Measurement of thermophysical properties by a pulse-heating technique

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A technique is described for the dynamic measurement of selected thermophysical properties of electrically conducting solids in the temperature range from 1100 K to the melting point. Based on rapid resistive self-heating of the specimen from room temperature to any desired high temperature in several seconds by the passage of an electical current pulse through it, this technique measures the pertinent quantities such as current, voltage, randiance temperature, with sub-millisecond time resolution. The pulse-heating technique is applied to strip specimens. The radiance temperature is measured by high-speed pyrometry, normal spectral emissivity of the strips is measured by integrating sphere reflectometry. The normal spectral emissivity results are used to compute the true temperature of the specimens. The heat capacity, electrical resistivity, total hemispherical emissivity are evaluated in the temperature range from 1100 K to the melting point.

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Coventional steady-state and quasi-steady-state techniques for accurate measurement of thermophysical properties are generally limited to temperatures below 2000 K. This limitation is the result of severe problems (chemical reactions, heat transfer, evaporation, specimen containment, loss of mechanical strength and electrical insulation, etc.) created by the exposure of specimen and the high-temperature environment[1]. The pulse-heating technique is considered as the most accurate method for the measurement of thermophysical properties at very high temperatures, because it overcomes several limitations of a steady-state experiment. Practical applications often require measurements to be performed on simple specimens[2]. The development of millisecond-resolution dynamic technique at the Harbin Institute of Technology (HIT, China) has enabled the accurate measurement of selected thermophysical properties of electrically conducting refractory solids at high temperature, primarily in the range from 1100 K to the melting point. The measured properties include heat capacity, electrical resistivity, total hemispherical emissivity, normal spectral emissivity, and melting temperature.

The method is based on resistive self-heating of the specimen from room temperature to any high temperature (in the range from 1100 K to its melting temperature) in several seconds by the passage of an electical current pulse through it. Experimental quantities, such as current through the specimen, voltage across the specimen, and specimen temperature, are measured with millisecond resolution.

The detail of this method has been given in earlier publication[3]. Data on the specimen current (I), the voltage drop across the measurement zone (U), the electromotive force (EMF) of the pyrometer, and the integrating sphere reflectometer at the center of effective specimen measurement zone (E) were collected during heating and the initial part of cooling period. The normal spectral emissivity $\varepsilon_\lambda(T)$ was measured by an integrating sphere reflectometer. At the same time, the spectral temperature T_λ at the same wavelength was measured by a high-speed pyrometer. A simpler and in many cases very useful expression can be derived using the Wien approximation:

$$T = \frac{c_2 T_\lambda}{\lambda T_\lambda \ln \varepsilon_\lambda(T) + c_2}. \tag{1}$$

So the normal spectral emissivity data $\varepsilon_\lambda(T)$ measured simultaneously was used to obtain the true temperature T from the spectral temperature T_λ measured in the experiment. The heating rates (up to 1000 K/s), the specimen geometry, and the high vacuum make radiative heat loss be the only significant energy loss from the effective specimen volume.

The power balance for the effective part of the specimen during the heating period is

$$UI = mC_P \left(\frac{\mathrm{d}T}{\mathrm{d}t}\right)_{\mathrm{h}} + P_{\mathrm{r}}, \tag{2}$$

where m is the mass of the effective specimen volume, C_p is the specific heat, T is the true temperature of the specimen, and t is the time. The radiated power P_{r} is given as

$$P_{\mathrm{r}} = \varepsilon\sigma A(T^4 - T_{\mathrm{a}}^4), \tag{3}$$

where T_{a} is the ambient temperature, ε is the total hemispherical emissivity, A is the effective surface area, and σ is the Stefan-Boltzmann constant. During the initial part of the cooling period, the power balance is given by

$$-mC_P \left(\frac{\mathrm{d}T}{\mathrm{d}t}\right)_{\mathrm{c}} = P_{\mathrm{r}}. \tag{4}$$

In the equations, the subscripts h and c refer to the heating and cooling periods, respectively. From Eq. (2) the specific heat is obtained as

$$C_p = \frac{UI - P_{\mathrm{r}}}{m(\mathrm{d}T/\mathrm{d}t)_{\mathrm{h}}}. \tag{5}$$

The total hemispherical emissivity at a specific temperature is calculated from Eqs. (2) and (4) as

$$\varepsilon = \frac{UI}{\sigma A(T^4 - T_a^4)[1 - (\mathrm{d}T/\mathrm{d}t)_h/(\mathrm{d}T/\mathrm{d}t)_c]}. \quad (6)$$

The specific electrical resistivity ρ is determined from the electrical resistance of the specimen as

$$\rho = \frac{U}{I}\frac{S}{l_e}, \quad (7)$$

where S is the sample cross-sectional area and l_e is the effective sample length between the potential leads.

