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Thermal Expansion of Niobium in the Range 1500–2700 K by a Transient Interferometric Technique[1]

A. P. Miiller[2] and A. Cezairliyan[2]

The linear thermal expansion of niobium has been measured in the temperature range 1500–2700 K by means of a transient (subsecond) interferometric technique. The basic method involves rapidly heating the specimen from room temperature up to and through the temperature range of interest in less than 1 s by passing an electrical current pulse through it and simultaneously measuring the specimen temperature by means of a high-speed photoelectric pyrometer and the shift in the fringe pattern produced by a Michelson-type interferometer. The linear thermal expansion is determined from the cumulative shift corresponding to each measured temperature. The results for niobium may be expressed by the relation

$$(l-l_0)/l_0 = 5.4424\times10^{-3} - 8.8553\times10^{-6}T + 1.2993\times10^{-8}T^2 - 4.4002\times10^{-12}T^3 + 6.3476\times10^{-16}T^4$$

where T is in K and l_0 is the specimen length at 20°C. The maximum error in the reported values of thermal expansion is estimated to be about 1% at 2000 K and not more than 2% at 2700 K.

KEY WORDS: high temperature; interferometry; niobium; pulse heating; thermal expansion.

1. INTRODUCTION

Measurements of thermal expansion at high temperatures ($T > 1100$ K) have generally relied on conventional steady-state or quasi-steady-state techniques such as push-rod dilatometry, X-ray diffractometry, or optical

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[2] Thermophysics Division, National Bureau of Standards, Gaithersburg, Maryland 20899, U.S.A.

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comparator methods. In these techniques, the long heating periods (minutes to hours) create problems associated with the increased heat transfer, loss of mechanical strength, specimen contamination, and evaporation, etc., which become particularly severe at temperatures above 2000 K. To minimize such difficulties, we recently developed a high-speed interferometric technique [1] in which the entire experiment is performed in less than 1 s. This involved adapting a Michelson-type interferometer to the existing pulse-heating system [2, 3] at the National Bureau of Standards (NBS).

The method is based on rapid resistive self-heating of the specimen from room temperature to the maximum temperature of interest by the passage of a subsecond-duration electrical current pulse through it and on simultaneously measuring the specimen temperature by means of a high-speed photoelectric pyrometer [4] and the shift in the fringe pattern produced by the interferometer. The polarized beam from a He–Ne laser in the interferometer is split into two component beams, one which undergoes successive reflections from highly polished flats on opposite sides of the specimen and one which serves as the reference beam. The linear thermal expansion of the specimen is determined from the cumulative fringe shift corresponding to each measured specimen temperature.

In the present study, the technique was used to determine the linear thermal expansion of niobium at temperatures in the range 1500 to 2700 K. Details regarding the construction and operation of the measurement system and other pertinent information, such as formulation of relations to determine thermal expansion from the fringe shift, error analyses, etc., are given in the earlier publication [1].

2. MEASUREMENTS

Three specimens were fabricated into precision-machined tubes from a (99.97% pure) rod of niobium by an electroerosion technique. A typical mass spectrometric analysis by the manufacturer of the rod yielded the following impurities (in ppm by mass): Ta, 200; C, 25; Si, 17; O, 15; Ag, 6; $N < 5$; Al and Fe, 4 each; Cr and Si, 2 each; Ni, 1; the total amount of all other detected elements was less than 5 ppm, the amount of each element being less than 1 ppm. The nominal dimensions of each specimen tube were as follows: length, 76 mm; outside diameter, 6.6 mm; inside diameter, 5.3 mm. A small rectangular sighting hole (0.5×1 mm) was fabricated through the wall at the middle of each tube, thereby approximating a blackbody cavity for the pyrometric temperature measurements. The sighting hole was positioned 0.8 mm off center from the tube axis to improve the blackbody quality. In order to compensate for the cross-

sectional nonuniformity created by the hole, a portion of the specimen was removed by grinding a flat along the length of the tube, excluding the 1-mm length of the hole. For the interferometric measurements, highly polished parallel flats were fabricated on opposite sides along the length of each tubular specimen. The distance between the optical flats (i.e., the specimen "length"; l_0) was measured at 20°C by the Precision Engineering Division at the NBS using a comparative gauge block technique. The results are given in Table I along with estimates of the axial nonparallelism and departure from flatness of the two reflecting surfaces.

Each specimen tube, in turn, was mounted vertically between two water-cooled electrodes inside the test chamber, which was then evacuated to a pressure of about 1 mPa ($\sim 10^{-5}$ Torr). The upper electrode is stationary, whereas the lower electrode is attached (through a linear guide) to a flexible connection which enables the specimen to expand downward along its length during pulse heating.

