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THE WORLD OF SUBSECOND THERMOPHYSICS

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Abstract: The measurement techniques developed in the last thirty-five years for the accurate measurement of thermophysical properties at high temperatures (800-3700 K) using subsecond current-heating are reviewed. Only experiments of total duration around 1 s are considered, with data acquisition performed at the sub-millisecond level. Emphasis is placed on the physics of the different techniques, as developed in major national measurement institutes around the world. A complete survey is provided, covering multiproperty experiments, extensions to thermal expansion and thermal conductivity and measurements at the melting point. An intercomparison of experimental results available in the literature provides an indirect evaluation of the validity of these techniques.

Keywords: dynamic methods, high temperature, thermophysical properties, heat capacity, electrical resistivity.

1. INTRODUCTION

The state of the art in subsecond current pulse-heating methods for the measurement of thermophysical properties is presented. Significant improvements were made possible by these techniques in the last thirty-five years, performing accurate measurements in temperature ranges difficult to reach with traditional measuring methods and characterizing possible high temperature reference materials.

The measurement of thermophysical properties with stationary methods at high temperatures presents considerable difficulties due to the interaction of the material under measurement with the environment, to chemical reactions, to the loss of mechanical strength, etc… Subsecond current pulse-heating techniques have been developed in relation to these experimental problems that increase exponentially above 1500 K. These current pulse-heating experiments are performed in short times, typically in less than one second. The electrically conducting specimen is taken from room temperature to its melting point using a current pulse (Joule heating) and several experimental quantities are measured with millisecond to microsecond time resolution. These techniques have been made possible by developments in electronics (computers and data acquisition systems) and in opto-electronics (semiconductor detectors, high-speed pyrometry) applied to the measurement of thermophysical properties.

The aim of this work is to provide an updated review of the different subsecond measuring techniques used for determining thermophysical properties, giving a complete overview of the capabilities of the pulse techniques for the characterization of electrical conductors at high temperatures. This review is strictly limited to experiments lasting approximately 1 s and performed at a metrological level. No attempt is made to cover faster experiments that may extend measurements to liquid metals (see ref. [1] for details). Some previous reviews in this research area have described the measurement principles [2], the construction of a typical experimental apparatus [3] and the specific application to pulse calorimetry [4]. Pulse-heating techniques have been made possible by continuous developments in high-speed pyrometry: the issues in this research area have been presented in Metrologia [5]. In relation to the review material already available in the literature, the main aim of this paper will be a comprehensive presentation of the different measuring techniques that were developed in some major National Measurement Institutes (NMIs) around the world. The reference to experimental results will be limited to what is necessary for a better understanding of the measurement techniques and for an informal evaluation of their uncertainty.

Table 1 presents a list of the main laboratories of the world that have been active in subsecond current-heating techniques. The entries are arranged by involvement date: laboratories with a long history of work in this research area appear at the top. A significant publication, generally providing a description of the measurement apparatus, is indicated for each laboratory. Most of the pioneering work was done in some NMIs. In recent years, after current-heating techniques proved their potential capabilities and became established methods, universities and research centers are continuing the effort with practical applications.

The largest part of this presentation is centered on the various subsecond measurement techniques using current pulse-heating originally developed at the National Bureau of Standards (NBS, USA, presently the National Institute for Standards and Technology NIST), at the Istituto di Metrologia "G. Colonnetti" (IMGC, Italy, presently the Istituto Nazionale di Ricerca Metrologica INRIM) and at the National Research Laboratory for Metrology (NRLM, Japan, presently the National Measurement Institute of Japan NMIJ). For obvious practical reasons most of the descriptions are related to the experimental apparatus developed in the author's institution and most figures are drawn from earlier publications.

Table 1. Active laboratories in subsecond experiments with data acquisition performed with millisecond time resolution.

Institution	Country	T range (K)	Ref.
National Institute of Standards and Technology (NIST)	USA	650 - 3700	[6]
Istituto Nazionale Ricerca Metrologica (INRIM)	Italy	800 - 3700	[7]
Physikalisch-Technische Bundesanstalt (PTB)	Germany	400 - 1500	[8]
Boris Kidric Institute of Nuclear Sciences (VINÇA)	Yugoslavia	300 - 1900	[9]
Institute for High Energy Densities of Russian Academy of Sciences (IHED)	Russia	1000-3700	[10]
National Metrology Institute of Japan (NMIJ)	Japan	1300 - 3700	[11]
Austrian Foundry Research Institute (ÖGI)	Austria	600 - 3700	[12]
Harbin Institute of Technology (HIT)	China	1000 - 3700	[13]

2. MULTIPROPERTY MEASUREMENTS

This definition applies to a single subsecond experiment in which up to four different thermophysical properties may be measured simultaneously in a wide temperature range: namely heat capacity, electrical resistivity, hemispherical total emissivity and normal spectral emissivity. Enthalpy might also be computed by numerical integration of the heat capacity data.

Four measuring techniques have been developed using different types of specimens. The original technique was developed at the National Bureau of Standards (NBS, USA, presently NIST) and has been extensively used both at the NIST and at the IMGC (presently INRIM). This classical technique provides the lowest measurement uncertainty, but requires tubular specimens machined with high precision. This is not always possible and sometimes materials are not available in tubular form. Two alternative techniques (described later for strip and rod specimens) differ from the classical technique only for the method of temperature measurement. In both cases only radiance temperature is measured during the experiment, but different physical principles are applied to perform at the same time a high-speed measurement of the normal spectral emissivity of the specimen that is being pulse-heated. One of the techniques (developed at the IMGC) involves a simultaneous fast reflectometric measurement and can be applied to simple strip specimens easy to machine to appropriate dimensions. Another technique (developed at the NIST in collaboration with Containerless Research Inc.) uses laser polarimetry to measure the normal spectral emissivity of rod specimens during the pulse-heating experiment. A final variant (developed jointly by the NIST and by the NRLM) combines laser polarimetry with a brief steady-state experiment to improve the determination of radiation losses.

