

Thermal Expansion of Tungsten in the Range 1500–3600 K by a Transient Interferometric Technique¹

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The linear thermal expansion of tungsten has been measured in the temperature range 1500–3600 K by means of a transient (subsecond) interferometric technique. The tungsten selected for these measurements was the standard reference material SRM 737 (a standard for thermal expansion measurements at temperatures up to 1800 K). The basic method involved 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 was determined from the cumulative shift corresponding to each measured temperature. The results for tungsten may be expressed by the relation

$$(l - l_0)/l_0 = 1.3896 \times 10^{-3} - 8.2797 \times 10^{-7}T + 4.0557 \times 10^{-9}T^2 \\ - 1.2164 \times 10^{-12}T^3 + 1.7034 \times 10^{-16}T^4$$

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 approximately 2% at 3600 K.

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

1. INTRODUCTION

Measurements of thermal expansion at temperatures above the limit of accurate Fizeau interferometric methods ($T > 1100$ K) have generally relied

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on other steady-state or quasi-steady-state techniques such as push-rod dilatometry, X-ray diffractometry, and optical 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 temperature. 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].

In the present study, the technique was used to determine the thermal expansion of tungsten at temperatures in the range 1500 to 3600 K. The tungsten selected for the present work was the standard reference material SRM 737, which has been certified by the Office of Standard Reference Materials (OSRM) at NBS as a standard for measurements of thermal expansion at temperatures up to 1800 K [5].

2. MEASUREMENTS

The tungsten (SRM 737) was supplied by the OSRM in the form of a polycrystalline rod which had been manufactured by a sintered powder metallurgical process. A chemical analysis of the material indicated a purity of 99.96 wt %, whereas the electrical residual resistance ratio ($RRR = 60$) indicated a purity of 99.98 wt % [6].

The tungsten rod was fabricated into three precision-machined tubes by an electroerosion technique. The nominal dimensions of each specimen tube were as follows: length, 76 mm; outside diameter, 6.4 mm; and 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 near the center of the tube defines the specimen "length" in the present measurement technique.

In preparation for the experimental work, the specimen "length" at 20°C (i.e., l_0) was measured by the Precision Engineering Division at NBS using a comparative gauge block technique. The specimens were then heat treated by subjecting them to five to eight heating pulses (up to about 2400 K). The reference dimension l_0 of each specimen was remeasured prior to the start of pulse experiments to determine thermal expansion and again upon completion of the experiments. The results of measurements performed on the specimen optical flats are given in Table I along with estimates of axial nonparallelism and departure from flatness.

Table I. Results of Measurements^a Performed at 20°C on the Specimen Optical Flats by the Precision Engineering Division at NBS Using a Comparative Gauge Block Technique

Measurement ^b	Specimen No.								
	1			2			3		
	A	B	C	A	B	C	A	B	C
Distance between flats, l_0 (mm)	6.105	6.108	6.106	6.095	6.095	6.096	6.092	6.092	6.092
Axial nonparallelism (min. of arc)	0.8	0.8	0.3	1.1	1.4	1.2	0.8	0.6	0.4
Flatness ^c (waves/25 mm)	9	9	1	3	3	2	3	3	2

^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 Measurements were performed in the following sequence: A, prior to heat treatment of the specimens; B, prior to the start of the pulse experiments to determine thermal expansion; C, after completion of the pulse experiments.

^c Given in terms of the wavelength 632.8 nm.

For pulse heating, each specimen tube was mounted vertically between two water-cooled electrodes inside the test chamber, which was evacuated to a pressure of about 1 mPa ($\sim 10^{-5}$ Torr). The upper electrode was stationary, whereas the lower electrode was attached (through a linear guide) to a flexible connection which enabled 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 ($\sim 16^\circ\text{C}$) to the desired temperature by passing an electrical current pulse through it; the duration of the current pulse varied from about 670 to 710 ms. The temperature interval of the measurements (1500–3600 K) was divided into seven overlapping temperature 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 typically varied from about $3500 \text{ K} \cdot \text{s}^{-1}$ in the lowest temperature range to about $6600 \text{ K} \cdot \text{s}^{-1}$ in the highest range.

