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A Dynamic Technique for Thermophysical Measurements at High Temperatures in a Microgravity Environment[1]

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Millisecond-resolution dynamic techniques for thermophysical measurements, when utilized in the laboratory, are limited to the study of materials in their solid phase because the specimen becomes geometrically unstable during melting and collapses, due (at least in part) to the influence of gravity. Therefore, a millisecond-resolution dynamic technique is being developed for use in a microgravity environment in order to extend accurate measurements of selected thermophysical properties of electrically conducting refractory materials to temperatures above their melting point. The basic method involves heating the specimen resistively from ambient temperature to temperatures above its melting point in about 1 s by passing an electrical current pulse through it, while simultaneously recording the pertinent experimental quantities. A compact pulse-heating system, suitable for microgravity simulations with NASA's KC-135 aircraft, has been constructed and initial experiments have been performed to study the geometrical stability of rapidly melting specimens. Preliminary results show that rod-shaped specimens can be successfully pulse-heated into their liquid phase.

KEY WORDS: dynamic techniques; high temperature; liquid metals; melting; microgravity; refractory materials.

1. INTRODUCTION

The development of millisecond-resolution dynamic techniques at the National Bureau of Standards (now National Institute of Standards and Technology), during the past two decades, has enabled the accurate

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measurement of selected thermophysical properties of electrically conducting refractory solids at high temperatures, primarily in the range 1500 K up to their melting point [1–3]. The measured properties include heat capacity, thermal expansion, electrical resistivity, hemispherical total emittance, normal spectral emittance, temperature and energy of solid–solid phase transformations, and melting temperature.

The basic technique involves resistively heating the specimen from room temperature through the temperature range of interest in less than 1 s by passing a large electrical current pulse through it and simultaneously measuring the pertinent experimental quantities, such as specimen temperature, power imparted to the specimen, etc., with millisecond resolution. Because of the short heating time, the technique circumvents, to a large extent, problems associated with specimen evaporation, chemical contamination, large heat transfers, etc., which tend to limit the accuracy of conventional steady-state techniques, particularly at temperatures above 2000 K. This "containerless" technique, however, when used on ground, is limited to measurements on solids only, since at the melting point, the specimen becomes unstable and collapses due, at least in part, to the influence of gravity.

There are two possible options for extending such measurements into the liquid phase: (1) much faster (submillisecond) heating of the specimen [4], which requires performance of the pertinent measurements with microsecond resolution, and (2) the use of millisecond-resolution techniques in a microgravity environment. Due to the much lower heating rates (about three orders of magnitude), the second approach has the potential of yielding significantly more accurate thermophysical data under conditions which better approximate thermodynamic equilibrium.

For these reasons, research has been undertaken to develop a millisecond-resolution dynamic technique for use in a microgravity environment to enable the measurement of selected thermophysical properties of electrical conductors in their liquid phase. The work involves two steps: (1) to establish the criteria for geometrical stability of molten specimens during rapid heating and (2) to demonstrate the applicability of the techniques by performing measurements of selected thermophysical properties of one or more key refractory substances.

In this paper, we describe the design and construction of a compact pulse-heating system suitable for stability studies during microgravity simulations with NASA's KC-135 aircraft [5]. The results of initial flight experiments, which were performed on rod-shaped specimens, are also presented.

2. MEASUREMENT SYSTEM

The measurement system was constructed as two separate units: (1) a compact pulse-heating system (volume, 0.8 m^3; mass, 200 kg), which consists of the experiment chamber and all measuring and recording instruments, and (2) the main battery pack (volume, 0.2 m^3; mass, 135 kg), which supplies the electrical power for pulse heating the specimen. A functional diagram of the measurement system is presented in Fig. 1.