Prior to each pulse experiment, adjustments were made to the voltage from a battery bank and to a resistance in series with the specimen in order to achieve the desired heating rate. The specimen was then rapidly heated from "room" temperature (~ 18°C) to the desired temperature by passing an electrical current pulse through it; the duration of the current pulse varied from about 630 to 690 ms. The temperature interval of the measurements (1500–2700 K) was divided into five overlapping tem-

Table I. Results of Measurements[a] Performed on the Specimen Optical Flats by a Comparative Gauge Block Technique

	Specimen No.		
	1	2	3
Distance between flats (mm)	6.0648 (6.0615)[b]	6.0625 (6.0592)	6.0620 (6.0541)
Axial parallelism (min of arc)	0.4 (0.2)	0.4 (0.4)	1.5 (3.6)
Flatness[c] (waves/25 mm)	1 (1)	5 (1)	7 (10)

[a] Measurements were performed along the center portion of the specimen tube, over an axial distance of 6 mm. Maximum downward movement (expansion) of the center portion during pulse heating was about 0.5 mm.

[b] Results of measurements performed upon completion of the pulse-heating experiments are given in parentheses.

[c] Given in terms of the wavelength 632.8 nm.

perature ranges in order to optimize the signal resolution of the high-speed pyrometer. For a given specimen, single pulse experiments were performed successively through each temperature range beginning with the lowest range. Heating rates varied typically from about 2600 $K \cdot s^{-1}$ in the lowest temperature range to about 3100 $K \cdot s^{-1}$ in the highest range.

During each pulse experiment, the specimen temperature was measured every 0.833 ms, whereas the fringe shift was measured every 0.1 ms. These data were recorded by means of two digital storage oscilloscopes, each capable of storing 16,000 data points with a full-scale signal resolution of about 1 part in 4000. After the experiment, the recorded data were transferred to a minicomputer for subsequent analyses.

Upon completion of the experiments, the high-speed pyrometer was calibrated using a tungsten-filament standard lamp which, in turn, had been calibrated against the NBS Photoelectric Pyrometer by the Radiometric Physics Division at the NBS. In addition, the reference dimension at 20°C (l_0) for each specimen was remeasured, and the results, along with estimates of nonparallelism and flatness of the reflecting surfaces after pulse heating, are given, in parentheses, in Table I. All temperatures reported in this work, unless explicitly stated otherwise are based on the International Practical Temperature Scale of 1968 [5].

3. RESULTS

The linear thermal expansion of the specimen was determined at each recorded temperature from the cumulative fringe shift Δn by means of the relation

$$(l - l_0)/l_0 = (\lambda/2l_0)\,\Delta n \tag{1}$$

where λ is equal to 632.8 nm and l_0 is the specimen "length" at 20°C. In order to account for any difference between the initial temperature of the specimen and 20°C, a "zero" correction was applied to Δn on the basis of expansion data near room temperature reported in the literature [6].

In order to determine the scatter in the data, the expansion/temperature data pairs for each specimen were fitted by a polynomial function of temperature by the least-squares method; the polynomial functions representing the results for individual specimens are given in Table II. The deviation of individual data points from the smooth function for each specimen is illustrated in Fig. 1. The random fluctuations among data points within a given temperature range are due primarily to uncertainty in determining the fringe count from a limited number of data points per fringe (about 20 at 1500 K, decreasing to about 9 at 2700 K). In certain

Table II. Results of Fitting, by Means of the Least-Squares Method, the Expansion/Temperature Data Pairs for the Individual Specimens and the Combined Data for All Three Specimens by Polynomial Functions in Temperature (in K) of the Form $(l-l_0)/l_0 = a_0 + a_1 T + a_2 T^2 + a_3 T^3 + a_4 T^4$

Specimen No.	Number of data pairs	SD[a] (%)	Polynomial coefficient[b]				
			$10^3 a_0$	$10^6 a_1$	$10^8 a_2$	$10^{12} a_3$	$10^{16} a_4$
1	564	0.05	2.7247	−3.2862	0.87094	−2.9585	4.5532
2	552	0.06	2.9597	−3.5586	0.89040	−3.0273	4.6674
3	561	0.05	7.5435	−13.242	1.6345	−5.5048	7.6646
All specimens	1677	0.31	5.4424	−8.8553	1.2993	−4.4002	6.3476

[a] Standard deviation of an individual value of $(l-l_0)/l_0$ from the smooth function.
[b] Based on the specimen reference length (l_0) at 20°C.

cases, differences between data from overlapping temperature ranges also contribute to the scatter of data points.

