing a noise check with and without the junction box, and a difference in noise was not detected. The random noise for this setup was determined to be approximately 0.2°C .

Next, the Phantom 7.1 camera and Phoenix Infrared camera are positioned accordingly as discussed in chapter 2. Alignment and focus of the infrared camera was achieved by using a hot point source such as a recently lit match. The Phantom camera was focused on a ruler placed even with the center probe. A snap shot of the ruler was taken, which was later used as a length scale reference for high speed video data. The lenses on both the infrared and visible spectrum cameras contain a focus, but do not

contain a zoom. To adjust the size of the plate seen by each camera, the camera distances to the front plate are adjusted until the desired plate size seen by each camera was achieved.

3.3 Equipment

~ Thermocouple data was recoded using the InstruNet DAQ manufacturer's software (InstruNet World). The settings for the software are shown in Table 1.

Table 1. InstruNet Software Settings

Hardware Type	Type E Thermocouple
Temperature Range	0 to 200 Celcius
Integration Time	0.000166667
Low Pass Filter	40 Hz
Connection Type	Single End
Sampling Frequency	250 Hz

InstruNet has preprogrammed conversions that allow the user to select the thermocouple type used via the hardware type setting. These preprogrammed conversions allow the data to be outputted as a temperature instead of a voltage, saving valuable analysis time. The integration time parameter sets the time range for an average to be calculated. During this time, many data point samples are read. The average of these points helps to avoid random erroneous 'spikes' in the data. Instead these erroneous 'spikes' are averaged out as these errors occur in both the positive and negative directions. The low pass filter allows a built in analog filter to be enabled. The connection type refers to whether a thermocouple is allowed to have a floating reference or whether a known ground state is utilized. This setup employs the grounded state setting.

The Phantom 7.1 Camera settings consisted of a 128 pixel width by 256 pixel height window. A frame rate of 40,000 frames per second was selected along with a $15\mu\text{s}$ time for both the exposure and EDR exposure times. The f-stop on the camera lenses was set at either 22 or 32. Both f-stops allowed for the determination of the initial flame front velocity, however, each setting allows for different information to be captured during the test.

Alligator clips are used to connect the ignition source to the power source. The power source was dependent on the ignition source. For resistance heating, a variac transformer (figure 18) was used while a 12V battery source was used for the rocket igniter (figure 19).

Figure 18. 12V battery for rocket igniter

**Figure 19. Variac transformer
for nichrome wire**

The sequence of events for the test included initiation of the InstruNet data acquisition, initiation of the infrared camera, ignition of the reaction via a power switch

(variac transformer) or completing the circuit between the battery lead and alligator clip and a post trigger after the reaction to stop the high speed camera recording. Upon completion of the test, the sensor face was scraped clean with a razor and wiped clean with an alcohol soaked rag.

Chapter 4

Results

4.1 Verification of Symmetry

Although the sensor is equipped with an array of probes that allow both the centerline symmetry to be verified as well as measurements at specified distances from the centerline, equipment limitations do not allow both of these criteria to be measured simultaneously. Therefore, before any tests were conducted that use the probes at specified distances from the center (probes 1 through 3 in figure 8), the probes surrounding the center probe (probes 3, 4, 6 and 7) were used to verify symmetry about the center probe. A test with an equivalence ratio of one ($\Phi = 1.0$) and a nozzle angle of 45 degrees (measured between the centerline and cone wall) was performed. The results of the front internal thermocouples are shown in figure 20.

Figure 20. Front thermocouples of centerline and surrounding probes

The infrared camera was used to achieve a qualitative verification of the temperature distribution symmetry. A still shot of the front plate immediately following the test is shown in figure 21.

Figure 21. Infrared image of the front sensor plate

It should be noted that the ‘hot regions’ seen on the bottom and top left corners are actually surface reflections from a hot nichrome ignition wire and should be disregarded.

