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The Role of National Measurement Institutes in Subsecond Current-Heating Methods[1]

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The historical role of three national measurement institutes (NMIs), namely, the NBS-NIST (USA), the IMGC (Italy), and the NRLM-NMIJ (Japan), in the development of different pulse-heating methods is reviewed. In relation to their institutional interests, the indicated NMIs were mainly interested in the development and application of new measurement techniques, in the accurate measurement of thermophysical properties at high temperatures, and in the characterization of possible reference materials. An informal intercomparison of published experimental results obtained via pulse-heating techniques over 30 years on the electrical resistivity and heat capacity of niobium, molybdenum, and tungsten is presented, comparing these results with recommended curves from the literature. Good agreement is found among the pulse-heating results from the indicated NMIs, always within the combined uncertainties.

KEY WORDS: electrical resistivity; heat capacity; molybdenum; niobium; tungsten.

1. INTRODUCTION

In the last 35 years subsecond pulse-heating techniques have become an established measurement method for the determination of thermophysical properties at high temperatures. Some metrological institutions have been at the forefront of these developments, contributing both to the establishment of new measurement techniques and to the characterization of

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high temperature materials. This paper intends to highlight the role played by three national measurement institutes (NMIs), namely, the National Bureau of Standards (NBS, presently the National Institute of Standards and Technology, NIST) in the U.S.A., the Istituto di Metrologia "Gustavo Colonnetti" (IMGC) in Italy, and the National Research Laboratory of Metrology (NRLM, presently the National Metrology Institute of Japan, NMIJ).

In relation to their institutional and scientific interests, the activities of the indicated NMIs were concentrated in the following areas:

(i) development of new measurement techniques
(ii) accurate measurements of thermophysical properties at high temperatures
(iii) characterization of possible reference materials

The most important scientific results in the mentioned areas are reviewed, presenting an informal intercomparison of electrical resistivity and of heat capacity of three possible reference materials (niobium, molybdenum, and tungsten), extracted from literature data published over more than 30 years by the research groups of the indicated NMIs.

2. NEW MEASUREMENT TECHNIQUES

One of the main scientific activities of NMIs has traditionally been the development and establishment of new and accurate measurement methods. It should be emphasized that the experimental techniques reviewed and briefly described here were completely new at the time of their appearance, establishing new research areas and providing completely new approaches to measurement needs in the high-temperature region.

The historical development of modern subsecond pulse-heating techniques started in the late 1960s, with the pioneering work of the research group at the NBS led by Ared Cezairliyan (1934–1997). The NBS experimental apparatus [1] established the base of modern pulse-heating experiments: the IMGC [2] and the NRLM apparatus [3] adopted the same experimental approach while using different components, in particular, for the important high-speed pyrometry. The NBS group acted also as the catalyst of the research area, hosting scientists from different countries: both the authors of this paper were guest workers at the NBS-NIST in the 1970s and 1990s, respectively.

For over two decades, from the early 1970s to the early 1990s, the experimental measurements at the NBS-NIST and at the IMGC, the two laboratories active at that time, were based on the classical multiproperty measurement technique (Fig. 1). The experiment consists of the rapid heating of a tubular specimen by the passage of a current pulse of subsecond duration. 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 power switch, large currents (up to several thousand amperes) flow into the specimen. The experimental quantities (current, voltage drop, blackbody temperature, heating and cooling rates) are measured with submillisecond time resolution. Experiments are performed on tubular specimens; a rectangular hole in the central portion defines a blackbody cavity with an emissivity greater than 0.98. The cross-sectional area is made equal 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