The final results for niobium were obtained by combining the expansion/temperature data pairs for the three specimens and then fitting them by a quartic polynomial function of temperature. The function that represents (standard deviation = 0.3%) the results for linear thermal expansion of niobium in the temperature range 1500–2700 K is

$$(l-l_0)/l_0 = 5.4424 \times 10^{-3} - 8.8553 \times 10^{-6} T + 1.2993 \times 10^{-8} T^2 - 4.4002 \times 10^{-12} T^3 + 6.3476 \times 10^{-16} T^4 \quad (2)$$

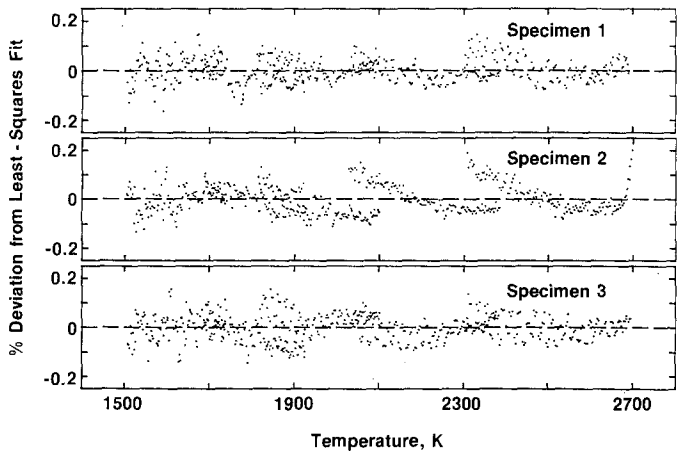

Fig. 1. Deviation of expansion/temperature data pairs for the individual specimens from the smooth functions (see Table II) representing the least-squares fits to the data.

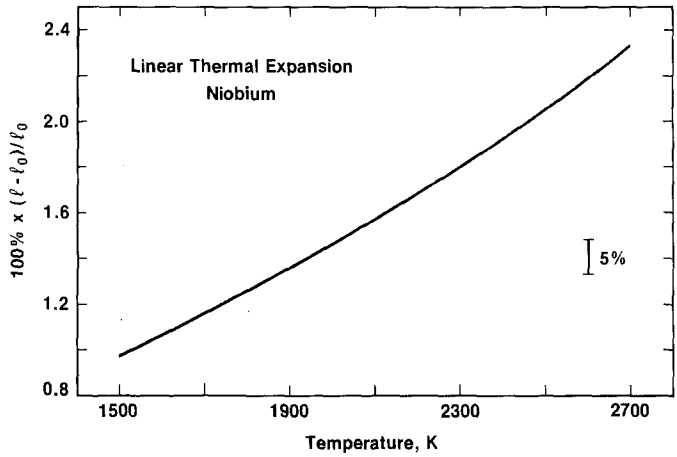

Fig. 2. Smoothed results for the linear thermal expansion of niobium as expressed by Eq. (2).

where T is in K. The smoothed results, as defined by Eq. (2), are illustrated in Fig. 2 and are given at intervals of 100 K in Table III.

Differences in data for the three specimens are illustrated in Fig. 3, which presents the deviation of the smoothed results for individual specimens from the "grand" fit given by Eq. (2). The maximum deviation of the smoothed results from the overall least-squares fit is about 0.4%.

Table III. Smoothed Results[a] for the Linear Thermal Expansion of Niobium

Temperature (K)	$10^2(l-l_0)/l_0$ (%)
1500	0.976
1600	1.067
1700	1.162
1800	1.260
1900	1.361
2000	1.466
2100	1.574
2200	1.686
2300	1.803
2400	1.926
2500	2.055
2600	2.192
2700	2.338

[a] Based on the specimen reference length (l_0) at 20°C.

