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Normal-Gravity Test Results**

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IGNITION AND COMBUSTION CHARACTERISTICS OF PURE BULK METALS: NORMAL-GRAVITY TEST RESULTS

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Abstract

An experimental apparatus has been designed for the study of bulk metal ignition under elevated, normal and reduced gravity environments. The present work describes the technical characteristics of the system, the analytical techniques employed, the results obtained from the ignition of a variety of metals subjected to normal gravity conditions and the first results obtained from experiments under elevated gravity. A 1000 W xenon short-arc lamp is used to irradiate the top surface of a cylindrical metal specimen 4 mm in diameter and 4 mm high in a quiescent pure-oxygen environment at 0.1 MPa. Iron, titanium, zirconium, magnesium, zinc, tin, and copper specimens are investigated. All these metals exhibit ignition and combustion behavior varying in strength and speed. Values of ignition temperatures below, above or in the range of the metal melting point are obtained from the temperature records. The emission spectra from the magnesium-oxygen gas-phase reaction reveals the dynamic evolution of the ignition event. Scanning electron microscope and x-ray spectroscopic analysis provide the sequence of oxide formation on the burning of copper samples. Preliminary results on the effect of higher-than-normal gravity levels on the ignition of titanium specimens is presented.

Introduction

Early research on combustion of metals was motivated by the knowledge of the large heat release and corresponding high temperatures associated with metal-oxygen reactions.

The advent of space flight brought about an increased interest in the ignition and combustion of metallic particles as additives in solid rocket propellants. More recently, attention has been given to the flammability properties of bulk, structural metals due to the high number of accidents involving burning metal components in high-pressure oxygen systems¹.

Less attention has been given to the ignition phenomena of bulk metals, i.e., the mechanisms that lead to a self-sustaining exothermic reaction. As with combustion studies, the majority of the early work was concentrated on the ignition of finely divided powders^{2,3}. The promoted ignition test developed by the NASA White Sands Test Facility utilizes easily ignitable metals or hydrocarbons to ignite a cylindrical metal rod on the bottom end for upward propagation studies⁴. This method has the disadvantages of the contaminating effect of the reaction between the ignition source material and the metal sample, and the poor estimate of the ignition energy transferred to the metal. Non-intrusive techniques have also been used to determine ignition behavior. Brzustowski and Glassman⁵ explored ignition by direct electric heating of metal wires and ribbons. The ignition point was determined by the balance of the rate of heat produced and the rate of heat removed from the metal sample. Bransford⁶ used a carbon dioxide (CO₂) laser to ignite metals and alloys. He defined the ignition point by monitoring the mass as a function of time. A large mass increase was found at this point due to accelerated oxidation. Nguyen and Branch⁷ utilized a CO₂ laser and a two-color pyrometer to determine the surface temperature of aluminum, 302 stainless steel and 1018 carbon steel specimens. Ignition was defined as the point at which there exists an abrupt change in the slope of the light intensity curve detected by the pyrometer.

These three studies show the importance of utilizing a non-intrusive ignition source which also provides a close estimate of the heat input flux. Nevertheless, it is evident from the results of these

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investigations that there is a lack of common agreement regarding the definition of an ignition point. The difficulty of providing an accurate ignition model is complicated by factors such as: metal type, oxygen pressure, formation of protective oxide layers, experimental apparatus and configuration, gravitational force and sample size. As a consequence, there is a strong need to provide a fundamental study that identifies all the important variables that characterize the ignition phenomena.

The following work represents a preliminary study that is part of a broader research effort aimed at providing further insight into this multi-variable dependent phenomena by looking at the effects of gravity on the ignition behavior of metals. Since spacecraft are subjected to higher-than-1g gravity loads during launch and reentry and to zero-gravity environments while in orbit, the study of ignition and combustion of bulk metals at different gravitational potentials is of great practical concern. From the scientific standpoint, studies conducted under microgravity conditions provide simplified boundary conditions since buoyancy is removed and make possible the identification of fundamental ignition mechanisms. The effect of microgravity on the combustion of bulk metals has been investigated by Steinberg, et al.⁸ using a drop tower simulator. However, no quantitative work has been found on the ignition of bulk metals at lower or higher-than-1g gravitational fields. This preliminary experimental study presents the results obtained on ignition at normal gravity and the first findings on the effect of high gravity loads on the ignition of bulk metals by using a geotechnical centrifuge.