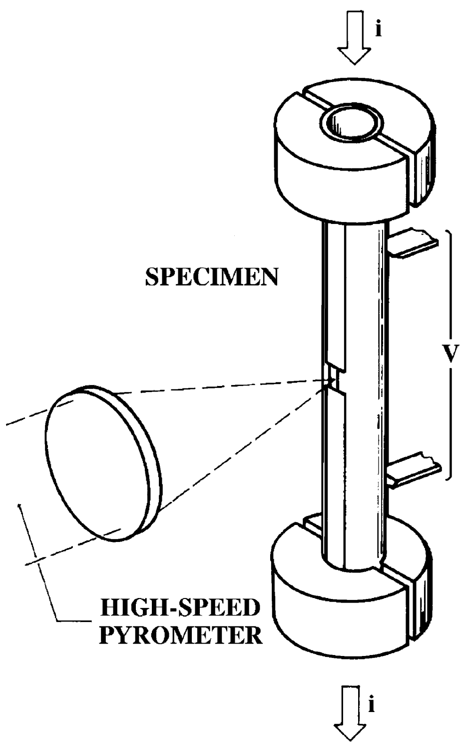

Fig. 1. Schematic representation of the classical technique.

experiment takes place in an environmental chamber and may be performed either in vacuum or in an inert atmosphere. The use of a tubular specimen with a blackbody hole provides several advantages such as mechanical stability, a stable geometry up to melting, and true temperature determinations. On the other hand, it also has some disadvantages such as the need to estimate for each specimen the blackbody emissivity, difficult machining through specialized equipment, the long manufacturing time, and the associated high costs.

During the 1990s some alternative measurement techniques were developed, in relation to the needs of application-oriented measurements. These new techniques used specimens of simple geometrical form (strips or rods), without a blackbody hole. Radiance temperature measurements performed on the specimen surface automatically required the development of techniques capable of simultaneously determining the normal spectral emissivity of the specimen under pulse-heating conditions.

The reflectometric technique [4], developed at the IMGC in the mid-1990s, is presented in Figs. 2 and 3. A small integrating sphere was inserted in the environmental chamber and the hemispherical spectral reflectivity of a strip specimen was measured with respect to the known reflectivity of a $BaSO_4$ reference specimen. According to Kirchoff's law for opaque materials, this quantity is the complement to one of the normal spectral emissivity. The technique is a high-speed version of the well known integrating sphere reflectometer and required the development and application of fast lock-in techniques to discriminate between the modulated reflected signal and the background radiation self-emitted by the specimen at high temperatures.

The laser polarimetry technique [5], developed at the NIST in collaboration with Containerless Research Inc. in the mid-1990s, is presented in Fig. 4. 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 is measured after reflection from the surface of the rod specimen during pulse-heating. 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 further development of laser polarimetry combined with a brief steady-state experiment was jointly developed by the NRLM and the NIST in the 1990s [6], and subsequently improved at the NRLM-NMIJ [3]. A block diagram of the NRLM-NMIJ experimental apparatus is shown in Fig. 5. 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 feedback loop to maintain the specimen at a

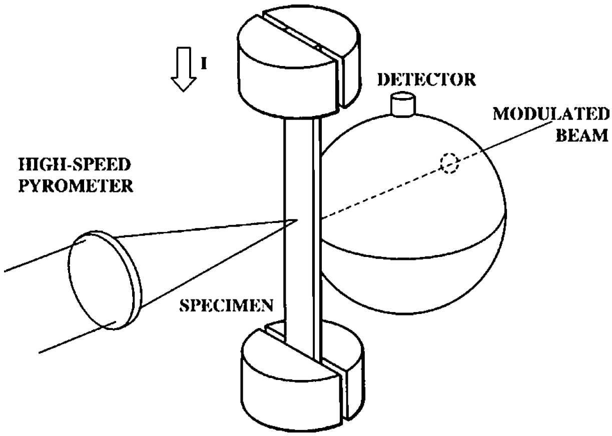

Fig. 2. Schematic representation of the reflectometric technique.

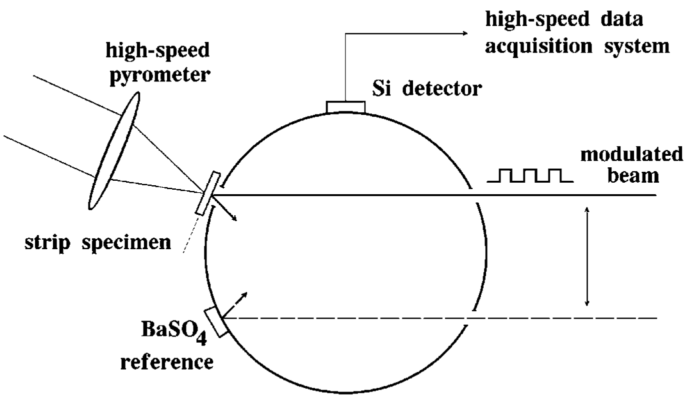

Fig. 3. Cross-sectional view of the reflectometric technique.

fixed high temperature, as shown in Fig. 6. During the temperature plateau (Fig. 7), 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 MOS-FET switches operating in their linear region under control by an appropriate software program.