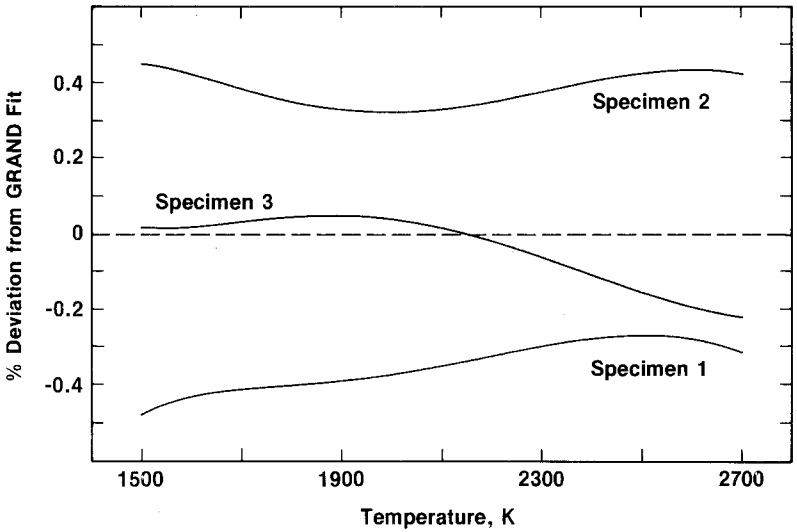

Fig. 3. Deviation of the smoothed thermal expansion results for the individual specimens from Eq. (2), which represents the least-squares fit to the combined data for three specimens.

4. ESTIMATE OF ERRORS

A detailed analysis of errors in such experimental quantities as temperature, fringe count, and specimen "length" at 20°C was given in an earlier publication [1]. Specific items in the error analysis were recomputed whenever the present conditions differed from those in the earlier publication. The resultant estimated maximum error in the reported values of thermal expansion is about 1% at 2000 K and not more than 2% at 2700 K.

5. DISCUSSION

Figure 4 compares the expansion data for niobium reported in the literature with present results as expressed by Eq. (2). A "zero" correction was applied to the literature data whenever the reported reference temperature differed from 20°C. No attempt was made to convert the reported temperatures to IPTS-68 since the particular temperature scale used in the different studies could not be determined unambiguously in all cases and since such "corrections" would be relatively small (less than 0.5%).

The earliest measurements of niobium expansion appear to be those performed by Edwards et al. [7] (curve 1) using X-ray diffractometry and

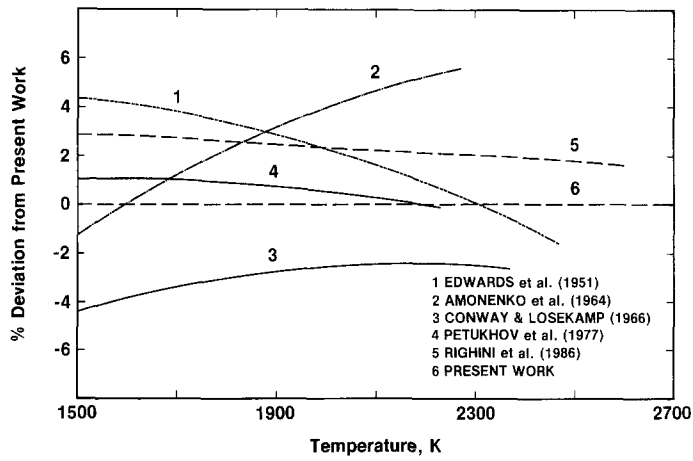

Fig. 4. Deviation of thermal expansion data for niobium reported in the literature from the present results as expressed by Eq. (2). Curve numbers refer to the following: (1) Edwards et al. [7]; (2) Amonenko et al. [8]; (3) Conway and Losekamp [9]; (4) Petukhov et al. [10]; (5) Righini et al. [11]; (6) the present work. The different measurement techniques are indicated as follows: X-ray diffractometry (—··—); push-rod dilatometry (—·—); optical comparator methods (——); high-speed pulse interferometry (----).

by Amonenko et al. [8] (curve 2) using push-rod dilatometry. As may be seen in Fig. 4, their reported values exhibit very different trends with changing temperature and deviate from the present results by as much as 6%. Better agreement with the present work is shown by the expansion data of Conway and Losekamp [9] (curve 3) and of Petukhov et al. [10] (curve 4), which were obtained by optical comparator methods; however, only those values reported by Petukhov et al. lie within experimental uncertainties.

The most recent measurements, reported by Righini et al. [11] (curve 5) involved a pulse interferometric technique similar to that used in the present work. The major difference is that, in their technique, thermal expansion during rapid pulse heating is determined from changes in the length of the specimen tube between the end-clamps rather than across the diameter of the tube at its center, as in the present case. This approach yields a much larger fringe shift (and, in principle, greater sensitivity) but at the expense of complicating the data analysis due to large temperature gradients in the specimen near the end-clamps. Righini et al. estimate the uncertainty in their expansion data to be about 2% throughout the entire temperature range of their measurements (1000–2600 K). It may be seen

from Fig. 4 that the agreement between their results for thermal expansion of niobium and the present work lies within the combined experimental uncertainties.

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