During each pulse experiment, the specimen temperature was measured every 0.8 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. All temperatures reported in this work, unless explicitly stated otherwise, are based on the International Practical Temperature Scale of 1968 (IPTS-68) [7].

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

$$(l - l_0)/l_0 = (\lambda/2l_0) \Delta n \quad (1)$$

where λ is equal to 632.8 nm and l_0 is the specimen "length" at 20°C (measurement B in Table I). In order to account for any difference between the initial temperature of the specimen and 20°C , a "zero" correction was

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 ⁷ a ₁	10 ⁹ a ₂	10 ¹² a ₃	10 ¹⁶ a ₄
1	781	0.08	1.3550	-7.2386	3.9555	-1.1811	1.6624
2	765	0.06	1.3732	-7.8587	4.0210	-1.2051	1.6877
3	778	0.06	1.1601	-4.7771	3.8696	-1.1731	1.6679
All specimens	2324	0.15	1.3896	-8.2797	4.0557	-1.2164	1.7034

^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.

applied to Δn on the basis of expansion data near room temperature reported in the literature [8].

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

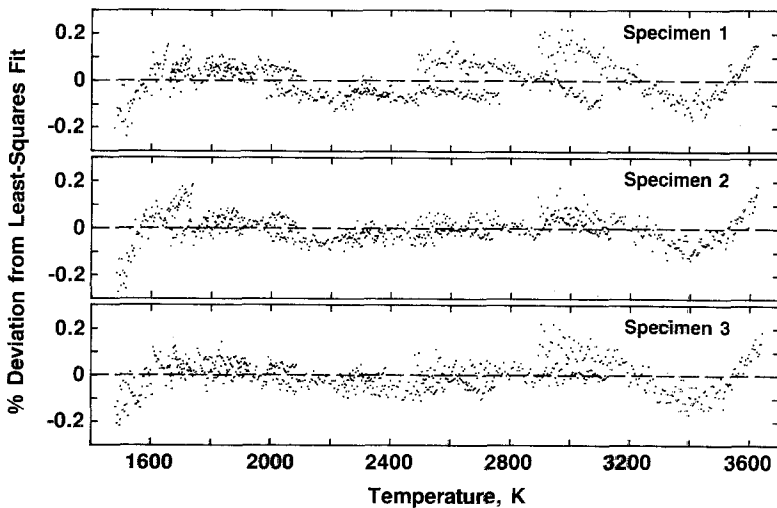


Fig. 1. Deviation of expansion/temperature data pairs for the individual specimens from the smooth functions (see Table II) representing the least-squares fits to the data.

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 25 at 1500 K, decreasing to about 9 at 3600 K). In certain cases, differences between data from overlapping temperature ranges also contribute to the scatter of data points.

The final results for tungsten 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 = 0.15%) the results for linear thermal expansion of tungsten in the temperature range 1500–3600 K is

$$(l - l_0)/l_0 = 1.3896 \times 10^{-3} - 8.2797 \times 10^{-7}T + 4.0557 \times 10^{-9}T^2 - 1.2164 \times 10^{-12}T^3 + 1.7034 \times 10^{-16}T^4 \quad (2)$$

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 along with values of expansivity determined from $(1/l_0)(dl/dT)$.

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 0.2%.

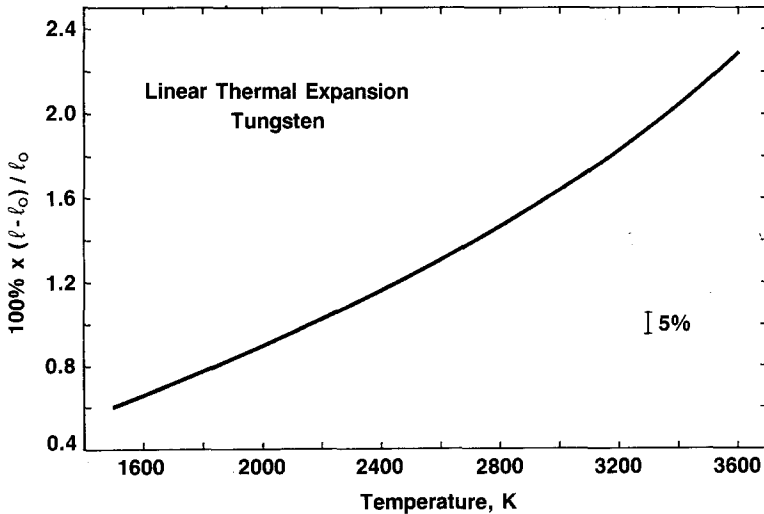


Fig. 2. Smoothed results for the linear thermal expansion of tungsten as expressed by Eq. (2).

