

A New Dynamic Technique for the Measurement of Hemispherical Total Emittance¹

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A new dynamic technique for the measurement of thermal conductivity under development at the IMG C requires accurate values of heat capacity and of hemispherical total emittance at high temperature. Until recently, these data were provided by subsecond pulse heating experiments performed on the same specimens in the same apparatus. The pulse heating technique is the most accurate method for the determination of heat capacity at high temperatures, but because of various experimental problems, the accuracy of hemispherical total emittance determinations is limited to 5%. A new method for a more accurate determination of hemispherical total emittance is proposed, which uses the same experimental data available from thermal conductivity experiments. An analysis of the temperature profiles measured during the free cooling indicates that regions with high-temperature gradients (toward the ends of the specimen) are the best regions for thermal conductivity measurements, while regions with low-temperature gradients (at the center of the specimen) are the best regions for hemispherical total emittance determinations. The new measurement method and some preliminary results are presented and discussed.

KEY WORDS: dynamic measurements; emittance; high temperature; scanning pyrometry.

1. INTRODUCTION

New dynamic measurement methods, based on applications of high-speed scanning pyrometry [1], are currently in development at the Istituto di Metrologia "G. Colonnetti" (IMG C). The new dynamic technique for the measurement of thermal conductivity [2] is based on the accurate measurement of temperature profiles during free cooling, after the specimen has been brought to high temperatures by a subsecond-duration current

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pulse. During cooling, the principle of energy conservation in each small volume of the specimen is expressed by a differential equation: the energy released by the small volume is either radiated or conducted away. The differential equation contains three terms related to the heat capacity, the hemispherical total emittance, and the thermal conductivity of the material. The new technique for thermal conductivity is based on accurate measurements of the temperature profiles during the cooling period: with experimental measurements of temperature and of temperature derivatives with respect to space and time, the differential equation may be transformed into a linear equation of the unknown thermal conductivity. An overdetermined set of linear equations obtained from the profiles at different temperatures may be solved by the least-squares method [2]. The solution of these equations to obtain the thermal conductivity function requires accurate values of the heat capacity and of the hemispherical total emittance of the material at high temperatures. In experiments performed so far, these data were provided by subsecond pulse heating experiments performed on the same specimens in the same apparatus.

The subsecond pulse heating technique is the most accurate method for the determination of the heat capacity at high temperatures [3]. In this technique a combination of measurements performed during heating and during the initial part of the cooling period provides the radiation loss and hence the hemispherical total emittance. Radiation losses in typical pulse heating experiments range from less than 1% of the input power to the specimen at temperatures below 1500 K, increasing to about 10–15% at temperatures above 2500 K. The free cooling of the specimen may be followed only for a short time (less than 100 ms) because afterward the temperature profile in the specimen becomes distorted due to axial heat conduction, and measurements performed inside the blackbody hole no longer refer to a portion of specimen at uniform high temperature (as it was during heating). When this happens, wrong cooling rates are measured. The hemispherical total emittance results in subsecond pulse heating experiments are also extremely dependent on the accurate machining of the specimen. Small machining imperfections (often not detectable with conventional tools) and the resulting small differences in cross-sectional area may lead to small temperature gradients in the specimen during rapid heating. When the current stops, these localized temperature gradients tend to diminish rapidly and often change significantly the initial cooling rate of the specimen. Because of these problems, the final accuracy of the hemispherical total emittance determinations in pulse heating experiments is limited to about 5%. These limitations are well known and are inherent in the technique: a pulse heating experiment to measure heat capacity is generally designed to minimize the radiation losses, and conse-

quently the accuracy of hemispherical total emittance determinations suffers.

In the present paper, a new method for a more accurate determination of hemispherical total emittance is proposed, making use of the same dynamic technique and of the same experimental arrangement both for thermal conductivity and for hemispherical total emittance measurements. A schematic diagram of the specimen and of the optical instrumentation is presented in Fig. 1.

2. MEASUREMENT METHOD

The new dynamic technique for hemispherical total emittance consists in performing a pulse heating experiment (lasting less than 1 s) and in measuring accurately the temperature profiles in the central region of the specimen during the free cooling period. The experiment does not differ from that used for thermal conductivity determinations [2], and different regions of the same temperature profiles may be used for thermal conductivity determinations and for hemispherical total emittance measurements.