4.2 Filtering of Data

Three steps were used to filter the thermocouple data. The first step was the use of an integration time. All tests included a $1/6000^{\text{th}}$ integration time. The second step, which took place simultaneously with the first step, was the use of low pass filter. This filter only allows or greatly dampens oscillations occurring at frequencies above 40 hertz. The last step implemented to reducing noise deals with a post test numerical filter. This ‘filter’ is a linear regression curve fit in which the current point, two previous points and two future points are used to produce a best fit slope and intercept. The slope and intercept along with the time at the current point are used to find a new point. This operation is shown mathematically in equations 3 through 5.

$$b = \frac{\sum_{i=n-2}^{n+2} (t_i - \bar{t})(T_i - \bar{T})}{\sum_{i=n-2}^{n+2} (t_i - \bar{t})^2} \quad (3)$$

$$a = \bar{T} - b\bar{t} \quad (4)$$

$$T_{n,new} = bt_n + a \quad (5)$$

In these equations, b represents the slope, a the intercept, t the time, T the temperature, and $T_{n,new}$ the new temperature. For the first and second temperature points, the limits of summation are adjusted to $i = n$ to $n+2$ and $i = n-1$ to $n+2$, respectively. Figure 22 shows a magnified view of the region with the greatest temperature change for the center probe front thermocouple of a test utilizing an equivalence ratio of one ($\Phi=1.0$) and a nozzle cone angle of 45 degrees.

Figure 22. Comparison of unfiltered and numerically smoothed data

Another measure of the quality of data to be used before the application of the inverse heat conduction code is a temporal derivative of the temperature. The temporal derivative for the same data shown in figure 22 is shown in figure 23.

Figure 23. Temporal derivative of numerically smoothed temperatures

The temporal derivative is calculated by subtracting the current temperature point from the immediate future temperature point and dividing by the time step. The temporal derivative plots for three other geometries can be found in appendix A.

4.3 Inverse Heat Conduction Analysis

An inverse heat conduction problem occurs when interior information is known instead of boundary condition information. When numerically solving the inverse heat conduction problem, many concepts that work well in a direct heat conduction problem (classical boundary value problem) do not carry over. In the direct problem, smaller temporal and spatial steps can increase resolution while in an inverse problem, reducing the temporal and spatial sizes can increase oscillations and result in an unstable solution

(Beck 2006). For the analysis performed to calculate the surface heat flux and temperature, a commercial software package (IHCP1D) from Beck Engineering Consultants Company was used. This software uses a function specification method, which allows the nonlinear case to be solved. This method as discussed by Beck, Blackwell and St. Clair, Jr. 1985 involves assuming a functional form of the surface heat flux. Examples of assumptions that could be made are constant, linear, parabolic, cubic or exponential heat flux functions. These functions can be assumed over a segment consisting of the current time step and a specified number of future time steps. At each new time step, the procedure is repeated. This approach provides stability to the problem, which allows much smaller time steps to be used over methods which only use the current time step. Further details such as the surface heat flux function along with any other algorithm details of this proprietary code are unavailable, and thus the specific algorithm can not be outlined. The calculated results of the surface heat flux and surface temperature are shown in figures 24 and 25, respectively. These two plots correspond to the same test as figures 22 and 23 for consistency. Surface temperature and heat flux plots for other geometries are shown in appendices B and C.

Figure 24. Heat flux for $\Phi=1.0$ and nozzle angle of 45 degrees

Figure 25. Surface temperature for $\Phi=1.0$ and nozzle angle of 45 degrees

4.4 Verification of Surface Heat Flux and Temperature

In order to verify the calculated surface temperature and heat flux, an algorithm using the outputted surface temperature and heat flux as one boundary condition, and a known surface temperature as the other boundary condition. This explicit, one dimensional, transient heat conduction scheme is shown below.

$$T_n^{P+1} = (1 - Fo)T_n^P + Fo * T_{n+1}^P + \frac{\Delta x}{k} Fo * q_s''(t) \quad (6)$$

$$T_n^{P+1} = (1 - 3Fo)T_n^P + Fo * T_{n+1}^P + 2Fo * T_s(t) \quad (7)$$

$$T_n^{P+1} = (1 - 2Fo)T_n^P + Fo * T_{n-1}^P + Fo * T_{n+1}^P \quad (8)$$

$$T_n^{P+1} = (1 - 3Fo)T_n^P + Fo * T_{n-1}^P + 2Fo * T_b \quad (9)$$

$$Fo = \frac{\alpha \Delta t}{(\Delta x)^2} \quad (10)$$

Equations 6 and 7 correspond to the boundary node of the calculated heat flux and temperature, equation 8 is used for the interior nodes and equation 9 is used for the known temperature boundary condition. The spatial terms originate from a central differencing scheme and the temporal term from a forward differencing scheme. This gives the algorithm an order of accuracy of $O(\Delta t) + O[(\Delta x)^2]$ (Taler and Duda, 2006).