Beyond the four different variants of the multiproperty pulse-heating method described previously, the NMIs introduced and applied several other techniques to extend pulse-heating measurements to other thermophysical properties. The NBS-NIST and the IMGC worked jointly for over 20 years in the accurate measurement of radiance temperatures and emissivities of selected high temperature metals [7]. A technique to measure

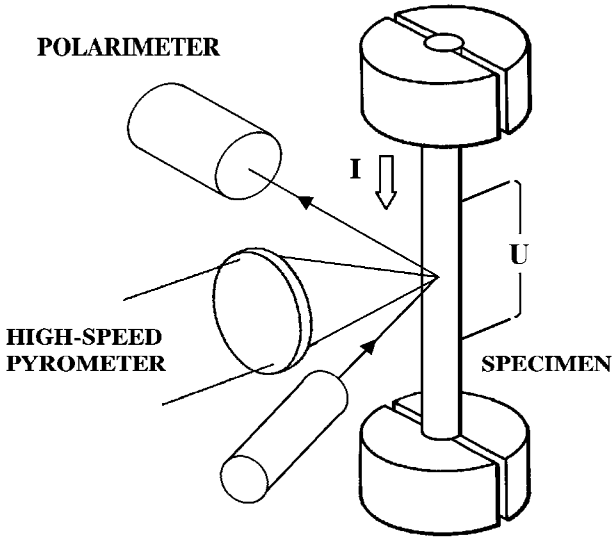

Fig. 4. Schematic representation of the laser polarimetry technique.

the heat of fusion using a combined three-strip specimen was developed and applied both at the NBS [8] and at the IMGC [9]. Thermal expansion at high temperatures was measured at the NBS-NIST by inserting a modified tubular specimen in the optical path of a polarization interferometer [10]. An alternative thermal expansion measurement technique was realized at the IMGC, measuring interferometrically the longitudinal expansion of the nonuniform specimen and simultaneously its temperature profile by high-speed scanning pyrometry [11]. A technique for measuring thermal conductivity under dynamic conditions using scanning pyrometry was also proposed [12].

3. INTERCOMPARISON OF THERMOPHYSICAL PROPERTIES

Another important area of scientific interest of the indicated NMIs was the accurate measurement of thermophysical properties at high temperatures, mainly for the characterization of possible reference materials. Naturally it is not easy to define *a priori* when a particular measurement is highly accurate; this is generally defined *a posteriori* by intercomparing results from different sources. The last decade has seen the creation and gradual implementation of the Mutual Recognition Arrangement (MRA) [13]. The basis of the MRA involves key comparisons (KC): formal