Objectives and approach

The primary objective of this investigation is the development of an experimental system capable of providing fundamental physical and chemical information on the ignition of bulk metals under different gravity levels. A feasibility study on the use of a continuous radiation short-arc lamp as an ignition source is given in reference 9. 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. Technical details on the combustion chamber and the techniques described above are given in the Experimental Apparatus and Procedures section. The metals tested in this study, iron (Fe), titanium (Ti), zirconium (Zr), magnesium (Mg), zinc (Zn), tin (Sn),

and copper (Cu) were selected 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 a wide range of melting and boiling temperatures, heats of combustion, oxide formation processes and adiabatic flame temperatures. High-purity metals (99.95% and above) were used for this investigation. The experimental approach provides surface temperature profiles, 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. The first phase of this project is intended to provide an experimental database of ignition of metals under normal gravity conditions. This information will be later compared to the results of experiments conducted under elevated gravity (second phase) and at microgravity levels (third phase). This paper contains the results from the first phase and preliminary results from the second phase.

Experimental Apparatus and Procedures

The experimental system is shown in Figure 1. The radiation source consisted of a 1000 W xenon short-arc lamp with 250 W of effective broadband output radiation power (with approximately 42% of output power between 800-1200 nm and the rest between 200-800 nm).

The high-intensity, non-coherent light comes out of the lamp in a highly collimated beam (4° half-angle) and is then intercepted by an aspheric lens (8 cm diameter, 6 cm focal length) which provides the maximum light collection efficiency for the available focal length under these conditions. The beam is then focused down to a 4 mm spot on the top surface of a metal sample providing approximately a 1 MW/m² power density. The cylindrical metal specimen is 4 mm in diameter and 4 mm high and it is held in place by an alumina-silica holder that rests in a ceramic pedestal. These components (lens, specimen and pedestal) are located inside a 4.5 L stainless steel cylindrical chamber which permits input radiation through the top quartz window. Optical access for the camera and spectrograph is

provided through three fused-silica side windows. An oxygen environment (99.5% min O₂) is used in all tests at an absolute pressure of 0.1 MPa. The absorption of radiation by O₂ at this pressure and spectral range is negligible.

