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IGNITION AND COMBUSTION OF BULK METALS AT NORMAL, ELEVATED AND REDUCED GRAVITY

M.C. BRANCH, J.W. DAILY AND A. ABBUD-MADRID
CENTER FOR COMBUSTION RESEARCH
MECHANICAL ENGINEERING DEPARTMENT
UNIVERSITY OF COLORADO
BOULDER, CO 80309-0427

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ABSTRACT

Knowledge of the oxidation, ignition and combustion of bulk metals is important for fire safety in the production, management and utilization of liquid and gaseous oxygen for ground based and space applications. This proposal outlines studies in continuation of research initiated earlier under NASA support to investigate the ignition and combustion characteristics of bulk metals under varying gravity conditions. Metal ignition and combustion have not been studied previously under these conditions and the results are important not only for improved fire safety but also to increase knowledge of basic ignition and combustion mechanisms. The studies completed to date have led to the development of a clean and reproducible ignition source and diagnostic techniques for combustion measurements and have provided normal, elevated and reduced gravity combustion data on a variety of different pure metals. The research conducted under this grant will use the apparatus and techniques developed earlier to continue the elevated and low gravity experiments, and to develop the overall modeling of the ignition and combustion process. Metal specimens are to be ignited using a xenon short-arc lamp and measurements are to be made of the ignition energy, surface temperature history, burning rates, spectroscopy of surface and gas products, and surface morphology and chemistry. Elevated gravity will be provided by the University of Colorado Geotechnical Centrifuge and microgravity will be obtained in NASA's DC-9 Reduced Gravity aircraft.

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I. INTRODUCTION

Over the past four years, metal combustion studies at the University of Colorado have focused on the effects of gravity (g) on the ignition and burning behavior of a variety of bulk, pure metal specimens. The impetus behind this effort is the interest to understand the flammability properties of structural metals found in a variety of oxygen systems in spacecraft. Since these systems are subjected to higher-than-one g loads during launch and reentry and to a zero-gravity environment while in orbit, the study of ignition and combustion of bulk metals at different gravitational accelerations is of great practical concern. From the scientific standpoint, studies conducted under low gravity conditions provide simplified boundary conditions since buoyancy is removed, and make possible the identification of fundamental ignition mechanisms.

In the early stages of our research effort, a computational model of the proposed experimental system was developed to explore the effects of gravity and pressure on the heating phase of a metal sample under non-oxidizing conditions[1]. The model includes the effects of conduction, convection and radiation heat transfer in the solid specimen and the surrounding gas. The results from the model predict a decreasing value on the steady-state temperature of the metal specimen (in an asymptotic fashion), slower heating rates and progressively longer delays for ignition as gravity is increased from zero to higher g's.

After completing the normal gravity tests we proceeded to explore the effects of high g's on the ignition and combustion of titanium (Ti) samples[2]. A geotechnical centrifuge was used to generate gravity levels above the terrestrial value. It was found that shorter ignition times and faster burning rates were possible in bulk metal samples subjected to gravity levels greater than 10 g's. This result, contrary to the heat transfer arguments proposed by the model, was attributed to the faster rates of oxygen transport produced by natural convection.

The influence of microgravity on the combustion of bulk metals has been investigated by Steinberg, et al. on a drop tower simulator[3]. All the metals and alloys tested (including titanium, aluminum, magnesium, zinc and iron), using the promoted ignition test, supported combustion in the absence of gravity. The study found that in microgravity, the regression rate of the melting surface of a sample ignited in the bottom is significantly faster than in normal gravity. In this particular case, a faster propagation of a reaction front was possible because the combustion products do not detach as it occurs in upward propagation in 1 g.

Glassman, et al.[4] made a number of theoretical predictions on the possible role of gravity on the burning of magnesium (Mg) and aluminum (Al) droplets by developing a quasi-steady, vapor-phase diffusion flame theory including flame radiation, transport of condensed oxide products, and evaporation of the metal. They argued that when the amount of condensed oxide in the flame front increases, its outward transport becomes governed by gravity, the interaction of the external flow field with the gas layer surrounding the flame, by inertial forces experienced by the droplet and by collisions between droplets.

To date, no experimental work has confirmed the above theories by studying the ignition of bulk metals on low gravity fields. This preliminary study is a first step to fill this void by presenting the first experimental findings on the effect of reduced gravity on the ignition of metals in bulk form.