2.1. Classical technique (tubular specimen)

The experiment consists of the rapid heating of a tubular specimen by the passage of a current pulse of subsecond duration (Fig. 1). During this short time the central portion of the specimen is self-heated to high temperatures with minimal loss of energy by thermal conduction toward the clamps. In this way a specimen at high temperature (central zone between the knife-edge probes, see Fig. 1) is obtained. The experiment is completely under computer control: on the closing of the control switch large currents (up to several thousand amperes) flow into the specimen. The experimental quantities (current, voltage drop, blackbody temperature, radiance temperature, heating and cooling rates) are measured with submillisecond time resolution.

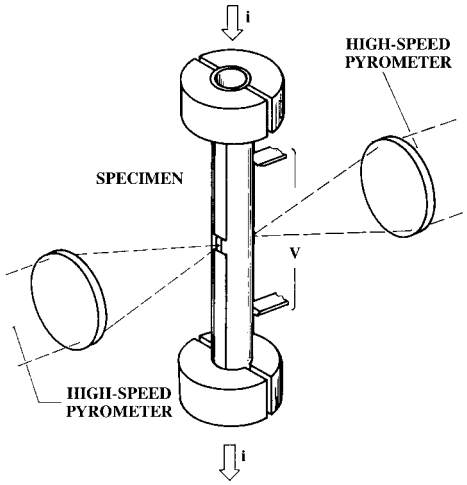

Fig. 1. Schematic representation of the classical pulse-heating technique. Dimensions are not to scale

Experiments are performed on tubular specimens (Fig. 1) with the following typical dimensions: length, 70-100 mm; diameter, 6-10 mm; wall thickness, 0.5 mm. A rectangular hole in the middle of the specimen defines a blackbody cavity with an emissivity greater than 0.98. The cross-sectional area is equalized by grinding away a strip of material to compensate for the material removed in providing for the blackbody cavity. The upper clamp is fixed, the lower clamp is movable to permit the thermal expansion of the specimen. The experiment takes place in an environmental chamber and may be performed either in vacuum or in inert atmosphere.

The blackbody and radiance temperatures are measured by high-speed pyrometers. The instruments used at the IMGC have been specifically designed and built for these experiments. Both are monochromatic instruments based on silicon detectors working in the visible or in the near

infrared according to the interference filter mounted during the calibration. Blackbody temperatures are measured by a millisecond resolution pyrometer [14] with a small target area (diameter 0.2 mm) to limit the size of the blackbody cavity. Radiance temperatures are measured with a microsecond resolution pyrometer [15]. Complete technical details regarding the high-speed pyrometers, their performance and calibrations may be found in the literature [16, 17].

During the experiment, the electrical signals corresponding to the experimental quantities are sent to a high-speed data acquisition system consisting of a multiplexer and a precision analog-to-digital converter. The data acquisition software is programmed to refer all measurements to the same time. Least-squares fittings of the experimental quantities versus time smooth the data and the properties of interest are computed at arbitrary temperatures within the experimental range.

Two thermophysical properties (electrical resistivity and normal spectral emissivity) may be computed directly from the experimental data collected during the heating period. If power balance equations (referring to the central portion of the specimen) are written for both the heating period and for the initial cooling period, a set of two equations in two unknowns (heat capacity and hemispherical total emissivity) is obtained. All the other quantities in these equations are either experimental data (input power, true temperature, heating and cooling rates) or specimen data (mass, geometric factors). Complete details regarding the experimental apparatus and the mathematics of the method, including different numerical techniques to compute the properties from experimental data may be found in earlier publications [2, 18, 19].

2.2. Reflectometric technique (strip specimen)

This technique developed at the IMGC is a high-speed version of an integrating sphere reflectometer of the comparison type (Fig. 2), in which the reflectivity of the specimen, that is being pulse-heated, is measured relative to the known reflectivity of a barium sulfate reference standard [20]. The measured quantity is the spectral directional-hemispherical reflectivity ρ of the specimen at the wavelength of the interference filter placed in front of the silicon detector placed at the top of the sphere. For opaque materials this quantity is the complement to one of the normal spectral emissivity ε of the specimen (Kirchhoff's law $\varepsilon = 1 - \rho$). A cut-off view of the measurement technique is shown in Fig. 3. The radiance temperature on one side of the strip is measured by a high-speed pyrometer. The strip is placed outside the porthole of a small integrating sphere placed inside the environmental chamber. The side of the strip facing the sphere reflects internally a modulated beam generated by a laser diode. The reflected beam is collected by the integrating sphere and measured by a silicon detector placed in the sphere ceiling. The detector operates at the same wavelength of the high-speed pyrometer and of the laser diode (at present, near 900 nm). A numerical lock-in method is used to discriminate between the reflected modulated beam and the continuous radiation emitted by the specimen itself at high temperatures. Additional technical details on the measurement technique and on the system can be found in an earlier publication [20]. A detailed evaluation of the measurement technique and a selection of experimental results have been published recently [21]. A multi-wavelength version of this type of measurement apparatus has also been developed in China [22].

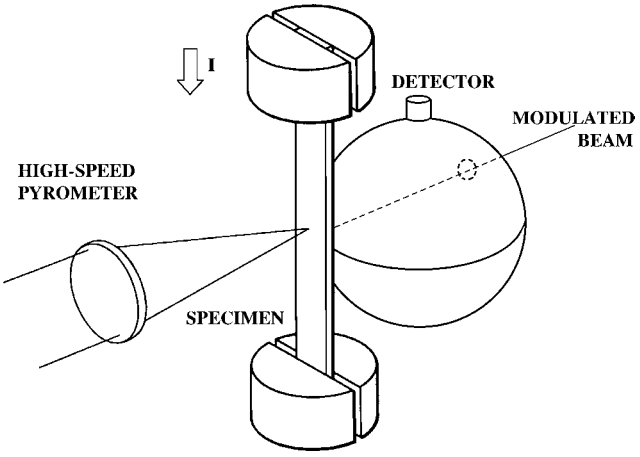

Fig 2. Schematic representation of the reflectometric pulse-heating technique. Dimensions are not to scale.