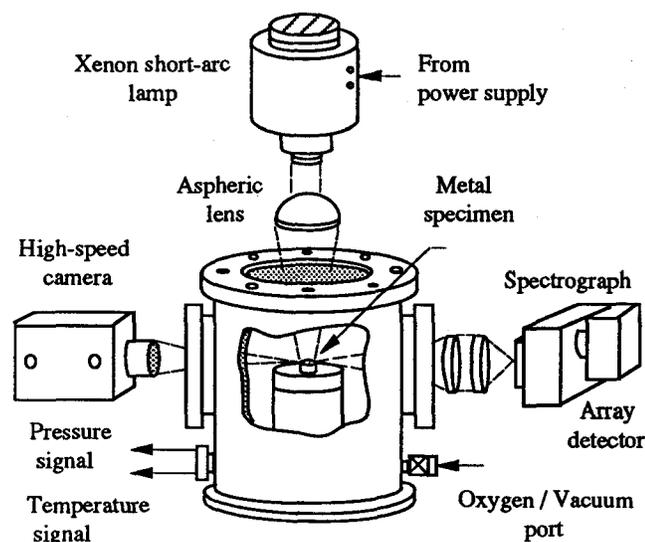


Figure 1. Experimental apparatus

The chamber pressure is monitored by a solid-state piezoresistive pressure transducer. The specimen surface temperature is measured by a Type B thermocouple (0.125 mm diameter) located in the center of the sample at approximately 0.5 mm below the top surface. A Teledyne DBM-45, 16 mm high-speed motion picture camera provides a qualitative visual record of the combustion event and an estimate of flame propagation rates. In addition to visible light imaging, time-resolved spectral information about the reacting species and products of combustion was obtained by a Jarrell-Ash Monoscope 27 Spectrograph and a Princeton Instruments IRY-700 diode-array detector. Surface morphology analysis is obtained with a Cambridge Stereoscan 250-Mk3 scanning electron microscope (SEM). Quantitative analysis of the chemical composition of quenched samples and combustion products is performed with a JEOL JXA-8600 electron microprobe (EMPA) with combined wavelength dispersive spectroscopy (WDS) and energy dispersive spectroscopy (EDS). A silicon photodiode is used to record total luminosity values and to monitor the light output from the xenon lamp. The high-gravity experiments were conducted on the University of Colorado Geotechnical

Centrifuge that is equipped with an onboard computer and signal conditioning system enabling the remote recording of temperature, luminosity and pressure.

Prior to the experimental run, the metal sample is thoroughly cleaned and placed on top of the ceramic pedestal. Care is taken to ensure good contact between the metal and the thermocouple. After closing the combustion chamber, three 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 light beam is focused on top of the metal specimen. 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 combustion products are analyzed for chemical composition and surface morphology. A minimum of 5 samples of each metal are tested to ensure experimental repeatability.

Results and Discussion

Qualitative Observations

A visual record of all experimental runs revealed the existence of three important and distinctive phases:

a) The heating period started with a sudden thermal expansion of the top surface of the specimen after radiation was applied. Surface structure changes were also detected by the variation of the radiation characteristics of the sample. These surface changes are related to the allotropic nature of some metals in the solid phase and the metal oxide layer formation.

b) The ignition phase was characterized by rapid oxidation and a noticeable increase in energy release evidenced by the intense radiation emitted from the reaction zone.

c) The combustion event varied in speed and strength among the different metals tested. In the case of Fe, Ti, and Zr, a well defined self-sustained downward propagation of the reaction zone was accompanied by a violent exothermic reaction including random outward expelling of small particles. The detection of intense luminosity was due to the high temperatures (3000-4000 K) reached in the reaction zone.

For Mg and Zn the combustion phase evolved much faster with the generation of a fine oxide powder that formed a dust cloud which deposited uniformly on the walls and windows of the combustion chamber. This behavior may suggest the existence of volatile metal or metal oxide products. In contrast with the other metals studied, Cu exhibited a smooth combustion phase characterized by a slow regression rate with a low-luminosity reaction zone. This behavior may be due to the low heat of combustion of Cu in the presence of high heat losses by conduction, convection and radiation. This in turn results in a low flame temperature and a slow reaction rate that give the observed slow propagation and low luminosity.

Temperature Profiles

Temperature records also revealed uniquely the existence of the heating, ignition and combustion stages. Figures 2 and 3 show the temperature histories of all the metal specimens studied at 0.1 MPa. In all cases there is a steep rise in surface temperature in the first second of heating caused by the sudden flux of radiant energy from the light source. This increase in temperature in turn generates conductive, convective and radiative heat transfer from the upper surface of the sample that significantly lowers the rate of temperature increase. In the case of Fe, Ti and Zr (Figure 2) this rate continues monotonically until a point of inflection is reached in the curve after which the temperature vs. time slope rapidly increases again. This transition point is identified as the temperature at which the heat generated by the metal oxidation first exceeds the heat lost through conduction, convection and radiation. This temperature is defined in this investigation as the ignition temperature of the metal sample. For Fe, Ti and Zr the ignition temperature occurs below their melting point.