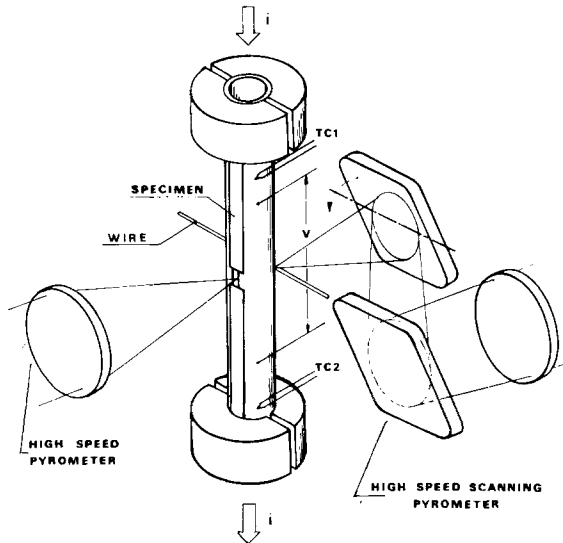


Fig. 1. Schematic representation (from Ref. 2) of the new dynamic technique used either for hemispherical total emittance or for thermal conductivity. i , current passing through the specimen; V , voltage drop across the central part of the specimen; TC1 and TC2, Chromel-Alumel thermocouples.

The concept is illustrated in Fig. 2. The flat temperature profiles in the central part of the specimen (zone "a") indicate a small contribution of the thermal conduction in the axial direction, and consequently a large percentage of the energy lost by this portion of the specimen is through radiation losses. Zone "a" is therefore the best region for an experimental determination of the hemispherical total emittance. Conversely, zone "b" in Fig. 2 shows high-temperature gradients, and in this part a large fraction of the energy released by the specimen during cooling is lost by thermal conduction. Zone b is therefore the optimum region for the determination of thermal conductivity [2].

Assuming the "long thin rod approximation" [4] (no thermal conduction in the radial direction), the differential equation applicable to each point of a cooling temperature profile is

$$\frac{\partial}{\partial x} \left(\lambda \frac{\partial T}{\partial x} \right) - \frac{\varepsilon_{ht} \sigma p (T^4 - T_a^4)}{S} = \delta c_p \frac{dT}{dt} \quad (1)$$

Equation (1) contains different thermal properties (λ , thermal conductivity; ε_{ht} , hemispherical total emittance; δ ; density; c_p , heat capacity), geometrical quantities (p , perimeter; S , cross-sectional area), temperature T with its

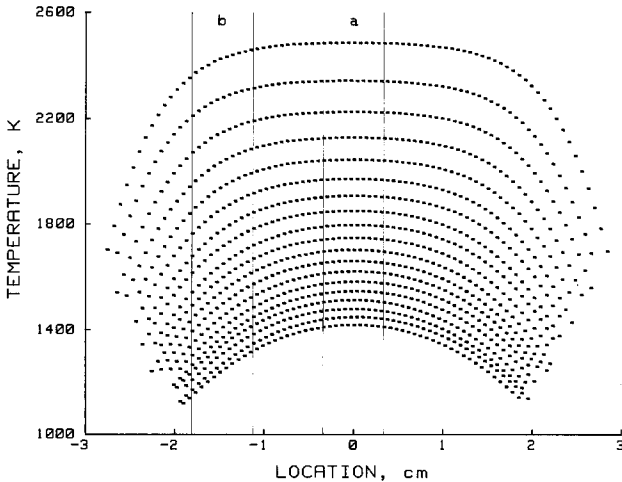


Fig. 2. Experimental temperature profiles and typical regions used for computations. For viewing purposes, a reduced data set is presented: one profile of every three is plotted, and on each profile shown, only one temperature of every two is plotted. Different regions are ideal for hemispherical total emittance determinations (zone a) and for thermal conductivity determinations (zone b).

space and time derivatives, ambient temperature T_a , and the Stephan Boltzmann constant σ . Both thermal properties and geometrical quantities are functions of temperature. For geometrical quantities thermal expansion effects must be considered; for thermal properties the temperature dependence of the property and possible thermal expansion effects must be taken into account.