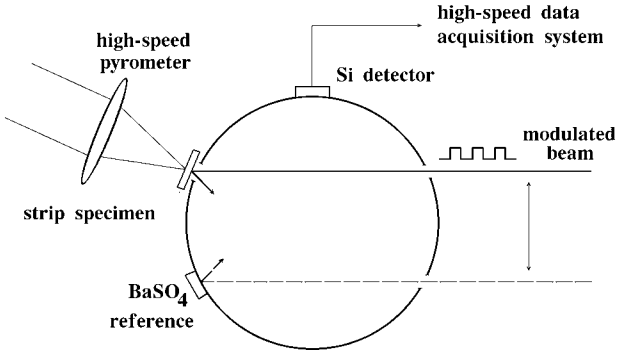

Fig 3. Cut-off view of the reflectometric pulse-heating technique. Dimensions are not to scale.

2.3. Laser polarimetry technique (rod specimen)

NIST in collaboration with Containerless Research Inc. has developed in the mid-1990s a laser polarimetry technique [23]. This is a method for determining the reflectivity of the specimen from the measurement of the polarization states of a laser radiation reflected from the surface of the rod-shaped specimen. Emissivity is then obtained in accordance with Kirchhoff's law for opaque materials. It is basically a high-speed version of the well-known ellipsometry measurements. Laser radiation is generated in accurately defined polarization states and the change of polarization after reflection from the surface of the specimen during pulse-heating is measured by an analyzer. The change of polarization is related to the optical constants and then to emissivity through a complex mathematical formalism based on the Fresnel equations. A

schematic diagram of the specimen and the configuration of the pyrometer and laser polarimeter used in the pulse-heating system at the NIST are shown in Fig. 4. The input beam consists of a He-Ne laser source at 633 nm. This beam enters an acousto-optic modulator, which provides 100% amplitude modulation of the transmitted radiation with a frequency of about 25 kHz. The modulated laser radiation then passes through some polarization optics. The reflected radiation from the specimen is collected by suitable imaging optics and is focused onto a field stop. Radiation transmitted through the field stop is then re-collimated and analyzed by a four-detector polarimeter. The outputs of the polarimeter and the pyrometer are digitized using 16-bit analog-to-digital converter cards installed in a personal computer. Data sets, that consists of four polarimeter and one or more pyrometer signals, are recorded at the rate of 2 kHz. Complete details of the millisecond laser polarimeter and of its integration into the pulse-heating facility at the NIST are given in the literature [23]. The laser polarimetry technique was also extended to microsecond resolution measurements and two systems have been described: one at the NIST [24] and another one at the Technical University of Graz, Austria [25].

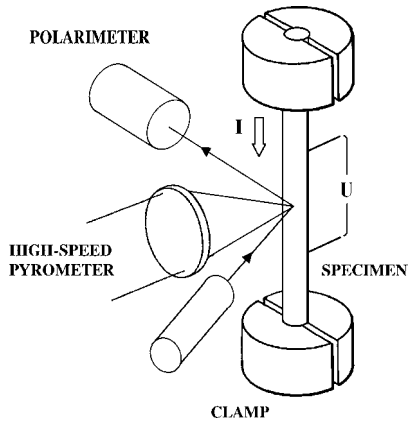

Fig 4. Schematic representation of the laser polarimetry pulse-heating technique. Dimensions are not to scale.

2.4. Laser polarimetry plus brief steady-state technique (strip specimen)

A further development of laser polarimetry combined with a brief steady-state experiment was jointly developed by the NRLM and the NIST in the 1990s [26], and subsequently improved at the NRLM-NMIJ [27]. In this variant of the pulse-heating method, the experiment is briefly stopped at a predefined high temperature, using the high-speed pyrometer signal in a feed-back loop to maintain the specimen at a fixed high temperature, as shown in Fig. 5. During the temperature plateau (Fig. 6), the input power matches the radiation losses, leading to an improved determination of the hemispherical total emissivity. The new technique is made possible by using mosfet switches operating in their linear region under control by an appropriate software program.

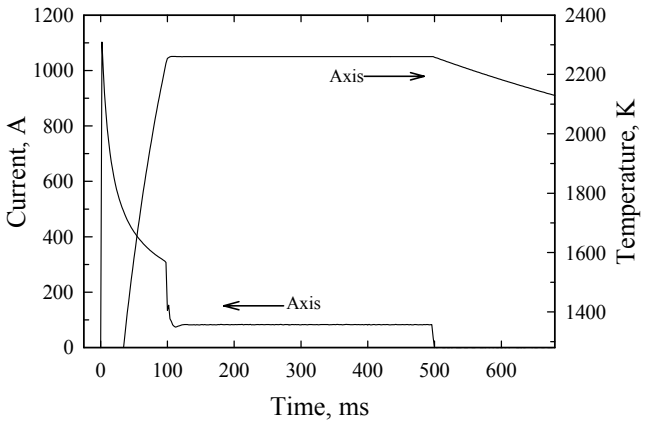

Fig 5. Variation of current and temperature in a typical experiment using the laser polarimetry plus brief steady state technique.

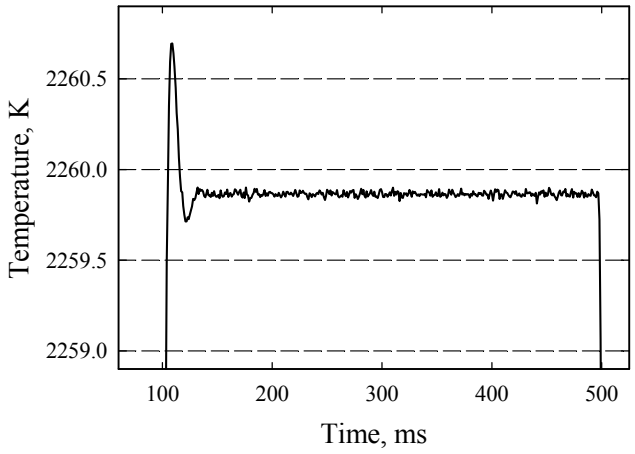

Fig 6. Typical temperature plateau in an experiment using the laser polarimetry plus brief steady state technique.