The pulse-heating system includes two major diagnostic instruments: a high-speed framing camera, which records the behavior of the specimen during rapid melting at rates of up to 10,000 frames per second (fps), and a high-speed pyrometer [6], which measures the spectral radiance at 0.65 μm from the specimen surface. The pyrometer signal is recorded by the digital data acquisition system and is also displayed on an analog oscilloscope whose image is relayed by means of mirrors through an auxiliary lens in the side of the camera and recorded on the backside of the film. This arrangement greatly facilitates the analysis of the film since frames in which the specimen is undergoing melting can be readily identified. The data acquisition system, which consists of two digital storage oscilloscopes, also records the current through the specimen as well as the signals from a three-axis accelerometer system. A four-channel time-delay pulse generator controls the sequencing of various events, such as

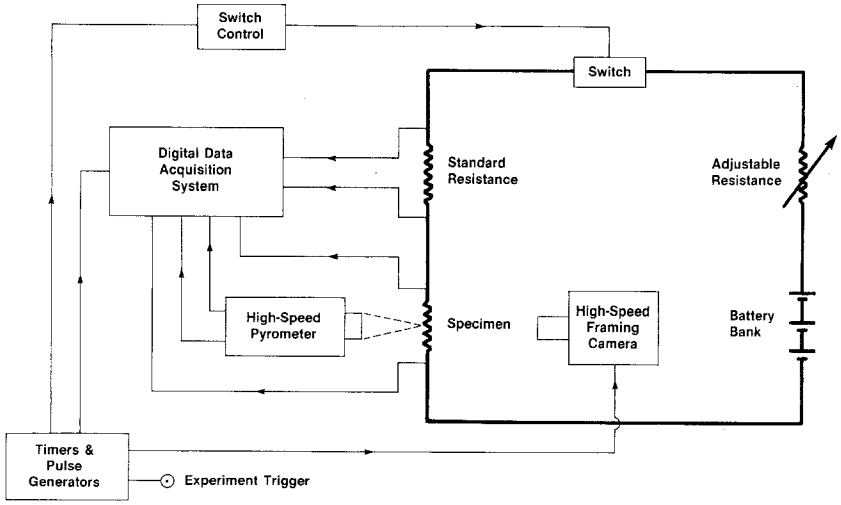

Fig. 1. Functional diagram of the measurement system for microgravity experiments with NASA's KC-135 aircraft. The heavy lines indicate the power-pulsing circuit.

triggering the data acquisition system, closing and opening the fast-acting switch in the power-pulsing circuit, starting and stopping the framing camera, etc. Due to the large current requirements during startup, the framing camera is powered by its own dc battery pack, which consists of 11 series-connected 12-V lead/acid cells (2.5 A · hr each); to avoid possible electrolyte leakage, the cells are hermetically sealed and contain only enough electrolyte to coat all internal surfaces of the cell.

The main battery pack consists of four 12-V lead/acid gel cells (55 A · hr each) which are hermetically sealed and can be operated in any orientation without leakage of the gelled electrolyte. The gel cells are connected into the power-pulsing circuit either as a single 24-V pair or as two 24-V pairs in parallel, depending on the desired heating rate. Further control of the heating rate is provided by an adjustable resistance (thin-walled Inconel tube) in series with the gel cell pair(s).

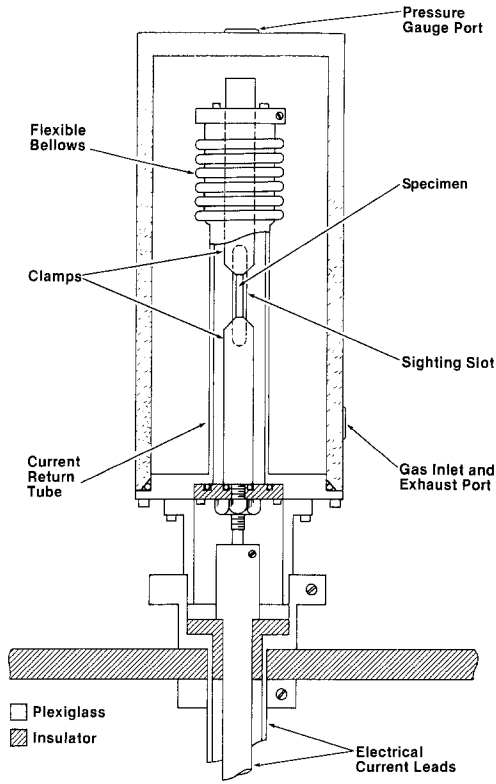

Fig. 2. Schematic diagram of an experiment chamber for studying the geometrical stability of rod-shaped specimens during rapid melting in a microgravity environment.