II. OBJECTIVES AND APPROACH

For this phase of our investigation, the primary objective is to collect the first evidence on the effect of reduced gravity on the ignition and combustion characteristics of bulk metals. It was decided early on the design process to use one of NASA's experimental aircraft as a microgravity simulator to provide the required amount of reduced-gravity time to heat up and burn the metal samples (approximately 20 seconds). In consequence, an experimental apparatus had to be designed to withstand the variable g environment in the aircraft, provide a totally automated operation of the process to reduce human interaction and error, and collect as much information as possible during the brief periods of reduced gravity allowed in the aircraft. Details of the experimental system used are given in the next section. The analytic techniques to be used in the study are: a) metal surface thermometry, b) surface and flame visualization, c) emission spectroscopy, and d) surface morphology and chemical analysis. The metals selected for this study, titanium (Ti) and magnesium (Mg), were chosen because of their importance as elements of structural metals and their simple chemical composition (pure metals instead of multi-component alloys to avoid complication in morphology and spectroscopic studies). These samples were also chosen to study the two different combustion modes experienced by metals: heterogeneous or surface oxidation and homogeneous or gas-phase reaction. In addition, these specimens exhibit different melting and boiling temperatures, heats of combustion, oxide formation processes and flame temperatures.

The experimental approach provides surface temperature profiles, transition, critical and ignition temperature values, pressure records, spectroscopic measurements, surface morphology, x-ray spectrometry of metals specimens and their combustion products, and high-speed cinematography of the heating, ignition and combustion stages of the metal specimen. These techniques are applied for both normal and low gravity conditions.

III. EXPERIMENTAL SYSTEM AND PROCEDURES

Figure 1 shows a schematic of the various components used in the Ignition and Combustion of Metals (ICOM) experiment. This system is used in both the normal and the low gravity tests. The non-intrusive, ignition source consists of a 1000 W xenon lamp with 250 W of effective broadband output radiation power. The high-intensity light comes out of the lamp in a highly collimated beam and is then intercepted by an aspheric lens. The beam is then focused down to a 4 mm spot on the top surface of a metal sample providing 1.75 MW/m^2 power density. An electric shutter was installed to block the light beam for precise sample heating.
The cylindrical metal specimen is 4 mm in diameter and 4 mm high and it sits on top of an alumina holder that rests in a adjustable ceramic pedestal. A 4.5-liter, stainless steel, cylindrical combustion vessel houses the lens, metal specimen, sample holder, and pedestal. Input radiation is allowed from the top quartz window. Optical access for the movie camera and spectrograph is provided through two fused-silica side windows. Another side window is used as the main access to the interior of the vessel for introduction and removal of samples. A pure-oxygen environment (99.5% min. O_2) is used at an absolute pressure of 1 atm. Evacuation and filling of the vessel is accomplished with the gas supply and vacuum system, which consists of a series of computer-controlled solenoid valves, manual ball valves and safety relief valves.

The chamber pressure is monitored by a solid-state piezoresistive pressure transducer. The metal specimen temperature is measured by type R thermocouples (0.125 mm diameter). Surface temperatures are measured by a thermocouple attached to the cylinder wall on the bottom of the sample. Another thermocouple is used in different spots to measure the thermal gradients inside the sample.
A high-speed, 16-mm motion picture camera provides surface and flame visualization, and estimates of metal burning rates. With a 7.5° shutter and speeds up to 500 frames per second, it provides exposure speeds as high as 1/20,000 sec. The film type used is a tungsten, color negative film, ASA 100 with normal-speed pitch and developed under normal processing. A 50-mm lens and various extension tubes are used for image magnification.
In addition to visible light imaging, time and space resolved spectral information on combustion products is obtained with an imaging spectrograph and a diode array detector. The spectrograph is equipped with motorized gratings for a variety of wavelength ranges and resolutions. The detector consists of a 1024 diode array with a 150 Hz readout rate and a 15 bits ADC resolution.