3. EXTENSION OF MEASUREMENTS TO OTHER THERMOPHYSICAL PROPERTIES

Significant research efforts have been devoted to the extension of the pulse-heating method to the measurement of other thermophysical properties, namely thermal expansion and thermal conductivity. At the IMGC these efforts were related to the development of a high-speed scanning pyrometer [28]. The instrument consists in a microsecond resolution pyrometer coupled with an optical scanning system based on a rotating mirror (details in Fig. 7) and associated electronics. The system is designed to effectively "freeze" in time the longitudinal temperature profile of the specimen by collecting 200-600 data points in 1-2 ms. A small linear correction is applied to take into account the temperature changes during the profile collection and to refer all temperature measurements to a single time.

3.1. Thermal expansion by scanning pyrometry

The thermal expansion apparatus is schematically represented in Fig. 7. The longitudinal expansion of the specimen is measured with a laser interferometer, while the temperature profile is determined by high-speed scanning pyrometry [28]. The experimental data are the expansion of a specimen (not at uniform temperature) and its temperature profile. If thermal expansion as a function of temperature in the range of interest can be represented by a low order polynomial, it may be demonstrated through some complex mathematics [29] that this polynomial is retrievable from the experimental data using least-squares techniques.

Different interferometer configurations are possible depending on the optical components mounted on the clamps, leading in all cases to a differential dilatometer that measures the displacement of the lower movable clamp with respect to the fixed upper clamp. The system is insensitive to small vertical movements and is easy to align. Accurate spatial positions are obtained by observing at each rotation the position of a wire placed across the specimen (detail in Fig. 7). The presence of the cold wire shows up as a marked dip in the pyrometer output and defines a fixed position in space. Spatial positions are computed from the speed of the rotating mirror and from geometrical considerations.

The thermal expansion experiment is completely under computer control and is performed during the heating phase. Additional experimental quantities are the current in the specimen, partial and total voltage drops in the specimen and the output from two thin-wire thermocouples spot-welded at the ends of the specimen. Pyrometric measurements cover approximately 80-90% of the temperature profile, but cannot be extended to the region near the clamps defined by sharp temperature gradients. The thin thermocouples provide temperatures at each rotation and are used to complete the profile by interpolation where no pyrometric measurements are possible. Further technical details and experimental results may be found in earlier publications [29, 30].

3.2. Thermal conductivity by scanning pyrometry

Any experiment to measure thermal conductivity must take place in a time scale compatible with the thermal conduction process, which is clearly what pulse-heating techniques generally try to avoid. Therefore the experiments must by adapted by either slowing down the pulse-heating technique (performing a dynamic heating experiment lasting 20-30 s) or by performing measurements during the free cooling period. In both cases thermal conduction becomes an important factor with the passing of time: the central portion of the specimen loses heat toward the clamps and the temperature profiles become rounded. With an accurate measurement of the temperature profiles, the differential equation governing the heat conduction process may be studied under dynamic conditions [31] and different techniques may be used to obtain the thermal conductivity.

Fig. 8 presents a schematic diagram of the thermal conductivity experiment. The largest part of the development work was done using cooling experiments. The specimen is brought to high temperatures with a current pulse; during cooling the heat content is dissipated by heat conduction and by radiation. The partial differential equation describing this process [31, 32] contains terms related to the heat capacity, to the hemispherical total emissivity and to the thermal conductivity. If the first two properties are determined using the same specimen during multiproperty pulse-heating experiments, thermal conductivity may be evaluated by accurate measurements of the temperature profiles established on the specimen during cooling.

Accurate temperature measurements via scanning pyrometry are essential for both the thermal expansion and thermal conductivity experiments. Radiance temperature measurements by the pyrometer must be transformed into true temperatures and the accuracy of the transformation is crucial for both techniques. Several methods are potentially

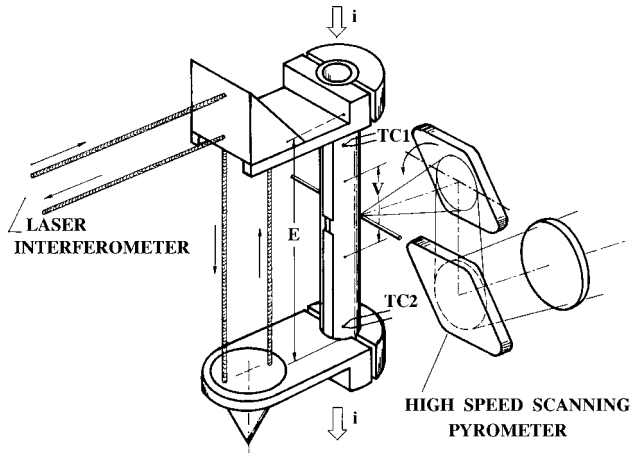

Fig. 7. Representation of the thermal expansion technique by scanning pyrometry. Dimensions are not to scale.