The known boundary condition consisted of the third thermocouple on each probe which was located 2 inches from the tip. The algorithm was written in Matlab, and allowed the user to determine the step sizes by entering the Fourier number and number of nodes to be used. These values were adjusted until convergence was achieved. The final parameters used correspond to a Fourier number of 0.005 and 2000 nodes. The calculated interior temperatures were subtracted from the measured temperatures. A plot of the temperature differences using calculated heat flux and surface temperature for the front thermocouple of probe 5 for $\Phi=1.0$ and a nozzle angle of 45 degrees is shown in figure 26. Additional plots can be found in appendix D.

Figure 26. Residuals of calculated and measured internal temperatures

4.5 High Speed Video Data

Video from the Phantom 7.1 camera was used to record the spray from each test. This data allowed for the profile of the spray to be analyzed in more detail. Several still shots from one test are shown in figure 27.

Figure 27. Still images of thermite spray

This series of images was produced by a test with $\Phi=1.2$ and a nozzle angle of 45 degrees. This data provides visual information to help determine if calculated results from the sensor are reasonable. Total burn duration varied between 12 and 18 ms.

4.6 Total Energy Absorbed

The total energy absorbed within a 0.9 inch (2.286 cm) radius of the center probe was approximated from the corresponding heat flux curves with respect to probes 1, 2, 3, and 5. First the area under the curve was approximated with a numerical integration scheme using rectangles at each time step. This operation is shown in equation 11.

$$Q'' = \sum_{i=1}^n q_i'' \Delta t_i = \Delta t \sum_{i=1}^n q_i'' \quad (11)$$

This first step yields an energy density for each probe. The numerical integration was performed from 0 to 1 second, which exceeds the heat transfer times for all tests. Plots of

energy densities comparing equivalence ratios and nozzle cone angles are shown in figures 28 through 30, respectively.

Figure 28. Plot of energy densities at various equivalence ratios using nichrome wire ignition

Figure 29. Plot of energy densities at various equivalence ratios using model rocket igniters

Figure 30. Plot of energy densities with various cone angles using model rocket igniters

Next, the shell method was used to determine a total energy transfer value based on the energy densities calculated at each probe location. A Gaussian curve fit was used to approximate the energy density as a function of radius. This calculation can be shown as:

$$Q = \int_0^R 2\pi x * Q_i''(x) dx \quad (12)$$

where,

$$Q_i''(x) = a_1 e^{-\left(\frac{x}{a_2}\right)^2} \quad (13)$$

The results of these computations can be seen in tables 2 through 4.

Table 2. Spray efficiencies as a function of equivalence ratio using nichrome wire ignition

Efficiency Measurements (45 Degree)			
	Equivalence 1	Equivalence 1.2	Equivalence 1.4
E _{in} Plate (J)	271.8	414.3	370.1
$\Delta h_{\text{combustion}} 2\text{Al} + 3\text{CuO} \rightarrow \text{Al}_2\text{O}_3 + 3\text{Cu}$	983.0	941.7	903.6
$\Delta h_{\text{combustion}} 2\text{Al} + 3\text{O}_2 \rightarrow 2\text{Al}_2\text{O}_3$	0.0	262.2	503.1
Lower Efficiency	27.65%	34.41%	26.31%
Upper Efficiency	27.65%	44.00%	40.95%

Table 3. Spray efficiencies as a function of equivalence ratio using model rocket igniters

Efficiency Measurements (45 Degree)			
	Equivalence 1	Equivalence 1.2	Equivalence 1.4
E _{in} Plate (J)	169.4	155.3	139.9
$\Delta h_{\text{combustion}} 2\text{Al} + 3\text{CuO} \rightarrow \text{Al}_2\text{O}_3 + 3\text{Cu}$	983.0	941.7	903.6
$\Delta h_{\text{combustion}} 2\text{Al} + 3\text{O}_2 \rightarrow 2\text{Al}_2\text{O}_3$	0.0	262.2	503.1
Lower Efficiency	17.21% ±3.10%	12.88% ±9.66%	9.94% ±0.90%
Upper Efficiency	17.21% ±3.10%	16.47% ±12.35%	15.48% ±1.40%

Table 4. Spray efficiencies as a function of cone angle using model rocket igniters

Efficiency Measurements ($\Phi = 1.0$)				
	15 Degree	30 Degree	45 Degree	60 Degree
E _{in} Plate (J)	90.7	148.6	200.7	127.0
$\Delta h_{\text{combustion}} 2\text{Al} + 3\text{CuO} \rightarrow \text{Al}_2\text{O}_3 + 3\text{Cu}$	983.0	983.0	983.0	983.0
Efficiency	9.22%	15.12%	20.42%	12.92%

The efficiencies reported are based on the available potential chemical energy before the reaction (heat of combustion). Tables 2 and 3 includes an upper and lower efficiency based on the assumptions that all the excess fuel reacts with the air (lower efficiency) and no excess fuel reacts with the air (upper efficiency).