The measurement system consists of an experimental chamber, an electric power pulsing circuit, the measuring and control instruments, a computer system, a fast data-acquisition system, and other I/O devices. A functional diagram of the system is presented in Fig. 1.

The pulse power circuit includes the specimen in series with a battery bank (12 V), a standard resistor, and a high speed switching system. The heating rate of the specimen and the shape of the current pulse are controlled by changing the number of batteries. The switching system is computer operated through metallic oxide semiconductor field effect transistor (MOSFET) drivers and is opened and closed in sequence with a predetermined time interval, whereas a standard 2-mΩ resistor is used to measure the pulse current generated by the computer-controlled switching unit.

The experimental chamber contains the specimen, the integrating sphere, the clamping electrodes, an expansion joint, the voltage probes, and other auxiliary components. The specimen is a strip of the following typical dimensions: length, 65−85 mm; width, 6−10 mm; thickness, 0.5−1 mm. Two 0.05- or 0.1-mm thermocouples are connected (electrically insulated) to the two end clamps to measure the specimen temperature before each pulse experiment. Depending on the temperature range, K- or S-type thermocouples are used. The expansion joint allows the expansion of the specimen in the downward direction. The voltage probes are knife edges made of the specimen material and are placed at a distance approximately 10 mm from the end clamps. The knife edges

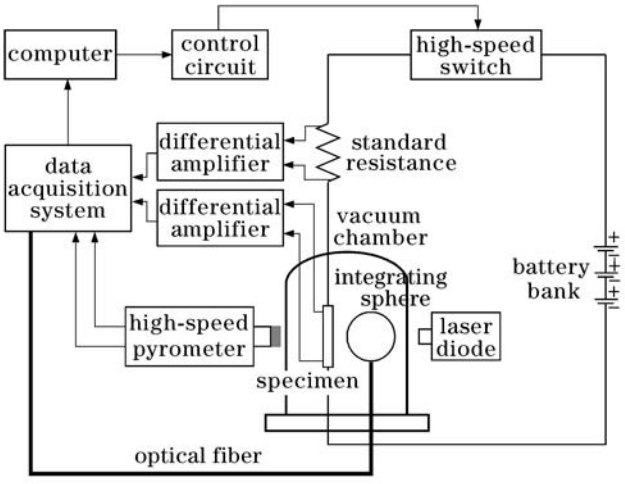

Fig. 1. Functional diagram of the dynamic thermophysical measurement system.

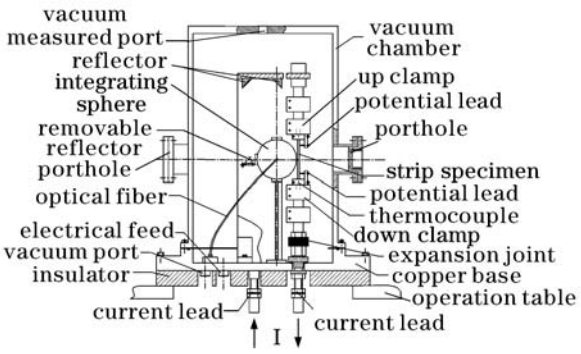

Fig. 2. Schematic diagram of the experiment chamber.

define a portion of the specimen, which should be free of axial temperature gradients for duration of the experiment. The chamber is designed in a vacuum of 10^{-3} Pa in order to prevent oxidation of the specimen. A schematic diagram of the experiment chamber is shown in Fig. 2.

One side of the strip faces the porthole of a high-speed integrating sphere reflectometer of the comparison type in which the reflectivity of the specimen (that undergoes pulse heating) is measured in relation to the known reflectivity of reference specimen barium sulfate ($BaSO_4$). The nominal dimensions of the integrating sphere are 80 mm in diameter and 2 mm in thickness. The inside of the sphere is coated with a very white paint ($BaSO_4$). For safety, the hot strip specimen must be placed at an appropriate distance (2 mm) from the sphere. A modulated beam generated by a laser diode strikes the side of the strip facing the integrating sphere. The reflected beam is collected by the integrating sphere, taken out with an optical fiber, and measured by a silicon detector placed outside the experimental chamber. The detector operates at the wavelength of the laser diode (about 633 nm) that is the same as one of the multi-wavelength pyrometer. A numerical lock-in technique is used to discriminate between the reflected modulated beam and the radiation emitted by the specimen itself at high temperatures[4−6].