An advantage of this new dynamic technique is the possibility to determine the hemispherical total emittance over a wide temperature range in a single experiment. In principle, one may follow the power balance of the central part of the specimen from very high temperatures down to the lowest temperature measurable by pyrometry (with present experimental conditions from approximately 3000 to 1100 K). A major problem in an experiment covering a wide temperature range is due to the fact that a pyrometer focused on one point in real space will "see" different points of the specimen during the experiment, on account of the thermal expansion of the specimen. Additional problems arise with the method of measurement of properties needed in Eq. (1) that may or may not be corrected for thermal expansion effects, as pointed out in Ref. 5. As previously done in the thermal conductivity technique [2], these problems are solved by using Eq. (1) in a hypothetical space where no thermal expansion takes place. During computations, all quantities are referred to an ideal tube-space (the one existing when the specimen is at room temperature). Temperature profiles measured in real space are made to shrink to the shape they would have had if the specimen did not expand and space derivatives are computed in this tube-space. The methods of measurement of the needed thermal properties are analyzed and adequate thermal expansion corrections are applied to work in tube-space. Once this is done, the temperature versus time derivative is obtained by fitting the evolving temperature of one point in tube-space. The final hemispherical total emittance results need again to be partially corrected to take fully into account thermal expansion effects.

After some simple mathematical operations, Eq. (1) may be rewritten as

$$\frac{\epsilon_{ht} \sigma p (T^4 - T_a^4)}{S} = \lambda \frac{\partial^2 T}{\partial x^2} + \frac{d\lambda}{dT} \left(\frac{\partial T}{\partial x} \right)^2 - \delta c_p \frac{dT}{dt} \quad (2)$$

and in any point of any profile the hemispherical total emittance may be computed assuming that the heat capacity and thermal conductivity of the material as functions of temperature are known. In Eq. (2) temperature and its derivatives are quantities obtained experimentally. This equation is somewhat misleading in its appearance: in a cooling experiment the heat

capacity term is positive (because dT/dt is negative) and represents the largest contribution. The other two terms on the right-hand side of Eq. (2) represent the thermal conduction contribution: the first one is always the dominant term and is negative (because $\partial^2 T/\partial x^2$ is negative). The second term is very small (generally less than 1% of the other conductivity term) and is either positive or negative depending on the sign of $d\lambda/dT$.

3. EXPERIMENTAL APPARATUS AND PRELIMINARY RESULTS

Measurements were performed with the apparatus developed for pulse experiments [6] with subsequent modifications for thermal expansion [7] and thermal conductivity measurements [2]. Detailed descriptions of the various parts of the apparatus including the experimental chamber, measurement electronics, and data acquisition systems may be found in earlier publications [2, 6, 8].

The temperature profiles during cooling are measured, with microsecond resolution, with a scanning pyrometer specifically developed for these dynamic techniques. The instrument consists of a microsecond pyrometer coupled with a rotating mirror (Fig. 1) and associated electronics for triggering and synchronization. The pyrometer is a monochromatic instrument with a silicon detector operating at 900 nm (80-nm bandwidth). Technical descriptions and performance data on this pyrometer are given in Ref. 9. The scanning pyrometer is connected to a fast data acquisition system triggered by the computer running the experiment. Software programming provides automatic pyrometer autoranging during both heating and cooling. During the experiment, cooling rates typically change from $500 \text{ K} \cdot \text{s}^{-1}$ at the highest temperatures to $50 \text{ K} \cdot \text{s}^{-1}$ at 1100 K. The frequency of the rotating mirror is 40 Hz (one temperature profile every 25 ms). In relation to the cooling rate, one profile per rotation is collected at high temperatures, gradually slowing the collection rate to one profile every 10–15 rotations at low temperatures.

Another high-speed pyrometer (silicon detector, operating near 900 nm, 80-nm bandwidth) is focused in the blackbody hole of the specimen (Fig. 1). This is an upgraded version of an instrument developed at the IMGC [10] with a small target area (0.3-mm diameter). Two pyrometers (fixed and scanning) are necessary for “apparent emittance” measurements during heating. The technique of performing “apparent normal spectral emittance” measurements during heating and using the results during cooling was developed for the dynamic thermal conductivity experiment [2]. With this technique, the experimental temperature profiles have direct ties to a blackbody temperature scale and the measurements take into account all the environmental conditions (coatings, transmission

changes, surface conditions, etc.) in the optics and in the specimen right at the time of the experiment.