4.7 Uncertainty Analysis

The uncertainty associated with the measured temperatures is primary driven by random measurement error and position. To account for random error as well as numerical error, a standard deviation of the differences between measured and calculated temperatures from 0 to 1 second (figure 25) is used. By evaluating the uncertainty of the temperature differences or residuals, both the random errors, as well as numerical uncertainty, are accounted for. To normalize the residuals, the residuals are divided by the measured (actual) temperature at each point. The uncertainty of the heat flux and surface temperature can be expressed as:

$$\frac{u_{q_s}}{q_s} = \sqrt{\left(\frac{u_T}{\Delta T}\right)^2 + \left(\frac{u_x}{x}\right)^2} \quad (14)$$

$$\frac{u_{T_s}}{T_s} = \sqrt{\left(\frac{u_T}{\Delta T}\right)^2 + \left(\frac{u_x}{x}\right)^2} \quad (15)$$

The position uncertainty deals with the thermocouple location uncertainty, u_x , and the distance to the nearest thermocouple from the tip. The uncertainty varies with each probe and each test, but was found to be at or below 7%. The equivalence ratio tests using

model rocket igniters included repeatability testing, and therefore a repeatability uncertainty is shown within the efficiency values.

Chapter 5

Discussion

5.1 Data Filtering and Inverse Heat Conduction

The data shown in figure 20 verifies that symmetry about the center probe exists. Probe 6 shows a slightly higher temperature than probes 3, 4 and 7, but was within a reasonable range for the symmetry assumption. Buoyancy effects would be negligible during a test due to the short test duration. With the alignment and careful positioning of the sensor and spray apparatus, the centerline of the spray should always correspond with the centerline of the center probe. The negligible buoyancy and consistent alignment along with the qualitative infrared image strengthen the symmetry assumption.

Thermocouple noise is always unavoidable to some extent. With the temperature range seen within the front thermocouples, the 0.2°C random noise would not be significant if this temperature data was not used for further calculations. However, the scheme utilized to determine the surface temperature and heat flux is sensitive to noise. The reasons for this sensitivity were discussed in section 3 of chapter 4. Given this sensitivity, the steps implemented to reduce this noise while avoiding a loss of resolution of data contribute significantly to the data quality.

Preliminary tests that did not take advantage of data filtering yielded data that could not be used to calculate surface temperature and heat flux without an excessively large number of future time steps. Although more future time steps increase stability, they also flatten the calculated heat flux and surface temperature curves due to a numerical diffusion effect. This has a small effect on the area under the curve when a sufficiently large time scale is used. In this case, 1 second is sufficiently large, as most of

the heat transfer occurs within approximately 0.2 second. Although the total energy values are only mildly affected, the peak values can be very significantly affected. The integration time greatly aids in avoiding relatively rare, but very large magnitude errors (i.e. data outliers) from being captured as the temperature at any specific moment. These outliers could create large, erroneous oscillations in heat flux if left unresolved.

Both the low pass analog filter and the post test numerical filter work hand in hand in addressing erroneous temperature oscillations. Higher frequency noise can originate from any number of sources, but the most important detail is the knowledge that none of the ‘real’ information occurs in these higher frequency ranges. This detail is what allows a low frequency filter to be utilized. The numerical filter depends on the sample rate and the rate at which temperatures change to determine the number of points that can be used for a linear curve fit. When the sample rate is sufficiently large enough, this approach is very effective in reducing remaining noise while avoiding the loss of peak values. It was determined experimentally that 5 data points provided appropriate ‘data smoothing’ while not obscuring peak values for all data sets analyzed.

The ‘data smoothing’ can often reduce the true bandwidth of the sensor. This sensor has a bandwidth of approximately 10 Hz, which is sufficient for capturing the data of interest. Although 5 to 10 kHz pulsations in the spray can be seen from the high speed video, the higher frequency heat flux values that result from these pulsations do not effect the total energy calculations.

The temporal derivative plot (figure 23) provides a quick and easy way to verify that a data set has the potential to yield quality data. The shape of the heat flux curve will bear some resemblance to this curve. When large oscillations are present, more future

time steps will be required, which will greatly dampen the peak values. By performing a quick visual analysis of this plot, valuable time can be saved if the data is determined to be too noisy to be analyzed. Further 'data cleanup' could potentially be used, but it is always good practice to compare original and 'filtered' data to ensure the 'cleanup' did not degrade the data beyond use.