Figure 2 presents a schematic diagram of an experiment chamber for studying the geometrical stability of cylindrical rod-shaped specimens during rapid melting. The chamber consists of a cylindrical plexiglass cover which can be removed from the chamber base to provide access to the specimen. The specimen is clamped in the base assembly between two electrodes: a lower electrode, which is stationary, and an upper electrode, which is connected to a flexible (phosphor-bronze) bellows to permit axial expansion of the specimen during rapid heating. The bellows are soldered to a cylindrical brass tube which serves as the current return path; axial slots in the wall of the tube enable the specimen to be viewed by the pyrometer and the framing camera. In order to approximate the desired condition of zero axial stress during melting, the specimen is mounted under a small tension at room temperature. After the specimen is mounted, the chamber is evacuated and then filled with argon gas (at ~ 0.2 MPa) to minimize problems associated with specimen oxidation and evaporation at temperatures near and above the melting point. A total of 10 identical experiment chambers was constructed to enable the performance of a series of melting experiments per KC-135 flight.

For the flight experiments, the measurement system is bolted to the aircraft floor since its size and mass preclude the possibility of operating in a "free float" mode. The operation of the measurement system in flight is relatively straightforward, requiring essentially three steps per melting experiment: (1) mounting an experiment chamber in the pulse-heating system and then, by means of a hand-held remote control box, (2) resetting the timers and recording instruments and (3) initiating the pulse heating sequence.

3. MEASUREMENTS AND RESULTS

Niobium was selected as the initial specimen material since a number of its high-temperature thermophysical properties in the solid phase had been studied earlier [3]. The specimens were fabricated from 99.95% pure niobium into the form of cylindrical rods, with the following nominal dimensions: 3 mm in diameter and 25 mm length between the electrodes.

In order to obtain some baseline data, a series of rapid melting experiments was performed first in our laboratory. A typical experiment involved heating the specimen from room temperature to its melting point in about 1 to 3 s by passing a large electrical current pulse through it, while simultaneously measuring the radiance temperature of the specimen surface by means of the high-speed pyrometer, and photographing the behavior of the specimen by means of the framing camera operating at 500 fps. A sequence of photographs of the specimen is shown in Fig. 3, where the time

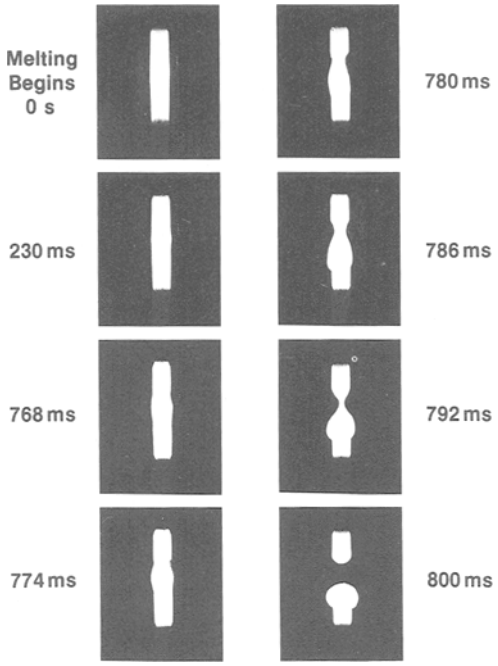

Fig. 3. Sequence of photographs of a rod-shaped niobium specimen taken during a rapid melting experiment on ground.

indicated beside each photograph is measured from the start of melting. The effect of gravity is clearly evident in the photographic sequence, which shows the "effective" portion of the specimen sagging rapidly downward and collapsing prior to the completion of melting.