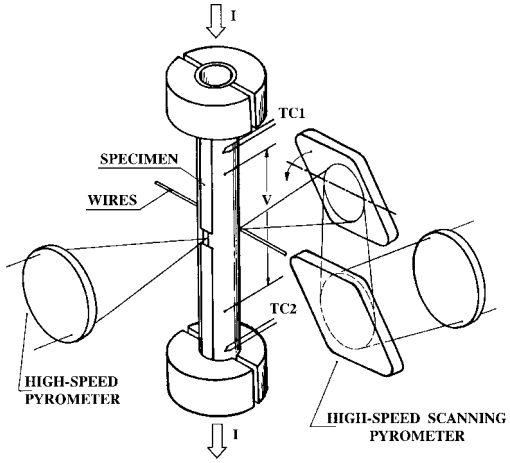

Fig. 8. Representation of the thermal conductivity technique by scanning pyrometry. Dimensions are not to scale.

available to obtain true temperatures from the scanning pyrometer measurements. If a tubular specimen with a blackbody hole is used, the normal spectral emissivity of the specimen surface may be measured during the heating phase and the results used during the cooling phase of the same experiment. Using a specimen without a blackbody hole (tube, rod or strip), the normal spectral emissivity of the specimen surface must be determined under pulse-heating conditions, using either a reflectometric technique or a laser polarimetry technique, as described before.

At present the thermal conductivity experiment is incomplete and needs further developments: the apparatus has been built and typical temperature profiles collected. Further work is needed to study the applicability of some of the methods described above (use of a symmetrical specimen, emissivity determinations). The computation of thermal conductivity from experimental temperature profiles is a complex mathematical task involving very large amounts of data [33]. The best results have been obtained by applying Kalman filter techniques [34], but further work is necessary to evaluate the uncertainty of this new measurement method.

An alternative technique for scanning pyrometry has been developed at the NIST using a linear array detector. A fast scanning pyrometer has been built, tested and used in various applications: complete technical details may be found in the literature [35].

3.3. Thermal expansion by interferometry

A different thermal expansion technique was developed at the NIST and extensively used for measurements on high temperature metals [36]. It is based on a modified Michelson-type interferometer with polarized radiation adapted to the pulse-heating apparatus, with the specimen acting as a double retro-reflector in one of the optical paths. A schematic diagram of the apparatus is shown in Fig. 9. Using a polarized beam splitter cube, the laser radiation is divided into a reference path and a measurement path. The reference path is in the lower part of Fig. 9, while the measurement path is in the central and upper part of the same figure and includes two optical flats machined on the sides of the specimen.

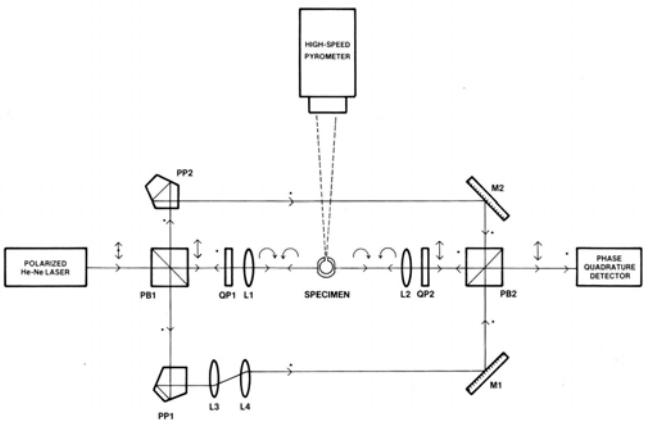

Fig. 9. Representation of the thermal expansion technique by interferometry. Dimensions are not to scale.

Various polarization and optical components are necessary to bend the measurement and reference paths and to recombine them into the four-quadrant detector. Temperature is measured directly in the blackbody hole machined in the tubular specimen. The technique has the advantage of combining directly the expansion and the true temperature, but measurements are possible only if the polished sides of the specimen retain their optical quality at high temperatures. Complete technical details of the technique and its implementation in the NIST system are described in the literature [36]. The same technique was used in a similar version of this interferometer built recently at the Austrian Foundry Research Institute [37, 38].

3.4. Mechanical properties with a Kolsky bar

The NIST has recently extended subsecond measurement techniques to selected mechanical properties, by combining a Kolsky bar apparatus with a pulse-heating system [39]. The Kolsky bar test consists of measurements performed on a small sample of material suspended by friction between two long steel bars. The incident bar is impacted on the end with a striker bar fired from an air gun. The impact produces a strain wave that moves down the incident bar, through the sample, and then to the second transmitted bar. The deformations in the sample material affect the strain pulse in the transmitted bar and the strain pulse reflected back into the incident bar. Signals from various sensors mounted in the center of the incident and transmitted bars recorded as a function of time are used to compute a stress-strain curve for the material under test. A schematic diagram of the apparatus under development at the NIST is presented in Fig. 10. The system can be operated either in a transient mode, where the current to the specimen is switched off at a preset temperature or time; or in the brief steady-state mode, where the specimen is heated rapidly to achieve a preset temperature (in the range up to 1300 K) in about 200 ms and then held isothermally for a for a brief period of the order of 1-2 s. The aim of the project is to determine stress-strain relationships at high temperatures for various test materials, simulating in the best possible way

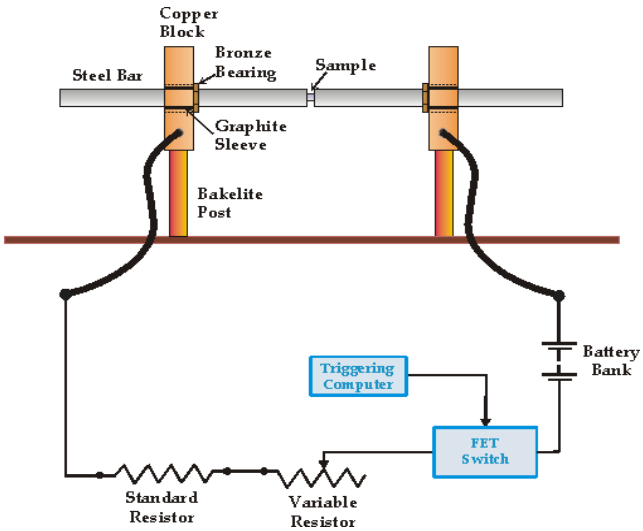

Fig. 10. Representation of the Kolsky bar apparatus. Dimensions are not to scale.

the conditions of high-speed machining processes. The present NIST group is particularly interested in the modeling of machining processes that need accurate high strain rate, high temperature material properties. A new microsecond resolution pyrometer based on an InGaAs detector was specifically built for these measurements [40], extending the capabilities of the NIST apparatus down to about 650 K. Further details of this new measurement system may be found in the literature [39].