Beyond the ignition temperature, the metal specimen is driven into a thermal runaway region as a consequence of the exponential dependence of the reaction rate on temperature. The acceleration of the reaction rate ultimately leads to the exothermic, self-sustained combustion event. At this stage, the temperature drastically increases to values in the range of 3000-4000 K depending on the metal type. As shown in Figure 2, the thermocouples are unable to follow the temperature increase in this last stage because their maximum operating temperature is around 1750 K. A small but noticeable change in the slope around 1100 K can also be detected in the case of Ti and Zr samples. This change corresponds to a solid

phase transition on both metals (Ti at 1155 K and Zr at 1135 K). 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 for Ti and 3.9 kJ/mol for Zr) is responsible for the slope change in the temperature profiles. The similar behavior encountered in the heating, ignition and combustion phases of Ti and Zr may be related to both metals being elements of Group IVb in the periodic table. Figure 3 shows the temperature histories for the Sn, Zn, Mg and Cu samples.

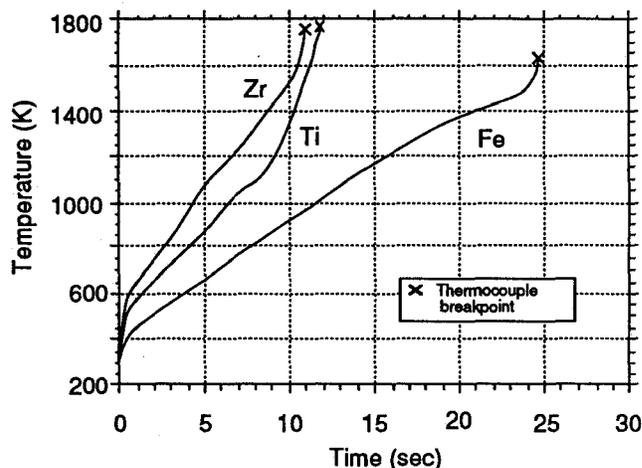


Figure 2. Temperature vs. time behavior of iron (Fe), titanium (Ti) and zirconium (Zr).

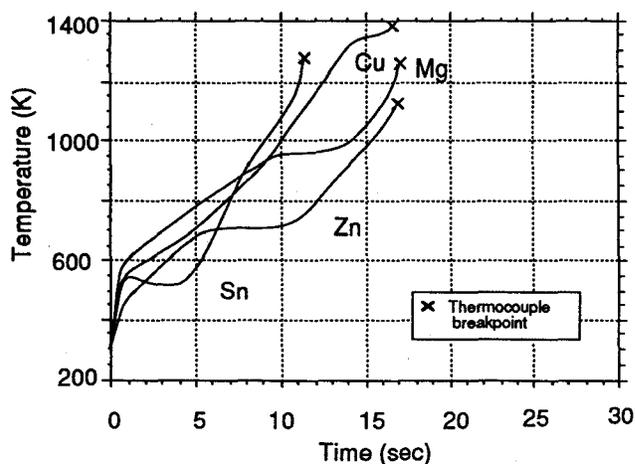


Figure 3. Temperature vs. time behavior of tin (Sn), zinc (Zn), magnesium (Mg) and copper (Cu).

In contrast with the results from Figure 2, these profiles show an intermediate plateau which corresponds to the solid-liquid phase transition point. The heat released by oxidation at this point is still lower than the heat lost by the specimen and the temperature continues to increase almost exclusively by the radiant flux from the xenon lamp. Eventually the ignition temperature is reached and the molten metal confined by the outside oxide layer bursts out and exothermically reacts with the oxygen gas. In the case of Mg and Zn the ignition temperature is so close to the boiling temperature of the metal that there is probably volatilization of the metal or the oxide product, explaining the existence of the metal oxide particle cloud that forms after ignition. This combustion behavior has been observed before with Mg, Zn and Al rods using the pressurized oxygen index apparatus¹⁰. For Cu, its ignition temperature coincides with the melting temperature of the metal.

Table 1 shows the heat of combustion, melting, ignition, and adiabatic flame temperatures at 0.1 MPa of the different metals used in this investigation. In the case of Fe, Ti and Zr it is evident that the ignition temperature is always below the melting temperature of the metal, whereas the opposite is true for Mg, Zn and Sn.