Prior to the flight experiments, the niobium specimens were modified by "necking-down" the center portion of each specimen rod in order to better define the melting zone. The nominal dimensions of the melting zone, that is, the "effective" specimen, were a diameter of 2.5 mm with an aspect ratio (length: diameter) in the range of 1:1 to 3:1. Portions of each specimen rod near the electrode/clamps were tapered to a diameter of about 2 mm in order to reduce axial heat conduction from the "effective" specimen.

Microgravity simulation experiments were performed on the niobium specimens during a series of parabolic maneuvers by the KC-135 aircraft. A profile of a typical maneuver is illustrated by Fig. 4, which presents an oscilloscope trace photograph of the output of the z-axis accelerometer

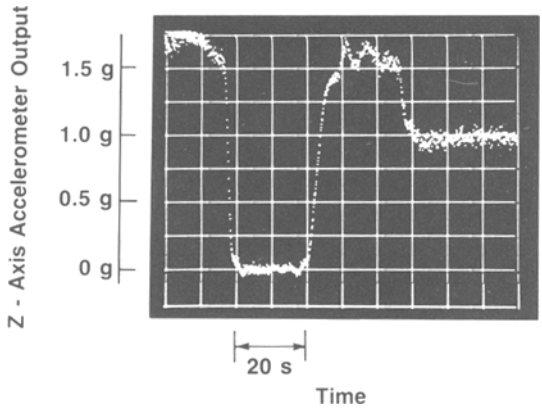

Fig. 4. Variation of the output of the z-axis accelerometer mounted at the experiment chamber base during a typical parabolic maneuver by the KC-135 aircraft.

mounted at the experiment chamber base. As may be seen, the g level is initially about 1.8 g during the ascent of the aircraft to maximum altitude, falls to near 0 g for a period of about 20 s as the aircraft passes through the apex of the parabolic trajectory, increases rapidly to about 1.7 g during the descent, and finally returns to 1 g as the aircraft resumes level flight. The average minimum g level during the melting period of a given experiment was typically about $1 \times 10^{-2}\, g$, with a standard deviation of approximately $6 \times 10^{-2}\, g$; deviations from the average g level were due to mechanical vibrations transmitted to the specimen mount from the aircraft and from the framing camera.

In these experiments, the duration of the electrical current pulse used to heat the specimen from ambient temperature through its melting point varied from about 1.5 to 4.5 s, depending on the experiment. The magnitude of the current pulse varied typically between 450 and 650 A, yielding heating rates in the range 700 to 1700 $\mathrm{K \cdot s^{-1}}$.

A sequence of photographs of the specimen taken during one of the melting experiments is presented in Fig. 5; the time indicated beside each photograph is measured from the onset of melting. As may be seen, the effective specimen retains its cylindrical geometry through the entire melting period of 320 ms, and even into the liquid phase, until at about 530 ms it begins to oscillate; the amplitude of oscillation increases until at 650 ms the specimen collapses.

Figure 6 compares the variation of radiance of the specimen surface during a microgravity experiment with that observed during one of the earlier laboratory experiments. On ground, the radiance rises smoothly to