Preliminary experiments to verify the applicability of this new technique were performed on some tubular niobium specimens previously used for the determination of several thermophysical properties [11, 12]. Their nominal dimensions were as follows: length, 76 mm; effective length (central portion), 25 mm; outside diameter, 6.3 mm; and wall thickness, 0.5 mm. Since hemispherical total emittance is a property strongly dependent on the surface conditions of the specimen, the best evaluation is obtained by comparing the results obtained by the subsecond pulse technique and by the new dynamic technique on the same specimen (Figs. 3 and 4). Figure 3a presents the results of several pulse experiments at different high temperatures. The plotted curve is a least-squares fit of the data extrapolated to lower temperatures (dashed line). The fit is defined by 117 data points from 13 experiments (relative standard deviation 0.5%). The data in Fig. 3a refer to a well-machined specimen without imperfections, because at very high temperatures the data points of a single experiment follow closely the overall emittance curve. Figure 3b presents the hemispherical total emittance results of one experiment with the new dynamic technique (computations performed for one location in the center of the specimen). The results were computed using the heat capacity obtained in subsecond pulse experiments (those in Fig. 3a) and the thermal conductivity as recommended by the TPRC compilation [13],

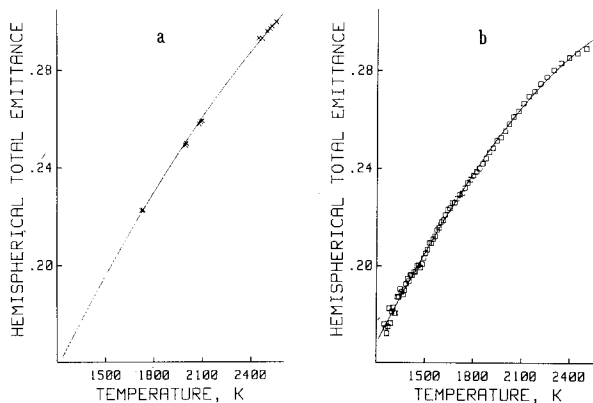


Fig. 3. Results of hemispherical total emittance measurements on the same specimen using subsecond pulse experiments (a) and the new dynamic technique (b). For viewing purposes, a reduced data set is presented in a.

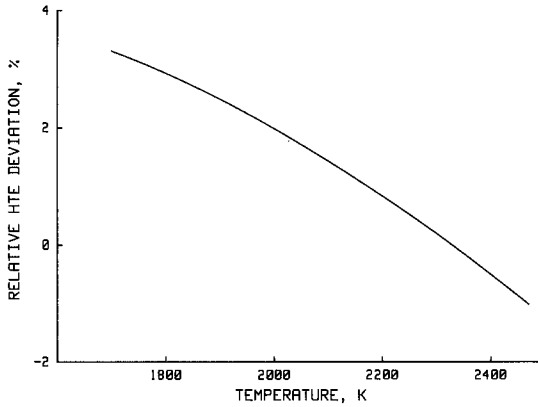


Fig. 4. Relative deviation of the hemispherical total emittance results on the same specimen. The deviation of the least-squares fits shown in Fig. 3 is plotted.

extrapolated above 2200 K. The least-squares fit in Fig. 3b is based on 72 data points (relative standard deviation 0.8%). The computation could be repeated for different locations in the central portion of the specimen: maximum differences of these computations ranged from $\pm 0.4\%$ at high temperatures to about $\pm 1.2\%$ at low temperatures. The increase in the low-temperature region is due to the more pronounced curvature of the profiles and the consequent increase of the conduction term that depends on the computed spatial derivatives.

Figure 4 compares the least-squares fits shown in Fig. 3 in the overlap-

Table I. Typical Values of Radiation Losses (as a Percentage of Input Power) in Subsecond Pulse Heating Experiments and in Experiments with the New Dynamic Technique

Temperature (K)	Radiation losses (% of input power)	
	Subsecond pulse heating experiment	New dynamic technique
2600	16.5	99.0
2400	11.5	97.9
2200	7.8	96.2
2000	5.2	92.6
1800	3.2	84.7
1600		71.0
1400		52.5
1200		30.0

ping temperature range. The hemispherical total emittance results by the new technique depend on the chosen thermal conductivity curve, with an increasing influence at low temperatures (details in Table I). A complete evaluation of the new method is therefore impossible at this stage on account of large uncertainties in the thermal conductivity of niobium at the high temperatures. Recent experimental results [14] show marked differences from the TPRC compilation. Therefore, the intercomparison in Fig. 4 is purely indicative and is dependent on the chosen thermal conductivity function.