Table III. Smoothed Results^a for the Linear Thermal Expansion and Expansivity of Tungsten

Temperature (K)	$10^2(l - l_0)/l_0$ (%)	$10^6(1/l_0)(dl/dT)$ (K ⁻¹)
1500	0.603	5.43
1600	0.658	5.60
1700	0.715	5.76
1800	0.773	5.92
1900	0.833	6.08
2000	0.895	6.25
2100	0.958	6.42
2200	1.024	6.61
2300	1.091	6.81
2400	1.160	7.04
2500	1.232	7.29
2600	1.306	7.57
2700	1.383	7.88
2800	1.464	8.23
2900	1.548	8.62
3000	1.636	9.06
3100	1.729	9.55
3200	1.827	10.09
3300	1.931	10.69
3400	2.041	11.35
3500	2.158	12.07
3600	2.283	12.87

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

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 approximately 2% at 3600 K.

5. DISCUSSION

Figure 4 compares expansion data for tungsten 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

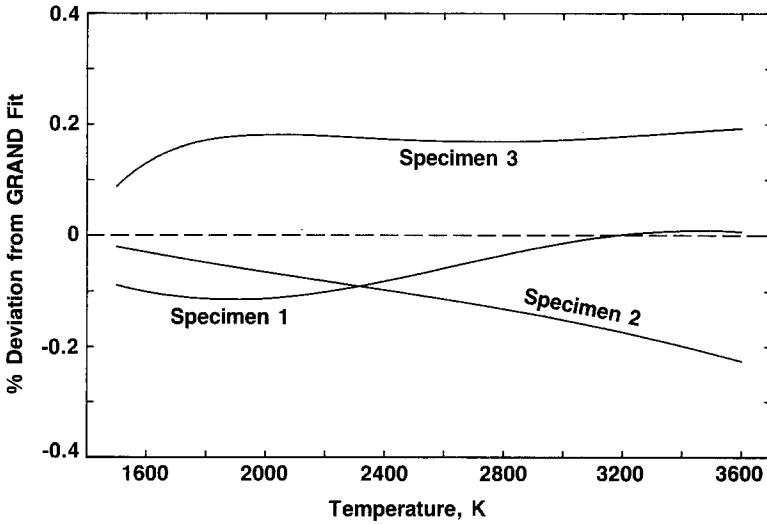


Fig. 3. Deviation of the smoothed thermal expansion results for the individual specimens from Eq. (2), which represents the least-squares fit to the combined data for three specimens.

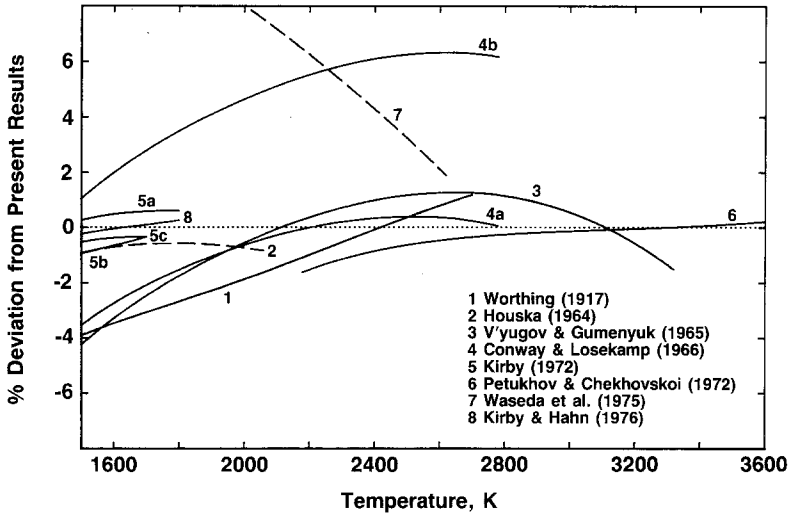


Fig. 4. Deviation of thermal expansion data for tungsten reported in the literature from the present results as expressed by Eq. (2). Curve numbers refer to the following: 1, Worthing [14]; 2, Houska [11]; 3, V'yugov and Gumenyuk [15]; 4, Conway and Losekamp [16]; 5, Kirby [9]; 6, Petukhov and Chekhovskoi [12]; 7, Waseda et al. [13]; 8, Kirby and Hahn [6]. The different measurement techniques are indicated as follows: X-ray diffractometry, ---; optical comparator methods, —.