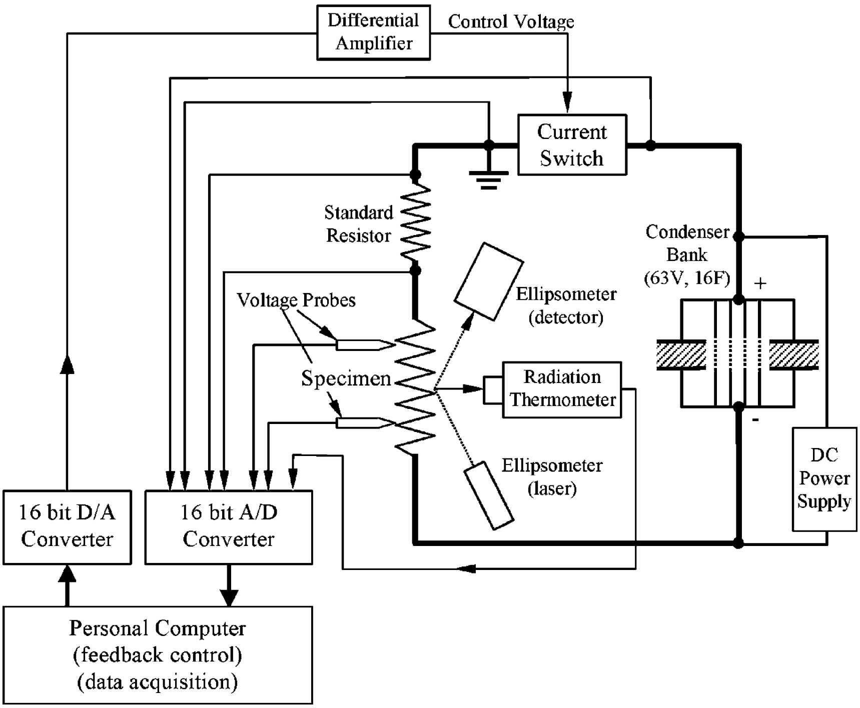

Fig. 5. Block diagram of the laser polarimetry plus brief steady-state technique.

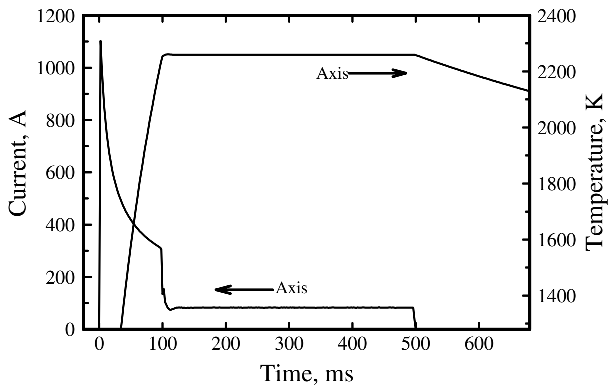

Fig. 6. Variation of current and temperature in a typical experiment using the laser polarimetry plus brief steady-state technique.

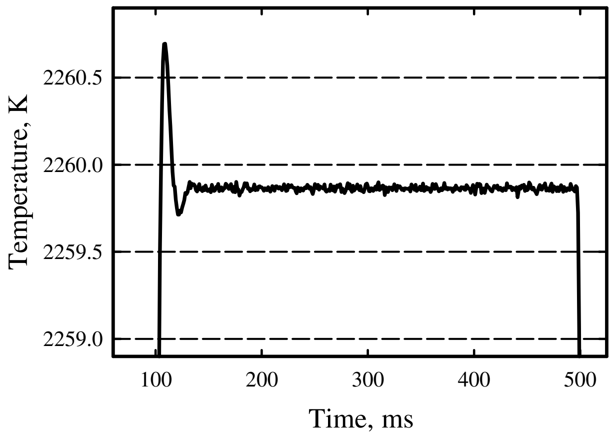

Fig. 7. Typical temperature plateau during an experiment using the laser polarimetry plus brief steady-state technique.

intercomparison exercises among the major metrological laboratories. The formalities of the MRA have gradually been extended to the area of thermophysical properties; a working group (WG9) of the Consultative Committee for Thermometry has been recently formed to evaluate the needs in this area.

The activities of the NMIs presented here took place in large part before the key comparisons became an established formality. Nevertheless, it is possible to demonstrate that the experimental results published by the indicated NMIs can be considered as informal intercomparisons as illustrated in the following paragraphs.

Literature results of two different properties, namely, electrical resistivity and heat capacity, for three different materials, namely, niobium, molybdenum, and tungsten, have been analyzed. The results are 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 on theoretical considerations. This procedure puts the NMIs on the same footing, but it has the obvious limitation that the "recommended curves" contain only the information available at the time when the review was made, in some cases in the 1970s.