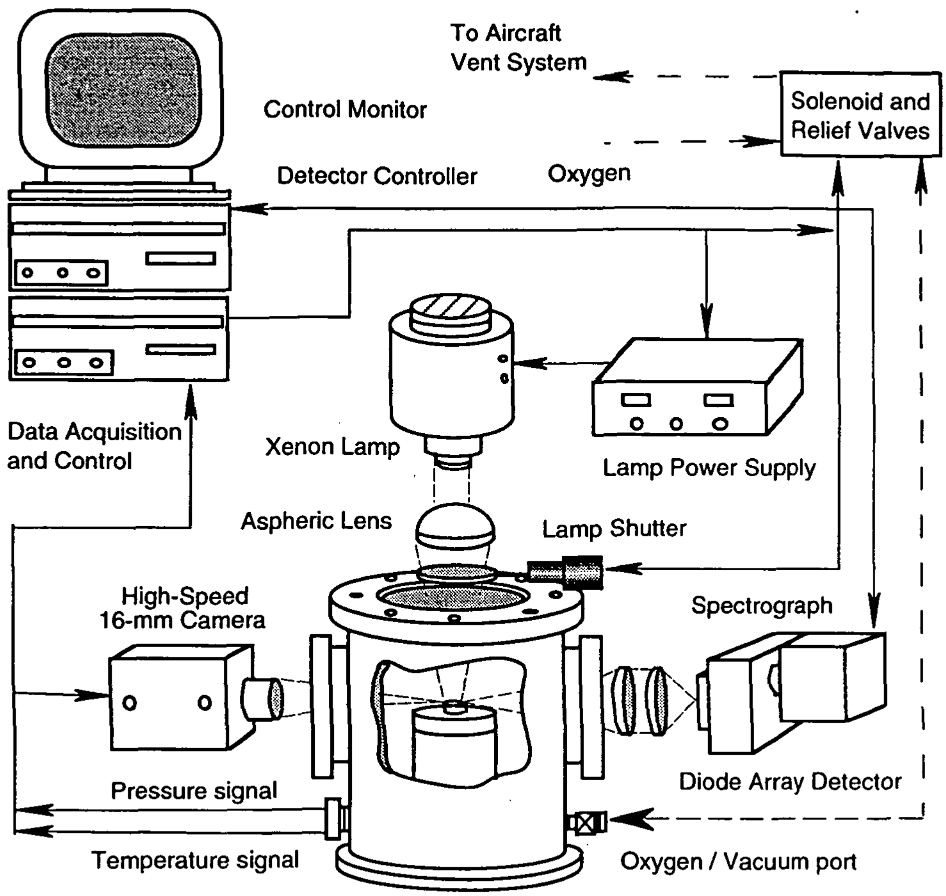

Figure 1. Experimental system used in the Ignition and Combustion of Metals (ICOM) research project.

The main data acquisition and control system consists of a computer, digital/analog data acquisition and control board and a signal conditioning unit. This system triggers the various events, times the experiment and records the temperature, pressure, energy input, luminosity, and gravity level throughout the test. A graphical software is used as interface. After a thorough inspection by NASA engineers, the complete rig was certified to fly onboard the DC-9 Reduced Gravity Aircraft in NASA Lewis Research Center in Cleveland, Ohio.

On a typical low-gravity run the clean metal sample is placed on top of the ceramic pedestal. After closing the combustion chamber, five evacuation and filling cycles are executed to provide a pure-oxygen atmosphere inside the vessel. The lamp is turned on to the desired output power and the computer signals the shutter to open when the target gravity level is achieved. Data acquisition starts and the signal from the thermocouple is used to trigger all the events: camera on/off, diode array acquisition, timing signals and shutter off. Immediately after ignition the lamp is turned off to remove all external heating to the sample. Temperature, pressure, spectral output and visible event are monitored throughout the experiment. After complete combustion, the final pressure is recorded to quantify oxygen consumption and the burned samples are stored for later analysis for chemical composition and surface morphology.

IV. RESULTS AND DISCUSSION

The first series of reduced gravity experiments was recently conducted onboard the NASA-Lewis DC-9 Research Aircraft. All tests were performed under g levels in the +/- 0.02 g's range as recorded from measurements using a three-axis accelerometer near the combustion chamber. The following section discusses some of the preliminary results on the effect of reduced gravity on the heating, ignition and combustion phases based on thermocouple records, high-speed movies, pressure records and spectroscopic measurements.

A. HEATING AND IGNITION BEHAVIOR

The results obtained on the effects of gravity on the heating and ignition of bulk metal samples show profound differences between the two type of metals under investigation.
Figure 2 shows the temperature history of typical Mg samples being radiatively heated at 1 atm under normal and low gravity.