Spectroscopic measurements

Time-resolved measurements of gas-phase emission spectra have provided a clear, non-intrusive picture of the rapid evolution of reacting species and the formation of oxide products during the ignition and combustion phases of metals burning in a homogeneous flame front. This technique can also give a good estimation of color temperature from the continuum spectrum of the molten material on metals exhibiting surface reactions.

Table 1. Heats of combustion, melting, ignition and adiabatic flame temperatures at 0.1 MPa of the metals studied.

Metal Specimen	Heat of Combustion (kJ/mol) ^a	Melting Temperature (K) ^b	Ignition Temperature (K) ^c	Adiabatic Flame Temp. (K) ^d
Titanium	1520	1939	1670	3993
Zirconium	1097	2125	1555	4278
Tin	738	504	1156	...
Magnesium	601	923	1250	3432
Zinc	432	692	1060	...
Iron	272	1809	1470	3400
Copper	156	1358	1360	...

^a From JANAF Tables¹¹. The heats of combustion correspond to the following metal/metal oxide system: Ti/Ti₂O₃, Zr/ZrO₂, Sn/SnO₂, Mg/MgO, Zn/ZnO, Fe/FeO, Cu/CuO.

^b From JANAF Tables¹¹.

^c From this investigation.

^d From Steinberg, et al.¹²

Ultimately, knowledge of species concentrations in time and space can be useful in determining fundamental information on kinetic mechanisms. Our first effort to implement spectroscopic measurements focused on obtaining the emission spectra of the Mg-O₂ reaction. Previous studies^{5,13} have found that atomic Mg and magnesium monoxide (MgO) have very strong electronic resonances (compared to other metallic atoms and diatomic radicals) and show prominent line and band emissions due to their high concentration in the vapor-phase reaction zone.

In our experimental set-up the volume immediately above the top surface of the metal sample was focused by two condensing lenses onto a 25-micron wide aperture slit at the entrance of the spectrograph. Neutral density filters were used to attenuate the incoming radiation. The output from the spectrograph was collected by a diode-array detector and sent to a computer for data acquisition. The calibration of diode number vs. wavelength was done with a Hg-Ar lamp. All spectra were taken with a 33-microsecond integration time and no signal averaging.

Figures 4 and 5 show emission spectra from the Mg-O₂ reaction at 0.1 MPa under normal gravity conditions. Figure 4 shows a broad spectra taken approximately 100 ms after ignition using a 300 groove/mm grating. This figure shows several bandheads of the MgO system and triplet lines of atomic Mg against a continuum background. This background radiation is produced by oxide molecules condensing in the high-temperature region and being carried away by convection. Due to the relatively high rate of particle condensation and a small rate of diffusion away from the reaction zone at 0.1 MPa, the continuum profile kept increasing with time eventually obscuring the features from the Mg and MgO spectra. This effect appeared to diminish at lower oxygen pressures. Figure 5 shows the time evolution of the gas-phase reaction in 33-ms intervals using a 2400 groove/mm grating to highlight the vibrational features of the green B-X system of MgO. The information provided by this figure becomes very important in order to estimate the location and relative abundance of the different species involved as well as the electronic and vibrational temperatures given by the intensity ratios of individual lines or bands. Comparison of time and space resolved spectra will be very useful on determining the effect of gravity-induced convection on the nature and character of ignition phenomena of bulk metals exhibiting gas-phase reactions.

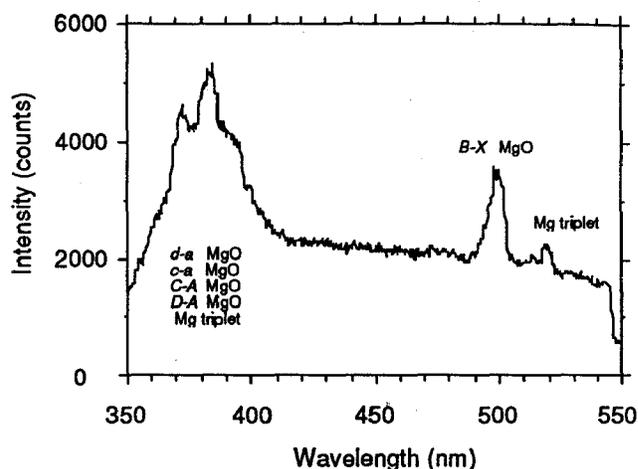


Figure 4. Emission spectra from the ignition of a Mg specimen in O₂ at 0.1 MPa taken approximately 100 ms after ignition.