Conversion to ITS-90 was made as necessary for all the reported literature data, both for NMI results and for "recommended curves." Thermodynamic data were converted according to the procedures suggested by Goldberg and Weir [14]. Minor corrections were also applied to the original NRLM data, to make them compatible with the experimental results

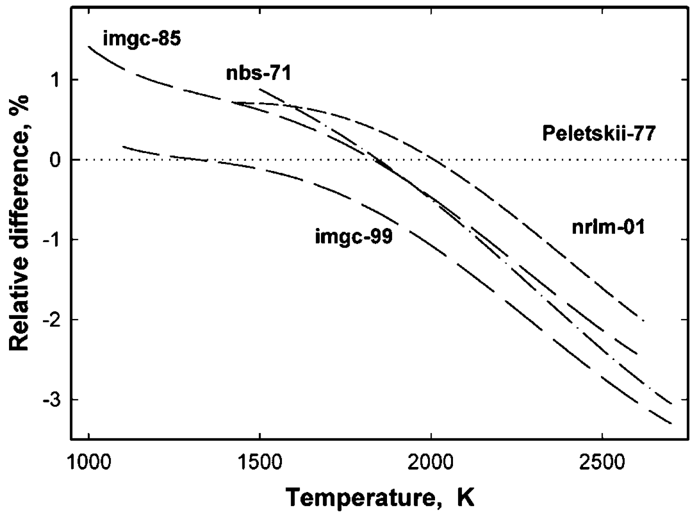

Fig. 8. Intercomparison of electrical-resistivity measurements of niobium by subsecond pulse-heating techniques.

at the NBS-NIST and at the IMGC. In these two laboratories the voltage probes that define the "effective" specimen are permanently fixed to the sample and therefore always include the same quantity of material. Probes at a fixed distance are used at the NRLM, with the specimen sliding over the probes during the experiment. The applied correction involves the thermal expansion function of each material; the data measured at the NBS-NIST for niobium [15], molybdenum [16], and tungsten [17] were used.

The various curves presented in the deviation plots (Figs. 8 – 13) are identified by the NMI acronym and by a number indicating the year of the related publication. A similar identification is provided for the zero baseline that represents the "recommended curve." Table I provides the complete correspondences between each curve identifier and the reference list.

3.1. Electrical Resistivity

The deviation plots of Figs. 8–10 present the literature results for the electrical resistivity of niobium, molybdenum, and tungsten. All the curves have been computed by using the geometrical dimensions at room temperature; no thermal expansion correction has been applied. The differences among experimental results should be considered in relation with the claimed uncertainties by the NMIs that are in the range of 1–2% with a coverage factor of 2.

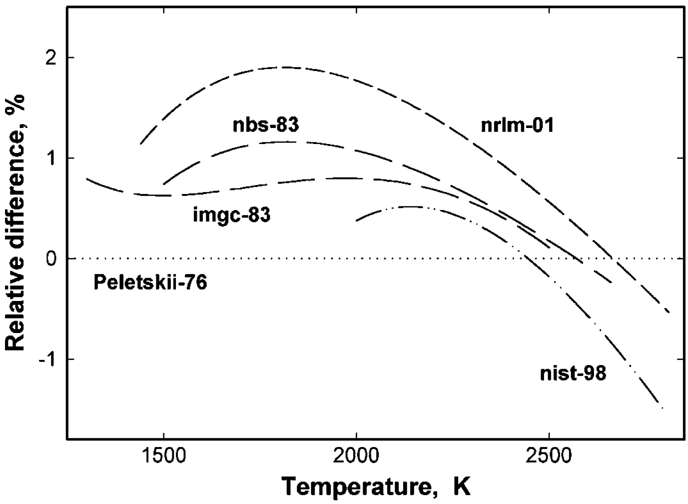

Fig. 9. Intercomparison of electrical-resistivity measurements of molybdenum by subsecond pulse-heating techniques.