4. MEASUREMENTS AT THE MELTING POINT

Experiments using subsecond pulse-heating are not limited to the solid phase, but may be extended to the solid-liquid phase transition. The time of the melting plateau and of the hydrodynamic collapse of the column of the liquid metal is long (from 50 to 100 ms depending on the mass to be melted) with respect to the time resolution of modern data acquisition systems. The introduction of mosfet switches capable of interrupting large currents with microsecond time resolution has also given the possibility of performing repeated experiments in which the specimen is taken to the melting point and the duration of the melting plateau is programmed [41]. A niobium strip may be taken 30-40 times to its melting point with a plateau of short duration (about 20 ms). Repeated plateaux of longer duration are possible, but mechanical stresses sometime bend the specimen in relation to electro-dynamic forces due to the lack of symmetry in the current flow.

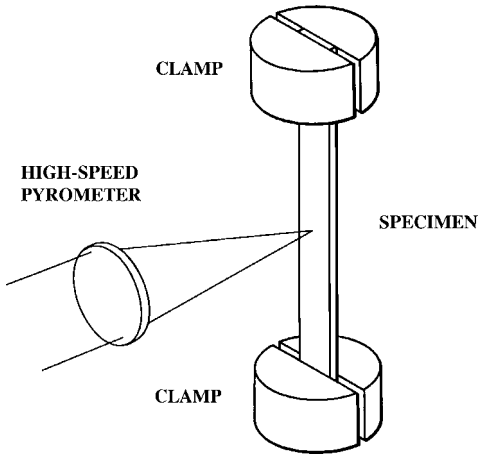

Fig 11. Measurement of the radiance temperature at the melting point. Dimensions are not to scale.

Typical examples are measurements of the radiance temperature of a material at its melting point (schematic diagram in Fig. 11). A series of joint experiments between the NIST and IMGC groups has demonstrated that this wavelength dependent quantity is well reproducible and may be considered as a possible secondary temperature standard [42]. The experiments have been performed on strip specimens and yield long and reproducible plateaux even if obtained from strips with widely different initial conditions. In relation to these results, the emissivities of selected materials at their melting point have also been proposed as possible reference values [43].

Measurements of the heat of fusion [44] may be performed using a composite specimen configuration (details in Fig. 12). The outer material must have a higher melting point than the inner material and the outside strips act as a container during melting. When the composite specimen is pulse-heated through the melting point of the inner material, the entire melting plateau is observed. The heat of fusion is computed by integrating the input power and by subtracting the energy lost by radiation during the melting plateau.

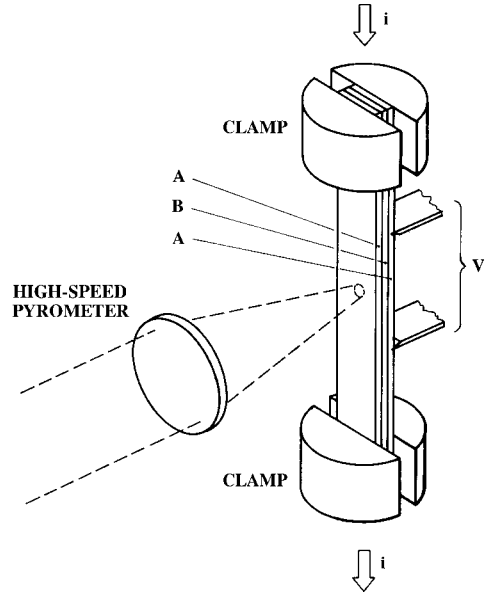

Fig 12. Representation of the measurement of the heat of fusion. Dimensions are not to scale.

Detailed studies of normal spectral emissivity changes at the melting point of niobium have been made possible by the development of the pulse-heating reflectometric technique. Repeated melting plateaux of short duration will produce a stable molten surface condition on the specimen after a few experiments [45] and it has been proved that experimental results of multiproperty measurements performed on strips with stable surface conditions have uncertainties comparable with results obtained on specimens with a blackbody configuration [46].

5. CHARACTERIZATION OF REFERENCE MATERIALS

The specific role of some NMIs in the development of pulse-heating techniques has been highlighted in a recent review [47]. That paper showed that the main interests of metrological institutions were related to: (i) the development of new measurement techniques, (ii) the accurate measurement of thermophysical properties at high temperatures, and (iii) the characterization of possible reference materials. The first item has already been considered, the other two items are presented here by summarizing some of the results of the indicated review [47]. Experimental results published in the literature by the indicated NMIs in the last thirty years for two different

properties (heat capacity and electrical resistivity) and for three candidate reference materials (niobium, molybdenum and tungsten) have been analyzed. The aim was to make an informal intercomparison of published experimental results, verifying at the same time the congruity between these data and the uncertainty figures quoted by the different NMIs. The results have been presented as deviation plots from some literature review, in which the reviewer analyzed the available experimental data and suggested a "recommended curve" based on his evaluation and theoretical considerations. This procedure has the advantage of putting the NMIs on the same footing, but it has the obvious limitation that the "recommended curves" contain only the information available when the analysis was made, sometime some decades ago.

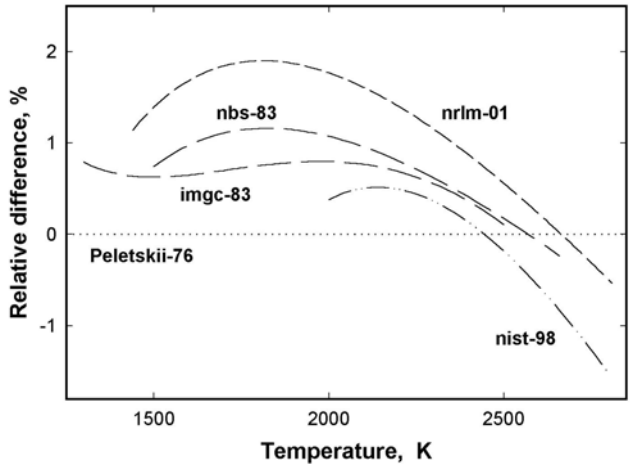

Fig 13. Intercomparison of published results of the electrical resistivity of molybdenum [47]. The curve identifications correspond to the following literature references: nbs-83 [48], imgc-83 [49], nist-98 [50], nrlm-01 [27] and Peletskii-76 [51].