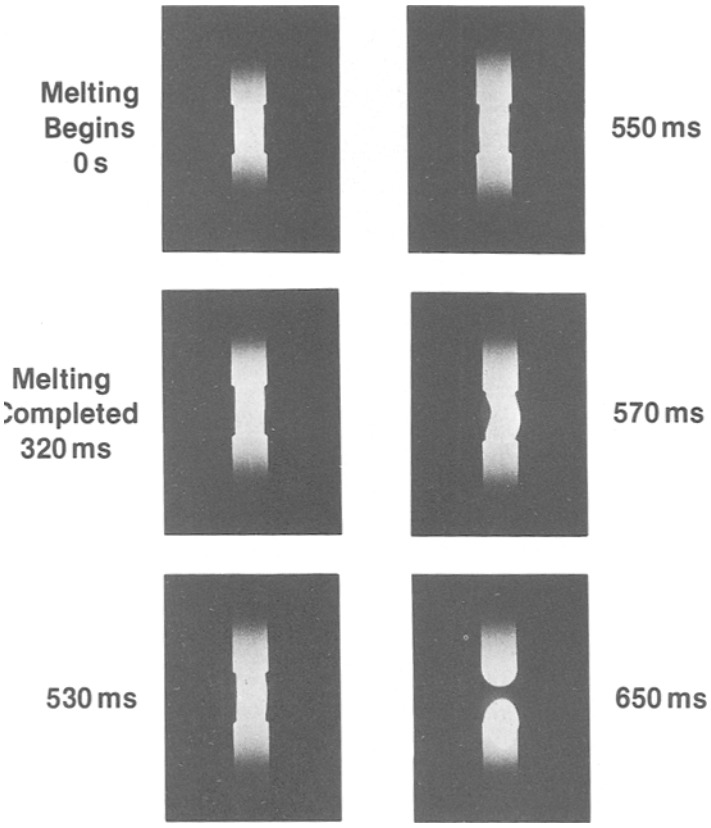

Fig. 5. Sequence of photographs of a rod-shaped niobium specimen taken during a rapid melting experiment in a microgravity environment.

a melting plateau, which ends abruptly when the specimen collapses before melting has been completed. In microgravity, the specimen radiance rises to a plateau and then continues to rise into the liquid phase for an excursion of about 50 K before the specimen collapses. (The sudden decrease in surface radiance at the onset of melting is due to surface roughness which resulted when specimens were "necked down.") The largest temperature excursions ($\sim$50 K) into the liquid phase were observed when effective specimens had a length:diameter ratio of 1.5:1. The oscillations in the radiance versus time function associated with the liquid phase have about the same frequency as those observed in the liquid column (see Fig. 5).

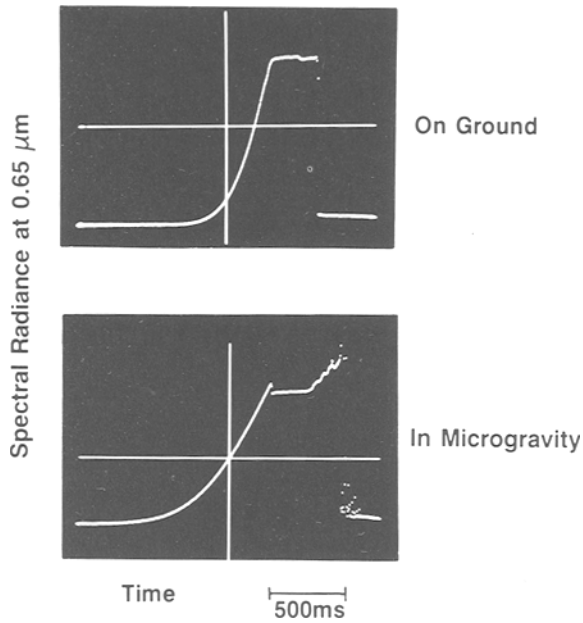

Fig. 6. Variation of the measured spectral radiance at 0.65 μm from niobium specimens during rapid melting experiments on ground and in a microgravity environment.

4. CONCLUDING REMARKS

The results described herein show that specimens in the form of cylindrical rods can be rapidly heated into their liquid phase when the experiments are performed under microgravity conditions. The excursion into the liquid phase was limited, however, and was characterized by the appearance of oscillations in the liquid column just before the collapse of the specimen. The oscillations are believed to arise from mechanical vibrations transmitted to the specimen from sources such as the aircraft and the framing camera. The vibrations probably initiated the specimen collapse because the present specimen geometry, namely, that of a current-carrying cylindrical liquid column, has been shown to be unstable with respect to perturbations of its surface [7].

It is expected that the temperature excursion into the liquid phase can be increased by improving the vibration isolation of the specimen. Further work is under way to improve the stability of rod-shaped specimens and also to study the stability of other geometries such as tubular specimens mounted in a "triaxial configuration" [8].

Significant excursions into the liquid phase will enable the determination of thermophysical properties, such as heat of fusion, and heat capacity and electrical resistivity of the liquid specimen.

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