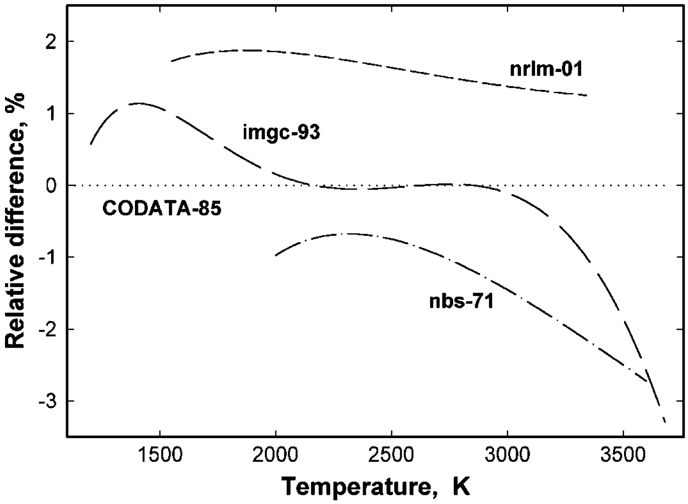

Fig. 10. Intercomparison of electrical-resistivity measurements of tungsten by subsecond pulse-heating techniques.

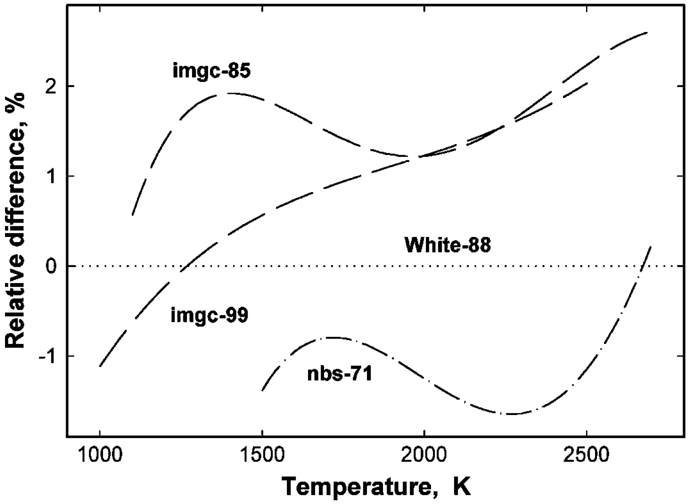

Fig. 11. Intercomparison of heat-capacity measurements of niobium by subsecond pulse-heating techniques.

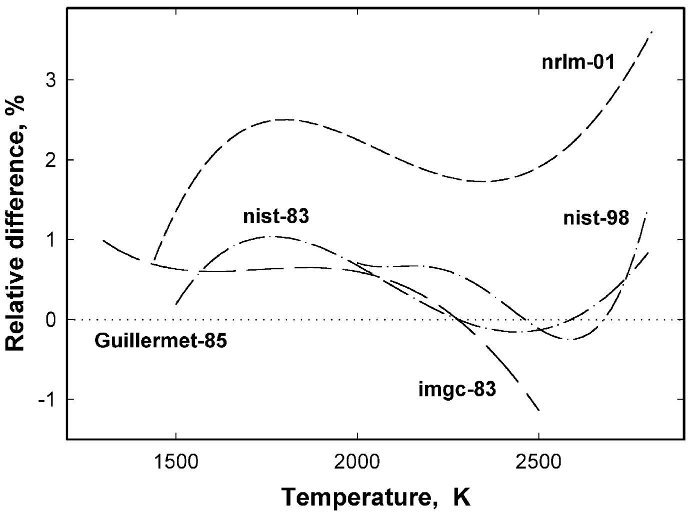

Fig. 12. Intercomparison of heat-capacity measurements of molybdenum by subsecond pulse-heating techniques.

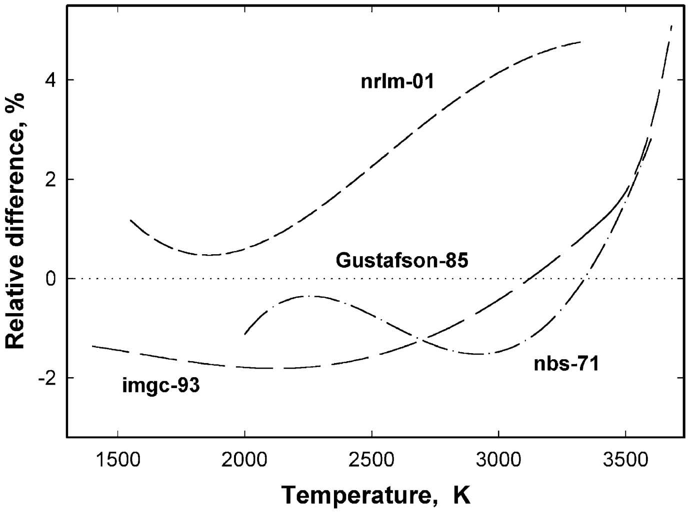

Fig. 13. Intercomparison of heat-capacity measurements of tungsten by subsecond pulse-heating techniques.