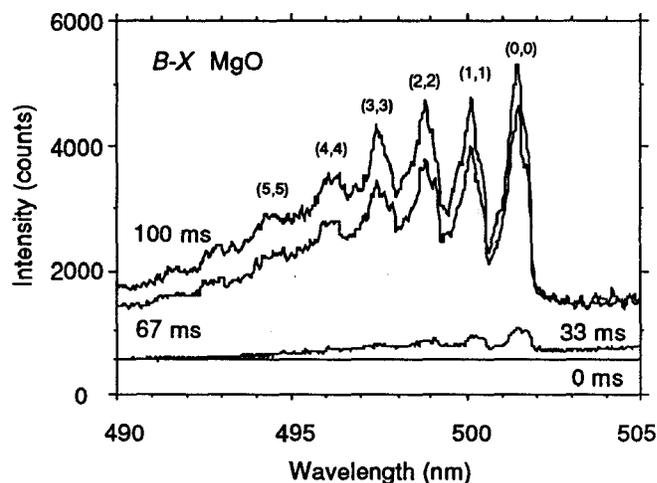


Figure 5. Time-resolved emission spectra from the ignition of a Mg specimen in O₂ at 0.1 MPa taken in 33-ms intervals to highlight the vibrational features of the green B-X system of MgO.

Surface Morphology and Chemical Analysis

As seen from the different behavior of all the metals studied, the character of the surface has a major effect on the nature of ignition and burning. By analyzing the surface morphology before and after combustion, one may determine the effect of surface structure.

On the other hand, the ignition of many metals involves the formation of solid or molten oxides of the metal on the surface of the heated specimen through which oxygen subsequently must diffuse. A more complete understanding of the ignition mechanism for these metals requires monitoring the formation of these oxide layers and their subsequent chemical transformations. We have conducted qualitative morphological scanning of unreacted, partially burned and completely oxidized samples to explore the nature and location of important features. From these observations, a quantitative analysis is performed to identify the chemical composition of all relevant surfaces.

Figures 6 and 7 are an example of the valuable information obtained from metallographic photographs to determine the oxide products from the Cu-O₂ reaction. Previous work^{4,10} has classified copper rods as non-flammable even at the highest possible pressure (69.5 MPa in some cases) due to the sudden extinction of the sample after ignition on upward propagation studies at normal gravity using the promoted combustion test (with 3.2 mm diameter rods). Under our experimental configuration, clear evidence of ignition and subsequent downward propagation on copper specimens (5 mm diameter rods) was obtained at a pure oxygen pressure of 0.1 MPa. Surface morphology studies (with SEM) and quantitative electron microprobe analysis (EMPA) were conducted to determine the oxidation sequence of copper by looking at samples quenched during different stages of the heating and burning processes. Figure 6 is a SEM photograph showing a 6000X magnification of the top outside layer of a copper specimen quenched during combustion in a pure oxygen environment at 0.1 MPa. In this highly porous region, copper crystals belonging to the hexoctahedral crystal class appear to have been deposited on a Cu+O matrix by the condensation of copper vapor after the quenching event. Figure 7 shows an EMPA photograph of the surface of a copper sample burned to completion. The presence of mostly Cu₂O agrees with the results reported by Kubaschewski and Hopkins¹⁴ on the oxidation of copper specimens rapidly heated at high temperatures. These metallographic results provide further evidence which complements the aforementioned visual observations of complete oxidation of bulk Cu samples in normal gravity at 0.1 MPa.