temperature differed from 20°C. In addition, the temperature scales used in measurements prior to 1968 were converted to IPTS-68, yielding "corrections" to the reported expansion values of not more than 0.5%.

The most recent expansion data for tungsten appear to be the certificate values for the SRM 737 given by Kirby and Hahn [6] (curve 8). As may be seen in Fig. 4, the agreement between these data and the present results for the SRM 737 is better than 0.3% at overlapping temperatures (1500–1800 K). Earlier measurements at temperatures up to 1800 K by Kirby [9], on a sintered rod obtained from the OSRM (curve 5a) and on a sintered rod (curve 5b) and an arc cast rod (curve 5c) obtained from the AGARD project [10], yielded expansion data which lie within 1% of the present values. Similar agreement with the present work may be seen in the case of expansion data reported by Houska [11] (curve 2) for temperatures up to 2100 K as well as expansion values obtained by Petukhov and Chekhovskoi [12] (curve 6) at temperatures between 2400 and 3600 K.

The other reported values of thermal expansion exhibit different trends with changing temperature. The data reported by Waseda et al. [13] (curve 7) deviate from the present results by as much as 11% and appear to be in error. A common trend may be seen in the values obtained by Worthing [14] (curve 1), V'yugov and Gumenyuk [15] (curve 3), and Conway and Losekamp [16] (curve 4a, sintered rod; curve 4b, arc cast sheet), although the disagreement among these data is as large as 6%.

As mentioned earlier, tungsten SRM 737 is certified as a standard for thermal expansion measurements at temperatures up to 1800 K. The present work should enable the temperature range of this standard material to be extended up to 3600 K.

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REFERENCES

1. A. P. Müller and A. Cezairliyan, *Int. J. Thermophys.* **3**:259 (1982).
2. A. Cezairliyan, M. S. Morse, H. A. Berman, and C. W. Beckett, *J. Res. Natl. Bur. Stand. (U.S.)* **74A**:65 (1970).
3. A. Cezairliyan, *J. Res. Natl. Bur. Stand. (U.S.)* **75C**:7 (1971).
4. G. M. Foley, *Rev. Sci. Instrum.* **41**:827 (1970).

5. Office of Standard Reference Materials, *NBS Standard Reference Materials Catalog, 1988-89 edition*, NBS Special Publication 260 (U.S. Government Printing Office, Washington, D.C., 1988).
6. R. K. Kirby and T. A. Hahn, SRM 737 Certificate (Office of Standard Reference Materials, Natl. Bur. Stand. U.S.), 1976.
7. The International Committee for Weights and Measures, *Metrologia* **5**:35 (1969).
8. Y. S. Touloukian, R. K. Kirby, R. E. Taylor, and P. D. Desai, *Thermophysical Properties of Matter, Vol. 12, Thermal Expansion* (IFI/Plenum, New York, 1975).
9. R. K. Kirby, *High Temp. High Press.* **4**:459 (1972).
10. E. Fitzer and S. Weisenburger, *High Temp. High Press.* **4**:559 (1972).
11. C. R. Houska, *J. Phys. Chem. Solids* **25**:359 (1964).
12. V. A. Petukhov and V. Ya Chekhovskoi, *High Temp. High Press.* **4**:671 (1972).
13. Y. Waseda, K. Hirata, and M. Ohtani, *High Temp. High Press.* **7**:221 (1975).
14. A. G. Worthing, *Phys. Rev.* **10**:638 (1917).
15. P. N. Vyugov and V. S. Gumenyuk, *High Temp. (USSR)* **3**:879 (1965).
16. J. B. Conway and A. C. Losekamp, *Trans. Met. Soc. AIME* **236**:702 (1966).