4. DISCUSSION

An evaluation of the new dynamic technique for hemispherical total emittance may be done by assessing advantages and disadvantages with respect to the subsecond pulse heating experiment. The two methods have several common points and the following major differences.

- The pulse method is based on the assumption that temperature profiles during the last part of heating and the initial part of cooling are identical in shape and that the central portion of the specimen has no thermal conduction losses. The new technique uses any point on the central part of the specimen and takes into account thermal conduction losses.
- The pulse method is based on measurements of the heating and cooling rate and on the influence of the temperature distribution of the central part of the specimen on the backbody hole. The new technique is based on measurements of the cooling rate and of the second derivative of temperature with space and on the availability of accurate values for the heat capacity and the thermal conductivity of the material.
- The blackbody hole measurements (pulse method) will integrate and average (in a somewhat unknown way) the temperature differences in the central portion of the specimen. The new technique is more susceptible to problems with the single point chosen for measurements, but temperature profiles are known and computations may be performed avoiding troublesome points (exhibiting small temperature gradients or normal spectral emittance variations with respect to the rest of the specimen).

The new dynamic technique is more indirect than the subsecond pulse method because it requires a knowledge of other thermophysical properties (heat capacity and thermal conductivity). On the other hand, it is not

based on a thermal model of difficult verification, but it takes into account all the physical processes occurring in a small volume element.

In both methods, the hemispherical total emittance is obtained from a measurement of the radiation losses. Radiation losses are always a part of input power, but the measurement situation is much more favorable in the new technique. Table I compares the radiation losses as a percentage of input power in a slow pulse heating experiment with that of a cooling experiment with this new technique. In fast pulse heating experiments the situation is worse, because the experiment is designed to minimize radiation losses (to improve the accuracy of heat capacity measurements).

Measurements below 2000 K are extremely difficult (or sometimes impossible) with subsecond pulse heating experiments because the radiation loss is very small and it is obtained as the difference between two large numbers. At these temperatures, the accurate measurement of the cooling rate is difficult because the free cooling is slow and must be followed for some time for an adequate temperature versus time fit. Unfortunately, long times are associated with changing temperature profiles that are reflected (with unknown mechanisms) in the measured blackbody temperature. The problem is much more less severe with the new dynamic technique (see Table I), where at 2000 K the radiation losses are about 90% of the input power.

Two experimental aspects of the new measurement technique need some further explanation. The experimental setup requires a wire placed across the specimen to be used for spatial measurements (Fig. 1). In each profile the wire location is detected as a sharp valley on the pyrometer voltage output. The wire (fixed in real space) establishes a reference point used for spatial computations. Clearly in hemispherical total emittance measurements the wire must be moved away from the center of the specimen to avoid interference with the measurement region.

The best location for hemispherical total emittance measurements is the center of the specimen where thermal conduction is minimum. At present this is also the location of the blackbody hole (opposite side of the specimen, Fig. 1) and the symmetry [implicit in the long thin rod approximation, Eq. (1)] is not perfectly realized in this location. The advantages of a blackbody hole (direct ties of the temperature profiles to a blackbody temperature scale) clearly outnumber any problem due to the lack of symmetry in the measurement region. Different solutions to this problem are possible, including the use of specimens with the blackbody hole machined off center, in a location where it does not interfere with the various experimental determinations.

In conclusion, the new dynamic technique for the measurement of hemispherical total emittance presents the following advantages:

- a great number of data points per experiment,
- measurements over a wide temperature range in each experiment,
- derivation of emittance values from large radiation losses (large percentage of input power),
- direct ties to a blackbody temperature scale, and
- the possibility of determining both thermal conductivity and hemispherical total emittance from one experiment over a wide temperature range.

This paper has presented the new method and some preliminary experimental results. The work on this new measurement technique is continuing to evaluate the accuracy of the method and to consider the possibility of a simultaneous measurement of both thermal conductivity and hemispherical total emittance.

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