We anticipate that the formation and influence of the various oxide layers will be substantially different in other gravity environments. The change of buoyancy-induced free convection will alter the

advective transport, thereby changing the chemical environment immediately adjacent to the metal surface. This will alter the chemical nature of the oxide layer and also possibly influence oxygen penetration and subsequent ignition of the underlying metal surface.

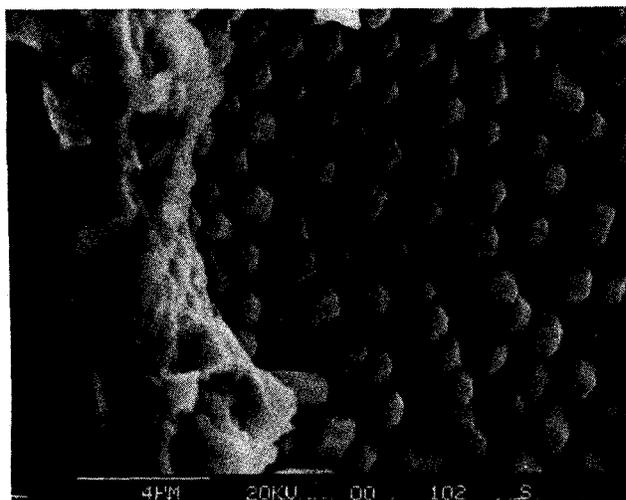


Figure 6. SEM photograph (6000X) taken from the porous top outside layer of a copper specimen quenched during combustion in a pure-oxygen environment at 0.1 MPa.



Figure 7. Electron microprobe photograph (500X) of the surface of a Cu sample burned to completion (mostly Cu₂O).

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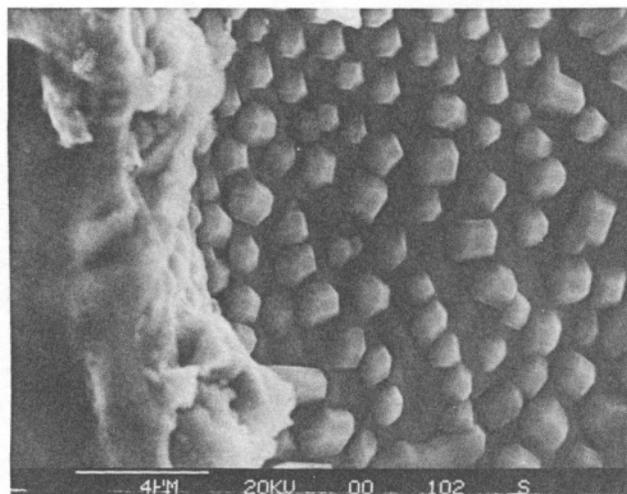


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Preliminary high-gravity results

Recently, the first experiments involving elevated gravity were conducted by using the University of Colorado Geotechnical Centrifuge Facility. The purpose of these tests was to evaluate the response of all components of the experimental system to high gravity loads and to assess the feasibility of remote operation and monitoring. The complete experimental system (excluding the spectrograph) was mounted on the swiveling baskets at both ends of the rotating arms of the centrifuge. An additional silicon photodiode was mounted to monitor any changes on luminosity from the xenon arc lamp that might be attributed to the increased gravity acceleration. All input power and control lines as well as all data signals (photodiode voltage, metal surface temperature and vessel pressure) were transmitted through slippery rings to an outside control panel. The experiment was subjected up to 130 rpm, an angular speed equivalent to an effective 20 g's acceleration. Figure 8 shows the temperature histories of Ti samples subjected to different gravity levels.