Table I. Table of Identifications of Curves in Figs. 8–13 with References

	Niobium		Molybdenum		Tungsten	
	Curve identification	Ref.	Curve identification	Ref.	Curve identification	Ref.
Electrical resistivity	Peletskii-77	[18]	Peletskii-76	[23]	CODATA-85	[27]
	nbs-71	[19]	nbs-83	[24]	nbs-71	[28]
	imgc-85	[20]	imgc-83	[25]	imgc-93	[29]
	imgc-99	[21]	nist-98	[26]	nrlm-01	[22]
	nrlm-01	[22]	nrlm-01	[22]		
Heat capacity	White-88	[31]	Guillermet-85	[32]	Gustafson-85	[34]
	nbs-71	[19]	nbs-83	[33]	nbs-71	[28]
	imgc-85	[20]	imgc-83	[25]	imgc-93	[29, 30]
	imgc-99	[21]	nist-98	[26]	nrlm-01	[22]
			nrlm-01	[22]		

The intercomparison of niobium results (Fig. 8) indicates a maximum difference of pulse-heating results of the order of 1%, with a temperature trend different from that of the "recommended curve" [18] above 2000 K, but highly consistent among the different NMI data.

The intercomparison of molybdenum results (Fig. 9) shows again reasonable agreement among NMI results with maximum differences of the order of 1.5%, and no significant differences in the temperature trend with respect to the "recommended curve" [23].

The intercomparison of tungsten results (Fig. 9) shows a wider scatter, with the maximum difference among NMI results being of the order of 3%. The results from two NMIs also show a different temperature trend above 3000 K, with respect to the "recommended curve" [27]. Somewhat higher uncertainties are to be expected at these very high temperatures.

3.2. Heat Capacity

The deviation plots of Figs. 11–13 present the literature results for the heat capacity of niobium, molybdenum, and tungsten. The differences among experimental results should be considered in relation with the claimed uncertainties by the NMIs that are in the range 3–4% with a coverage factor of 2.

The intercomparison of niobium results (Fig. 11) indicates a difference of pulse-heating results of the order of 2–3%, with a temperature trend slightly different (higher) from that of the "recommended curve" [31].

The intercomparison of molybdenum results (Fig. 9) shows again a reasonable agreement among NMI results with a maximum difference of the order of 3%. The NMI results at temperatures above 2500 K seem to indicate a larger heat capacity increase with respect to the "recommended curve" [32].

The intercomparison of tungsten results (Fig. 9) shows a wider scatter, with the maximum difference among NMI results being of the order of 3–4%. The results from all NMIs also exhibit a different temperature trend above 2500 K, with respect to the "recommended curve" [34].

It should be noted that in all cases the maximum deviations shown in the plots are lower than the maximum overlapping uncertainty regions.

4. CONCLUSIONS

The historical development of the activities of some NMIs in subsecond pulse-heating techniques was reviewed, demonstrating that their main interests in this research area were related to the developments of new measurement techniques and to the accurate measurements of thermophysical properties, mainly for the characterization of possible reference materials.

An informal intercomparison of published literature data from the NBS-NIST, the IMGC and the NRLM-NMIJ of the electrical resistivity and heat capacity of niobium, molybdenum, and tungsten has been presented. It should be noted that these intercomparisons, while not being conducted according to the formalities of KC protocols, refer to measurements performed at three different NMIs over a time span of 30 years, which involved different measurement techniques, materials from different sources, specimens of different geometries, and many different electronic and temperature calibrations. In all cases the intercompared results are within the overlapping combined uncertainties.

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