In figure 23, Probe 1 (center probe) has some minor oscillations in the derivative, but an intuitively expected curve shape was produced. It can be seen that the oscillations become more dominant with respect to the maximum derivative value when moving away from the center probe. This relationship is due to the equivalent of a signal to noise ratio problem. For each probe that is located further from the center probe, the maximum temperature rise becomes lower, but the random noise does not decrease (i.e. lower signal but the same noise). During the numerical smoothing, the large temperature changes dominate the noise and a smoother derivative curve is produced. As the noise becomes more dominant at further distances, the data smoothing is not as effective. If the oscillations within the derivative become too large, the data will become too degraded to yield any quality results.

5.2 Calculated Surface Temperature and Surface Heat Flux

The first parameter to be varied was the equivalence ratio. The equivalence ratios of 1.0, 1.2 and 1.4 were chosen to see if a slightly fuel rich mixture could produce larger heat flux values. If all of the excess aluminum reacts with the oxygen in the air, the potential energy per gram of thermite would be greater than the stoichiometric ratio; however, if none of the excess aluminum reacted with the air, the potential energy per gram of thermite would be less. Table 3 indicates that an equivalence ratio of 1.0 results

in a higher total energy transfer. This higher energy transfer coincides well with a previous study in which it was found that higher gas production for Al + CuO occurred at an equivalence ratio slightly above 1.0 (Sanders, et al. 2007). In this case, higher gas production would have lead to higher convective heating at the center.

In figure 30, differences in the total energy transfer as a function of nozzle angle are difficult to identify. However, when the total energy transfer is calculated, there is a clear performance trend that indicates optimal performance for a nozzle angle of 45 degree.

Although is has been shown that a different nozzle angle did have an impact on the performance of the spray in this setup, it is not reasonable to conclude that the performance as a function of the nozzle angle has been fully evaluated. The nozzle angle is a function of both the length and diameter of the cone. This study shows that the spray is dependent on the length, but does not provide any conclusions about the diameter.

Chapter 6

Conclusions

The need for a diagnostic technique for quantification of a thermite spray was presented along with some test results used to validate the diagnostic technique. Test results included an examination of three different variables. The approach was to utilize interior temperatures, which protected the thermocouples from damage, and employ an inverse heat conduction code to provide reasonable estimations of surface heat flux and temperature. Details of the equipment used along with necessary steps taken to address and minimize noise were discussed. A commercial code along with an algorithm to validate the results of the code was employed. The methodology and approach used were shown to provide reasonable results for situations in which quick response time is needed in a 'hostile' testing environment. The simplicity of this approach can be seen to have potential for a variety of other applications as well.

Chapter 7

Suggested Future Work

Although several parameters were examined, several more parameters could be analyzed to provide a more complete evaluation of performance. Modifying the distance between the nozzle and sensor could yield large changes in heat transfer, particularly at the center point. In addition, changing the ratio of the cross-sectional area between the cone entrance and nozzle orifice could affect the stream velocity, which in turn could affect the convective heat transfer. The total mass of the thermite could be modified to evaluate the affects of scaling the system. It has already been seen that the ignition source can have a significant impact, and therefore, a study evaluating different ignition sources could be valuable as well. Last, different thermite mixtures could yield very different results.

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Appendix A

Additional Temporal Derivative Curves

Figure 31. Temporal derivative of 15 degree nozzle test

Figure 32. Temporal derivative of 30 degree nozzle test

Figure 33. Temporal derivative of 45 degree nozzle test

Figure 34. Temporal derivative of 60 degree nozzle test

Appendix B

Additional Calculated Surface Temperature Curves

Figure 35. Surface temperature of 15 degree nozzle test

Figure 36. Surface temperature of 30 degree nozzle test

Figure 37. Surface temperature of 45 degree nozzle test

Figure 38. Surface temperature of 60 degree nozzle test

Appendix C

Additional Calculated Heat Flux Curves

Figure 39. Surface heat flux of 15 degree nozzle test

Figure 40. Surface heat flux of 30 degree nozzle test

Figure 41. Surface heat flux of 45 degree nozzle test

Figure 42. Surface heat flux of 60 degree nozzle test

Appendix D

Residuals of Surface Temperature and Heat Flux Calculations

Figure 43. Residual values for probe 1 at various geometries

Figure 44. Residual values for probe 2 at various geometries

Figure 45. Residual values for probe 3 at various geometries

Figure 46. Residual values for probe 5 at various geometries

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