DYNAMIC MEASUREMENTS OF THERMOPHYSICAL PROPERTIES OF METALS AND ALLOYS AT HIGH TEMPERATURES BY SUBSECOND PULSE HEATING TECHNIQUES

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Abstract

Rapid (subsecond) heating techniques developed at the National Institute of Standards and Technology for the measurements of selected thermophysical and related properties of metals and alloys at high temperatures (above 1000 °C) are described. The techniques are based on rapid resistive self-heating of the specimen from room temperature to the desired high temperature in short times and measuring the relevant experimental quantities, such as electrical current through the specimen, voltage across the specimen, specimen temperature, length, etc. with appropriate time resolution.

The first technique, referred to as the millisecond-resolution technique, is for measurements on solid metals and alloys in the temperature range 1000 °C to the melting temperature of the specimen. It utilizes a heavy battery bank for the energy source, and the total heating time of the specimen is typically in the range 100-1000 ms. Data are recorded digitally every 0.5 ms with a full-scale resolution of about one part in 8000. The properties that can be measured with this system are: specific heat, enthalpy, thermal expansion, electrical resistivity, normal spectral emissivity, hemispherical total emissivity, temperature and energy of solid-solid phase transformations, and melting temperature (solidus).

The second technique, referred to as the microsecond-resolution technique, is for measurements on liquid metals and alloys in the temperature range 1200 to 6000 °C. It utilizes a capacitor bank for the energy source, and the total heating time of the specimen is
typically in the range 50-500 μs. Data are recorded digitally every 0.5 μs with a full-scale resolution of about one part in 4000. The properties that can be measured with this system are: melting temperature (solidus and liquidus), heat of fusion, specific heat, enthalpy, and electrical resistivity.

The third technique is for measurements of the surface tension of liquid metals and alloys at their melting temperature. It utilizes a modified millisecond-resolution heating system designed for use in a microgravity environment.

Presently, effort is underway in the following two directions: (1) to lower the operating temperature range of the high-speed pyrometers in order to extend the property measurements to lower temperatures and (2) to develop methods for the measurements, by pulse heating techniques, of other relevant properties, such as thermal conductivity, speed of sound, elastic constants, etc.

DYNAMIC MEASUREMENTS OF THERMOPHYSICAL PROPERTIES AT HIGH TEMPERATURES

- Dynamic Techniques (What?, Why?, How?)
- Properties
- Materials
- Examples
  - Millisecond-Resolution Technique
  - Microsecond-Resolution Technique
- Future Directions
WHAT ARE DYNAMIC TECHNIQUES?

Techniques for the measurement of
selected thermal and related properties
at high temperatures (above 1000 °C) in
very short times (less than one second)

WHY ARE DYNAMIC TECHNIQUES NEEDED?

• To perform measurements where steady-state methods fail
• To simulate actual dynamic conditions
• To perform measurements efficiently
• To study nonequilibrium and nonlinear phenomena

HOW ARE DYNAMIC TECHNIQUES PERFORMED?

Rapid resistive self-heating of the
specimen by the passage of an electrical
current pulse through it and measuring
the experimental quantities (I,V,T,...)
with appropriate time resolution
MATERIALS

- Metals
- Alloys
- Graphite
- Composites
- Conducting Oxides, Carbides, Nitrides, Borides

PROPERTIES

Solid Phase (1000°C to Melting Point)
- Specific heat, Enthalpy
- Thermal expansion
- Thermal diffusivity
- Thermal conductivity (*)
- Electrical resistivity
- Emissivity - normal spectral
- Emissivity - hemispherical total
- Elastic constants (*)
- Temp. and Energy of solid-solid transformation
- Melting temperature - solidus, liquidus

Liquid Phase (1200°C to 6000°C)
- Heat of fusion
- Specific heat, Enthalpy
- Electrical resistivity
- Surface tension
- Thermal expansion (*)

(*) Under development
MILLISECOND-RESOLUTION SYSTEM

- Specimen: Solid Conductor
- Temperature Range: 1000-4000 °C
- Experiment Duration: 100-1000 ms
- Data Acquisition: Every 0.5 ms

Functional diagram of the millisecond system.
Schematic diagram of the experiment chamber of the millisecond system.
Melting Points, K

<table>
<thead>
<tr>
<th>Element</th>
<th>Melting Point, K</th>
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<tbody>
<tr>
<td>Ni</td>
<td>1729±4</td>
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<tr>
<td>Pd</td>
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<td>Ti</td>
<td>1945±5</td>
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<td>V</td>
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<td>Nb</td>
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<tr>
<td>Mo</td>
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<td>W</td>
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</table>

Heat capacity of selected materials at high temperatures.

Electrical resistivity of selected materials at high temperatures.
Variation of the radiance temperature as a function of time of graphite at its triple point.

MICROSECOND-RESOLUTION SYSTEM

- Specimen: Solid And/Or Liquid Conductor
- Temperature Range: 1200-6000 °C
- Experiment Duration: 50-500 μs
- Data Acquisition: Every 0.5 μs
Functional diagram of the microsecond system.
Schematic diagram of the experiment chamber of the microsecond system.
Variation of power and radiance temperature of a niobium specimen as a function of time in solid and liquid phases.

Absorbed energy as a function of temperature for solid and liquid niobium.
Electrical resistivity as a function of temperature for solid and liquid niobium.

Heat capacity as a function of temperature for solid and liquid niobium.
THERMOPHYSICAL MEASUREMENTS IN MICROGRAVITY ENVIRONMENT

Functional diagram of the measurement system for microgravity experiments with NASA's KC-135 aircraft.
Schematic diagram of an experiment chamber which utilizes a triaxial configuration for studying the stability of tubular specimens during rapid melting in a microgravity environment.
FUTURE DIRECTIONS

- Extension of measurements to lower temperatures (down to 600 °C)
- Extension of measurements to higher temperatures and pressures
- Development of new techniques for dynamic measurements of other properties
- Development of new techniques for dynamic measurement of nonconductors
- Investigations of nonequilibrium phenomena