The different experimental results on the electrical resistivity of molybdenum, extracted from literature data and harmonized with respect to different temperature scales and minor differences in measurement techniques, are presented in Fig 13. The data are given as a deviation plot from a literature review [51] (the zero line labeled "Peletskii-76") and clearly show a very good agreement among the NMI results, even if with a different temperature dependence with respect to the analyst recommendation. The uncertainty in electrical resistivity results claimed by the NMIs is of the order of 1-1.5% and all the published data over about twenty years are within the uncertainty band.

A similar analysis has been performed for the published experimental results on the heat capacity of molybdenum (Fig. 14). In this case the results show a spread of 2-3% with respect to a claimed uncertainty of 3-4%. It is important to note that these experimental results refer to measurements performed on different specimens, using different measurement techniques and were performed by different laboratories each using internal calibration methods and their respective realizations of the International Temperature Scale over a twenty-year time period. Even if these measurements were not performed according to the strict rules of the Mutual Recognition Arrangement protocols [52], they seem to indicate an adequate evaluation of the estimated uncertainties.

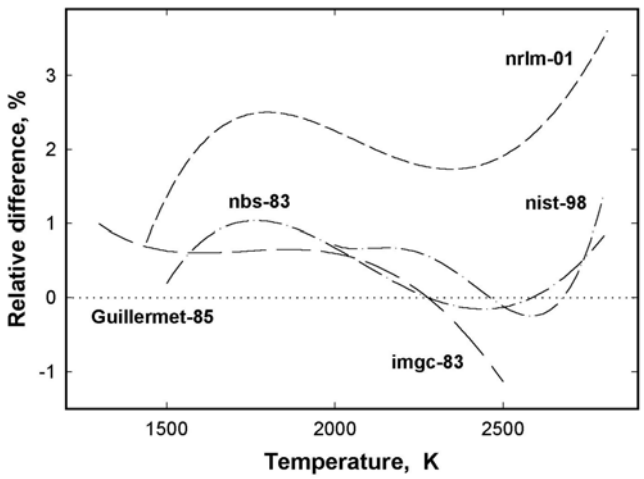

Fig 14. Intercomparison of published results of the heat capacity of molybdenum [47]. The curve identifications correspond to the following literature references: nbs-83 [53], imgc-83 [49], nist-98 [50], nrlm-01 [27] and Guillermet-85 [54].

Further specific details regarding these informal intercomparisons and additional results related to data on niobium and on tungsten can be found in the literature [47]. It may be concluded that published experimental results by the indicated NMIs over more than thirty years are compatible with each other and are an indirect confirmation of the validity of these pulse-heating techniques.

6. WORK IN PROGRESS

Some of the techniques previously described have been in use for several years, even if the related experimental apparatus have been constantly improved by taking advantage of the availability of new opto-electronic components and of better computers and data acquisition systems. The present section briefly describes areas where further work is in progress or where attention is needed for additional improvements.

6.1. Single point surface temperature measurements

It has already been noted that the current trend in this research area is toward applications, performing measurements on specimens with simple geometrical configurations (rods and/or strips). Specimens using a blackbody configuration (classical technique) require very accurate machining, but no particular precautions are necessary regarding the quality of the inner or outer specimen surface, because the blackbody integrates emitted radiation. On the other hand, in the two techniques using surface temperature measurements, laser polarimetry and integrating sphere reflectometry, the temperature measured on a small portion of the specimen surface is assumed to be representative of the entire specimen. This is a rather big assumption, and considerable research work is necessary to verify the limits of such an assumption. Some specific examples of the related problems are reported below.

The first important consideration is that the surface quality of the specimen should be reasonably uniform, so that the radiance temperature measurement is not influenced by special features, such as scratches that might change considerably the normal spectral emissivity of the measurement point with respect to the rest of the specimen surface. It should also be noted that the specimen moves during pulse-heating on account of its thermal expansion and therefore different parts of the specimen surface are seen by the pyrometer that measures on a fixed space position.

Repeated experiments performed on niobium strips using the reflectometric technique have shown that accurate measurements of thermophysical properties may be performed only after the specimen has reached a stable surface condition [45]. Repeated meltings change the specimen surface structure through diffusion and this process is a slow one in comparison to the pulse-heating experiments. Appropriate surface conditioning becomes an important factor in the accuracy of measurements performed on strips.

In a laser polarimetry experiment coupled with a brief steady-state experiment, the feedback control loop does maintain constant only the surface temperature of the measurement point. Experiments performed recently at the NMIJ have indicated that the electrical resistance of the central portion of the specimen does not remain constant during the brief (500 ms) steady-state portion of the experiment and therefore appropriate corrections are necessary, along with a re-consideration of the related mathematical analysis [55].

6.2. Modeling

The computation of thermophysical properties from experimental quantities in current-heating experiments has generally been performed using a mathematical formulation known as the "long thin rod" approximation.