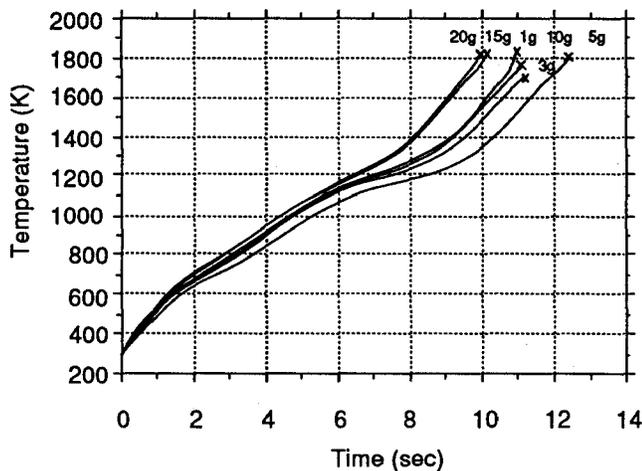


Figure 8. Temperature vs. time behavior of titanium samples subjected to different gravity loads.

As predicted by exclusively heat transfer arguments, the 3g and 5g temperature curves show slower heating rate and a progressively longer delay for the ignition time. A higher buoyancy-induced convective flow increases the heat loss from the metal sample keeping the temperature lower than in

the normal gravity case. However, this trend seems to reverse after the 5g temperature curve, leading to faster ignition times for metal specimens subjected to higher gravity loads. This behavior may be explained by the existence of a competing effect between the convective heat loss and heat generation (by oxidation) mechanisms. At high gravity levels increased convection may lead to an increase of oxygen transport to the metal surface resulting in higher oxidation rates. Higher heat generation (specially at high temperatures) may overcome the heat lost by convection promoting a faster temperature rise. Luminosity curves from the photodiode did not indicate any change on the radiant power output from the xenon lamp.

Further experiments involving different metals, spectroscopic measurements and various oxidizing atmospheres as well as metallographic analysis at different stages during the heating process will give a clearer picture on the effect of higher-than-normal gravity environments on the ignition of bulk metals.

Conclusions

An experimental system to study the ignition characteristics of bulk metals has been designed and tested under normal and high gravity conditions. Ignition and combustion tests were performed on different metal specimens, including Fe, Ti, Zr, Mg, Zn, Sn and Cu. Qualitative observations and temperature profiles have revealed the existence of the heating, ignition and combustion stages. These zones are characterized by well-defined surface structure changes, surface temperature behavior, luminosity and chemical activity. Values of ignition temperature (defined here as the point of inflection in the temperature vs. time curve) below (as in Fe, Ti and Zr), above (as in Sn, Zn and Mg) and in the range of the melting point of the metal (as in Cu) were obtained from the temperature records. Spectroscopic measurements of the Mg-O₂ gas-phase reaction revealed the dynamic evolution of the ignition event by providing time-resolved emission spectra from atomic Mg and the MgO diatomic molecule. Scanning electron microscope studies and x-ray spectroscopic analysis have given information on the oxide formation process of the Cu-O₂ reaction and have provided further evidence of complete oxidation of bulk Cu samples in normal gravity at 0.1 MPa. Preliminary results on the effect of higher-than-normal gravity levels on the ignition of Ti specimens have been obtained by mounting the experimental apparatus on a geotechnical centrifuge. These results

show the existence of an increasing ignition delay for metal samples subjected up to 5g with a trend reversal after this acceleration. Ignition times shorter than those experienced by experiments under normal gravity were obtained for the 15g and 20g cases.

The experimental work is being complemented by modeling studies which include the effects of conduction, convection and radiation heat transfer in the solid specimen and the surrounding gas. The computational model explores the effects of gravity and pressure on the heating phase of the metal sample under non-oxidizing conditions. The heat transfer studies are designed to help evaluate the energy balance on the specimen during heating prior to ignition. Addition of surface and gas-phase reaction processes to the model will make possible a more detailed comparison of the modeling and experimental results.

Future work will focus on completing the centrifuge experiments, obtaining spectroscopic measurements from high-gravity experiments and implementing a compact schlieren system to be used in any gravity environment. The results obtained from the normal and elevated gravity experiments will be later compared with experiments done under microgravity conditions using an aircraft flying Keplerian trajectories.

Acknowledgments

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