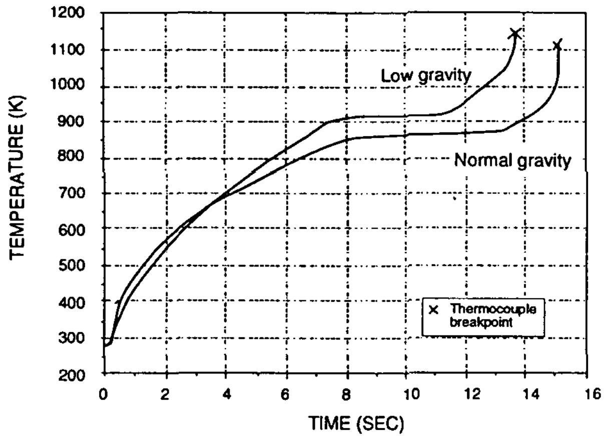

Figure 2. Temperature profiles of Mg samples during the heating and ignition stages at normal and low gravity.

At the beginning of the heating period, a steep rise in surface temperature on the 1-g case caused by the sudden flux of radiant energy from the lamp is immediately followed by a sharp decrease in the heating rate due to the onset of conduction, convection and radiation heat transfer from the surface. A more gradual decrease in the curve is observed in the low-g case due to the absence of convective heat loss. After a few seconds of heating, the transition temperature of the metal is achieved (750 K) after which a clearly visible, white oxide layer starts forming rapidly during linear oxidation. Since the thermocouple is located in the bottom of the sample, initial melting on the top surface of the metal is recorded at a higher temperature in the low-g case; this is due to less surface temperature differences in the absence of convective heat losses. The same physical phenomenon explains the faster phase transition on the sample subjected to low-g's. After the melting process is complete, the temperature of the surrounding solid metal oxide layer rapidly resumes its rise creating a larger temperature difference with respect to the molten metal inside. Liquid metal expansion and large thermally-induced stresses eventually break the thin oxide crust causing rapid evaporation of the metal and subsequent fast reaction with the oxygen gas surrounding the sample. Apparently, under our experimental conditions, the faster ignition of bulk Mg samples at low-g can be explained based on purely heat transfer arguments (as predicted by the computer model explained in the Introduction section) regardless of the heat generated by surface reactions.

At this point it is worthwhile to explain the fact that in both the 1-g and low-g cases, a higher critical and ignition temperature is obtained in comparison with all the previous ignition studies done on Mg[5]. This disagreement is probably due to the high heating rate provided in our study (100 K/sec in the early stages), a rate two orders of magnitude greater than most reported rates in past investigations. The fast heating rate applied to the top surface of the metal creates significant thermal gradients between the surface and the interior of the sample. In addition, the surrounding gas remains at a temperature close to ambient during the entire heating phase. The phase transition occurring at 930 K acts as an additional heat sink as the metal melts from top to bottom. In contrast to this significant increase on heat losses from the specimen, the high applied heating rate does little to increase the heat generated by oxidation which varies linearly with time. This imbalance between heat losses and gains shifts the critical and ignition temperatures upwards. The critical temperature (or temperature at which the heat generated by metal oxidation first exceeds the heat lost through conduction, convection and radiation) becomes the melting point of Mg but only after the phase transition has occurred. If the heat source is turned off at the beginning of the melting process, the sample rapidly cools down to ambient temperature, eluding ignition. If the lamp is turned off only after complete melting is achieved, ignition will occur after a 3-4 second delay. Due to the fast heating rate, the temperature of the oxide surface continues rising after reaching the critical temperature until the expanding liquid metal and thermal stresses cause ignition, i.e., the appearance of the first flame on top of the sample.

A completely different behavior is observed in the case of Ti. Figure 3 shows the temperature history of typical Ti samples being heated at 1 atm under normal and low gravity. In both cases there is an initial steep rise in temperature that is followed by a sharp decrease in heating rate caused by the onset of heat transfer mechanisms as seen in the magnesium case.

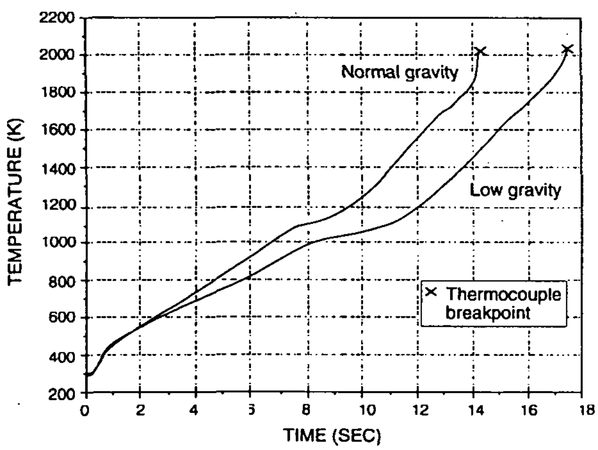

Figure 3. Temperature profiles of Ti samples during the heating and ignition stages at normal and low gravity.