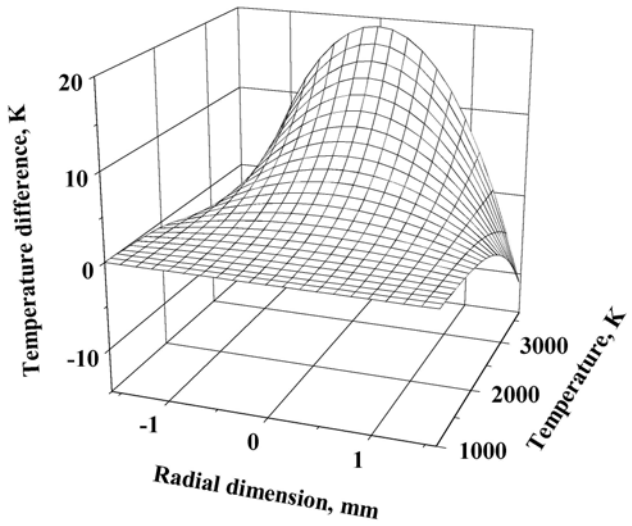

Fig. 15. Simulated temperature profile inside a tungsten rod of 3.2 mm diameter taken to its melting point in a pulse-heating experiment of 0.42 s duration

This simplification implies that the specimen is infinitely long and sufficiently thin to have no significant temperature gradients along both its length and its cross section. For very accurate experimental work, this assumption may have significant limitations. Some modeling work performed at the IMGC has considered the limitations of this assumption, developing a mathematical model that describes a long thick specimen, with a temperature gradient along its cross section [56]. A typical example of the estimated radial temperature differences on a pulse-heated tungsten rod is presented in Fig. 15.

Tubular specimens with a blackbody hole are not symmetrical on account of the presence of the hole and the necessity of a geometrical compensation of the cross-section (as shown in Fig. 1). Extensive modeling work performed at the Austrian Foundry Research Institute (OGI) has demonstrated a significant lack of temperature uniformity in the specimen due to the presence of the blackbody hole [57]. A typical example for a tubular specimen with a blackbody hole is shown in Fig. 16. Further work is going on the topic to assess the relevance of such non-uniformity on the blackbody temperature measurements and its effect on the computed thermophysical properties.

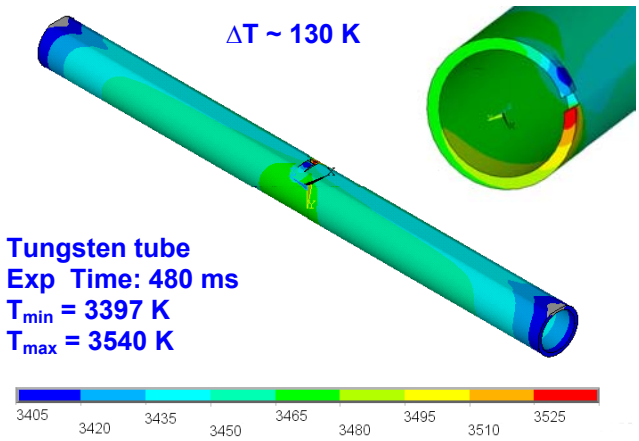

Fig. 16. Simulated temperature differences in a tungsten tube with a blackbody configuration taken to around 3500 K in a pulse-heating experiment of 0.48 s duration

6.3. Experimental problems

Pulse-heating systems are very complex pieces of apparatus. While every effort has generally been made over the years to improve them with new components, sometime early designs provide constraints that might be bypassed only with a new design and a complete reconstruction, and this is not always possible for reasons of time and cost.

An important problem with the current version of the integrating sphere reflectometry apparatus is the lack of symmetry in the experiment itself, due to the presence of the integrating sphere and to the fact that the experimental apparatus is an adaptation of a previous system. During the experiment, large currents flow into the specimen and in its support columns, creating electro-dynamic forces acting on the various parts of the circuit. As long as the specimen is in the solid phase, its stiffness prevents any movement. When the beginning of melting occurs, the specimen loses

much of its mechanical strength and there are some force components that tend to bend it away from the sphere. If this happens and the strip moves, the experiment is lost and the specimen no longer usable. Care must be exercised, both in the clamping action and in the correct duration of the melting plateau, to be able to perform repeated experiments at the melting point. The construction of a new experimental chamber, where the specimen is exactly symmetrical between the support columns, would improve considerably the possibility to obtain longer melting plateaux and limit the danger of specimen bending.

Using strip specimens, another experimental problem is related to the poor applicability of the measurement technique to very thin specimens. Experiments with strips with thickness over 0.25 mm are accurate and reproducible. Experiments with thinner specimen are sometime unpredictable, showing preferential melting paths on the specimen surface for unknown reasons. The uneven surface emissivity distribution makes results from such an experiment unusable for accurate thermophysical property measurements. This uneven emissivity distribution is observed during and after the first experiment only, with the second experiment to the melting point providing an uniform emissivity distribution over the entire specimen surface.

7. CONCLUSIONS

The modern development of current pulse-heating techniques started more than thirty-five years ago when some NMIs appreciated the potential of these methods and applied to them the developments in electronics and opto-electronics occurring in complementary fields. The large improvements in electronics and instrumentation (computers, data acquisition systems, software, detectors) have made the work simpler and more affordable in recent years. In relation to the interests of the NMIs, most of the earlier work was related to improve the capabilities of the measurement method, to develop new measurement techniques and to characterize possible high temperature reference materials [47]. The gradual increase of interest of other institutions (universities, applied research centers) in this research area seems to indicate that we are entering a period where the applications of these techniques for the characterization of materials and for the modeling of high temperature processes might become the driving factors. It is important to notice that pulse-heating techniques are still an open field with a large potential and that they can be further expanded to measure additional properties. This is a highly multidisciplinary area where a generation of young scientists versatile in physics, materials science, computers and applied electronics can provide important contributions both in further developments of the techniques and in their applications.

ACKNOWLEDGMENTS

Ared Cezairliyan (1934-1997) was the driving force in the world development of subsecond current-heating techniques: I was privileged to spend a post-doctoral period in his laboratory at the NBS and to be able to collaborate with him for many years.

The development of any current pulse-heating system requires a multidisciplinary effort based on different competences. Many people participated in the design, construction and operation of the various experimental systems presented in this review. The contributions of all the members of the various research groups around the world to the development and improvement of subsecond current-heating techniques are gratefully acknowledged.

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