The radiance temperature is measured on the other side of the strip at 633 nm by a high-speed pyrometer with microsecond time resolution. Figure 3 is the optical scheme of the pyrometer designed and constructed specifically

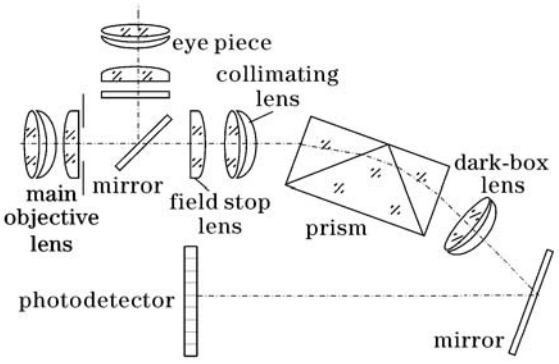

Fig. 3. Optical scheme of the high-speed pyrometer.

for this apparatus on the basis of the multi-wavelength pyrometer that has been constructed in cooperation between HIT and University of Rome "Tor Vergata"[7]. The pyrometer operates at a fixed distance from the target (300 mm). The response time of the pyrometer is less than 5 μs. It covers a temperature range from 1100 to 4000 K using an auto-ranging feature and the spectral range is $0.5-1.1$ μm. Pyrometer auto-ranging is accomplished by inserting three amplifiers connected in series between the pyrometer signal output and the data acquisition system and each amplifier output signal is recorded with the data acquisition system.

The experimental procedure is performed using a computer consisting of data acquisition, control of the current pulse, and subsequent data processing. Electrical signals from the pyrometer as well as those corresponding to the current through and the voltage across the specimen are recorded as functions of time. This is accomplished by use of a 16-bit data acquisition system capable of one measurement every 10 μs. It consists of a sample and hold array and a high-speed analog-to-digital (A/D) converter. The data are collected during the heating and the initial cooling periods at 1-kHz sampling rate. At the end of an experiment, the data stored in the memory are analyzed by precompiled software. Additional technical details on the measurement technique and on the system characteristics are as follows.

- Specimen voltage measurement: across knife-edge probes in the central portion of the specimen.
- Current measurement: across standard resistor (2 mΩ) in series with the specimen.
- Temperature measurement: high-speed pyrometer operating near 633 nm.
- Lock-in measurement: single phase lock-in amplifier; modulation frequency ~ 800 Hz.
- Specimen environment: high vacuum $\sim 10^{-3}$ Pa.
- Power source: battery bank; up to ten series-connected 1.2-V elements with a capacity of ~ 1700 Ah each.
- Data recording: high-speed data acquisition system (16 bit, 100 kHz, range $0-10$ V, least significant bit 0.15 mV).
- Temperature range: $1100-4000$ K with pyrometer autorange.

The dynamic technique described in this paper has been used successfully for the measurements of several thermophysical and related properties in the range from 1100 K to the melting temperature of the specimen. The measurements are performed with a pulse-heating technique, a pyrometry, and a high-speed integrating sphere reflectometer. The design is based on systems previously developed at Istituto Nazionale Ricerca Metrologica (INRM) for the measurement of several thermophysical properties with millisecond time resolution. In the present stage, the experimental apparatus has been constructed and measurements are performed on various materials such as iron, copper, niobium, tungsten, etc.. The experimental results will be published later. The technique has been perfected to a level of competitiveness with the most accurate conventional steady-state techniques in the overlapping temperature range ($1500-2000$ K) for two types of techniques[1].

At temperatures above 2000 K, it becomes very difficult or even impossible to perform accurate steady-state experiments; thus the dynamic technique becomes a unique and indispensable tool for thermophysical measurements. The dynamic technique has the advantages of covering a wide temperature range in a single experiment and yielding high resolution in temperature measurements in comparison with most of the conventional techniques. These are important features, especially in measurements near and at phase transformations.

The potential of the dynamic technique has not been explored completely. In addition to the properties investigated so far, the dynamic technique may be used to measure other thermal, electrical, and related properties in both the solid and liquid phases, even to measure properties of more complex substances such as certain carbides, borides, nitrides, and oxides. Furthermore, the dynamic technique may provide the means of studying time-dependent phenomena in substances.

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