The temperature rise continues monotonically until a small but noticeable change in the slope around 1100 K. This change corresponds to a solid phase transition. The latent heat associated with this crystal structure change from an hexagonal close-packed structure to a body-centered cubic lattice (4.4 kJ/mol) is responsible for the slope change in the temperature profile. A significant increase in the temperature rise follows this phase transition due to the change in the oxidation mechanism from parabolic to linear. This point corresponds to the transition temperature. The reason for the transition may be due to mechanical stress cracking at a critical oxide thickness, oxide porosity and to the non-isothermal nature of the fast heating process. After this point, the temperature continues to increase until a point of inflection is reached in the curve after which the temperature vs. time slope rapidly increases again. This point is identified as the critical temperature (1700 K). Beyond the critical temperature, the metal specimen is driven into a thermal runaway region as a consequence of the exponential dependence of the reaction rate on temperature. After a time delay, an ignition temperature close to the melting point of the metal is reached (around 1910 K). This corresponds to the appearance of a bright, luminous reaction zone on top of the metal sample.

The difference between the normal gravity and the low-g curves becomes significant after reaching the transition temperature. In contrast with Mg, the low-g curve experiences slower temperature rise and longer ignition times. This behavior may be explained by the role of gravity on inducing convective flow around the sample. At normal gravity, increased convection leads to an increase of oxygen transport to the metal surface resulting in higher oxidation rates and also faster absorption of oxygen in solution with titanium which may increase the rate of internal reactions within the sample. This process is significantly enhanced during the linear oxidation stage. In this case there is no solid-to-liquid phase transition (until 1939 K) and heat losses increase at a slower rate than heat gains by oxidation. With an exponential dependence on temperature, heterogeneous reactions soon become self-accelerating in this high temperature, low heat loss condition, promoting a faster temperature rise. Figure 3 clearly illustrates the quick divergence of the curves shortly after reaching the transition temperature. Critical temperatures are similar in both cases and the difference between ignition temperatures is solely due to the longer time that it takes the combustion front to reach the thermocouple in the low gravity case (see next section).
The differences found on the ignition behavior of Ti in the normal and low-g cases are in agreement with the results obtained at higher-than-normal gravity levels where even faster ignition times were observed after 10 g's [2].

B. BURNING BEHAVIOR

As with their ignition characteristics described above, the burning of bulk Ti and Mg specimens presented totally different features. In the case of Mg, a gas-phase ignition wave propagated initially through the sample at 100 mm/sec. Immediately after the complete surrounding of the specimen by the diffusion flame, explosions occur at different spots due to the break up of the initial oxide layer by the metal vapor rushing out to the flame front. High-speed movies clearly show a diffusion flame of reacting metal vapor and oxygen detached 3 mm from the surface. Time and space resolved spectroscopic measurement revealed the existence of not only the green and UV bands of MgO caused by chemiluminescence, but also several lines of Mg, showing the existence of dissociation of oxide at the high flame temperatures. In the normal gravity case, most of the condensed oxide products formed in the flame are carried upwards by convection and deposited on the quartz window protecting the aspheric lens. The complete burning of the sample took one and a half seconds, leaving few remnants of the original oxide layer on the alumina holder.

In the case of low gravity, without the presence of a convective plume, the condensed oxide products remain on the flame front causing rapid agglomeration and intense heat radiation, as predicted by the Brzustowski-Glassman model[4]. Eventually, some particles formed in the lower part of the sample stick to the alumina holder and form a circular anchoring point. This attracts a growing number of particles which rapidly start building an oxide wall around the original space occupied by the sample. These formations redirect the metal vapor jets that continue producing oxide condensates that later find their way back to the sample due to the bulk motion of oxygen towards the reacting front. After all the metal is consumed the majority of oxide formed stays in the holder in the form of walls of fused magnesia with an outer coating of white flakes and hollow spheres. The agglomeration and accumulation of condensed oxides and the reduced transport of oxygen to the reaction front caused by the absence of buoyancy, significantly slows down the burning of the metal sample to approximately half the value at normal gravity.

In the case of Ti the ignition starts in a point on the outer rim of the top surface and evolves into a liquid mixture of metal, oxygen and oxides which propagates in a smooth, non-explosive fashion with the apparent absence of gaseous products as evidenced by the spectroscopic measurements. In normal gravity, the self-sustained heterogeneous reaction initially propagates downward through the specimen at approximately 25 mm/sec. This rate increases afterwards due to the downward pull caused by gravity on the molten product. The sample melts from top to bottom and the luminosity rises steeply achieving the highest brightness during the combustion phase. After meltdown, Ti exhibits a more vigorous reaction with random outward expelling of small particles. The shower of particles starts slowly and rapidly transforms into a continuous spray with flying particles showing secondary fragmentation. Emission spectra taken during this phase show line and band features from oxides and metal vapor. The combustion event lasts a third of a second.

In the case of low gravity, a spherical molten ball forms in the top of the sample and propagates uniformly and slowly through the sample at 12 mm/sec, half the rate observed in normal gravity. The spherical shape of the molten blob is maintained throughout until the liquid touches the alumina holder. At this point the liquid-solid surface tension force pulls down the molten products with a violent shower of particles following afterwards.
Pressure records indicate a 2% rise in pressure during combustion and a decrease of the same magnitude after the products have cooled down. This provides further indication that oxygen gas went into forming solid oxides after combustion.

V. CONCLUSIONS

An investigation on the effects of low gravity on the ignition and combustion characteristics of bulk metals has been conducted. To our knowledge, this is the first experimental attempt to explore and analyze the ignition behavior of bulk metals in a simulated spacecraft environment. The experimental configuration used consists of bulk Ti and Mg cylindrical samples ignited by radiation from the top in pure-oxygen environments at 1 atm pressure. A fully automated system controls the timing and occurrence of all the different events of the experiment while providing at the same time a fast acquisition of all the important variables of the process. Surface thermometry, pressure records, spectroscopic measurements, and high-speed movies are some of the diagnostic tools used to characterize the variety of phenomena encountered in metal burning experiments.
Preliminary results obtained from the normal and low gravity tests show a series of significant differences under the two gravity regimes. Diametrically opposed behavior is also found for the two metals chosen for this study, Ti and Mg. In the case of Mg samples, faster ignition times (by 15%) and slower burning rates (by 100%) were found at low-g. It is believed at this point that Mg ignition is mostly affected by the removal of convective heat losses in the absence of gravity. The reduction of oxygen transport to the flame front and the build-up of combustion products are believed to be responsible for the slower burning rates encountered at low-g. For Ti, slower ignition times (by 20%) and slower burning rates (by 100%) in reduced gravity are observed. The former are mostly due to the decrease of heat generated by surface reactions during the pre-ignition heating phase, while the latter may be attributed to the lack of convective flow of oxygen to the reaction front as in the case of Mg.
A spinoff of this investigation has been the study of the effect of high applied heating rates on critical and ignition temperatures. The biggest effect is seen with the Mg specimens where both temperatures are significantly shifted upwards due to the imbalance created between heat loss and heat generation mechanisms. These findings only point out to the strong dependence of metal combustion on the experimental configuration used. Research on this area continues with further experiments to be conducted on low-gravity and with the developing of a general model that can reproduce the results under our particular experimental conditions.

VI. FUTURE RESEARCH

Future research will use the appartus and techniques developed here to continue normal, elevated, and microgravity measurements, and to develop the modeling of the overall ignition and combustion process for additional bulk metals. The normal gravity experiments have provided new spectroscopic and electron microscopic data on a number of pure metals. The elevated gravity measurements are to be continued in the geotechnical Centrifuge operated by the University of Colorado. Metal combustion has already been achieved and monitored in the centrifuge for a small number of specimens. Since the time required for heating, ignition and combustion of the metal specimens is typically 20-25 secs, the microgravity environment will continue to be provided by NASA's DC-9 Reduced Gravity aircraft. The modeling studies will focus on the description of the essential effects of gravity body forces on the ignition and combustion process and the details of the ignition mechanism.

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IX. PERSONNEL AFFILIATED WITH THIS RESEARCH

1. Melvyn C. Branch, Professor of Mechanical Engineering, Co-Principal Investigator.
2. John W. Daily, Professor of Mechanical Engineering, Co-Principal Investigator.
3. Angel Abbud-Madrid, Graduate Research Assistant, Ph.D. Candidate.
4. Wesley T. Ramm, Undergraduate Student.
5. David T. Bunting, Undergraduate Student.