

U.S. Department of Commerce
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National Bureau of Standards
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National Bureau of Standards

Certificate

Standard Reference Material 737

Tungsten - Thermal Expansion

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Thermal Expansion and Expansivity as a Function of Temperature (IPTS-68)

T	$\frac{L-L_{293}}{L_{293}}$	$\frac{1}{L_{293}} \frac{dL}{dT}$	T	$\frac{L-L_{293}}{L_{293}}$	$\frac{1}{L_{293}} \frac{dL}{dT}$
80 K	-814×10^{-6}	$2.30 \times 10^{-6}/K$	520 K	1040×10^{-6}	$4.70 \times 10^{-6}/K$
90	-790	2.61	560	1229	4.73
100	-762	2.88	600	1418	4.76
110	-732	3.11	650	1657	4.79
120	-700	3.30	700	1898	4.83
130	-666	3.46	750	2140	4.86
140	-631	3.59	800	2384	4.90
150	-595	3.71	850	2630	4.93
160	-557	3.81	900	2878	4.97
180	-479	3.97	950	3127	5.01
200	-398	4.10	1000	3378	5.04
220	-315	4.20	1100	3887	5.12
240	-231	4.27	1200	4404	5.21
270	101	4.36	1300	4930	5.31
293	0	4.42	1400	5467	5.43
320	120	4.47	1500	6016	5.56
360	300	4.53	1600	6578	5.70
400	483	4.59	1700	7157	5.87
440	667	4.63	1800	7754	6.07
480	853	4.66			

The above values of expansion and expansivity were calculated from equations based on a least squares analysis of the expansivity data from six specimens taken from various positions of the stock. A description of the experimental methods, fitting procedure, and estimate of uncertainties is given in this certificate.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. E. Michaelis.

Washington, D. C.
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J. Paul Cali, Chief
 Office of Standard Reference Materials

(over)

This SRM is available as a rod 6.4 mm (1/4 inch) in diameter and 51 mm (2 inches) in length. Inquiries for longer rods may be directed to the Office of Standard Reference Materials, National Bureau of Standards. This is a sintered powder metallurgical product made by compacting the starting powder at 15 tons/inch² and then heating to about 3400 K. The final diameter was obtained by swaging and grinding after 7 rolling operations. The density of this material is 19.23 g/cm³. Chemical analysis indicates a purity of 99.96 wt. % while the residual electrical resistance ratio, RRR = 60, indicates a purity of 99.98 wt. %.

This SRM should not be heated above 700 K in air. Vacuum or dry nonoxidizing atmospheres (helium, argon, hydrogen, etc.) should be used when heating it to higher temperatures. Cutting and shaping of tungsten is best accomplished by electrical discharge machining.

Procedure

In the temperature range below 1120 K the expansion measurements were made with a Fizeau interferometer [1]. The green spectral line of a mercury lamp was used to produce the interference fringes. Fringe motion was measured with a filar-micrometer eyepiece. Each test specimen was made by fastening three 1-cm rods of the SRM to a tungsten ring. Temperatures above 293 K were measured with a Pt vs. Pt-10% Rh thermocouple while those below were measured with a platinum resistance thermometer. The specimens were heated and cooled in a low pressure helium atmosphere and the expansion measured between equilibrium temperatures. With the uncertainties in temperature and fringe measurements, the expansivities were determined with an uncertainty of $\pm 0.03 \times 10^{-6}/\text{K}$.

In the temperature range above 970 K the expansion measurements were made with a twin-microscope technique [2]. Knife edges machined into the specimens defined their lengths (~10 cm). The changes in length on heating between equilibrium temperatures were measured with filar micrometer eyepieces. The temperatures were measured with W/Re and Pt-30% Rh/Pt-6% Rh thermocouples. The specimens were heated in a vacuum furnace. With the uncertainties in temperature and length measurements, the expansivities were determined with an uncertainty of $\pm 0.06 \times 10^{-6}/\text{K}$.

Because it was not always possible to project the measured values of expansion back to 293 K in an unambiguous manner, the calculated values of expansivity were used in the analysis of the data. Tests on the data from the six specimens indicated that no significant differences existed between them. All of the data were pooled and a polynomial of the form

$$\frac{1}{L_{293}} \frac{\Delta L}{\Delta T} \times 10^6 = a_0 + a_1 T + a_2 T^2 + a_3 T^3 + b_1 (T-\theta_1)^3 + \dots + b_4 (T-\theta_4)^3$$

where

$$\begin{aligned} (T-\theta_i)^3 &= (T-\theta_i)^3 \text{ if } T > \theta_i \\ (T-\theta_i)^3 &= 0 \text{ if } T \leq \theta_i \end{aligned}$$

was fit to them by the method of least squares using an Omnitab routine. The values of θ_1 , θ_2 , θ_3 , and θ_4 , were determined by the shape of the second derivative of the polynomial and a minimizing of the residual standard deviation. After the initial fit to the measured values was made, corrections for finite ΔT 's were applied to give the expansivity defined by

$$\frac{1}{L_{293}} \frac{dL}{dT} = \frac{1}{L_{293}} \frac{\Delta L}{\Delta T} - \frac{1}{24} \left(\frac{1}{L_{293}} \frac{\Delta L}{\Delta T} \right)'' (\Delta T)^2$$

where values of the second derivative,

$$\left(\frac{1}{L_{293}} \frac{\Delta L}{\Delta T} \right)''$$

were obtained from the initial fit. A final fit to the corrected data was then made which resulted in the following values:

$$\begin{aligned} a_0 &= -2.3835532 \\ a_1 &= +8.8699747 \times 10^{-2} \\ a_2 &= -4.411438 \times 10^{-4} \\ a_3 &= +8.0325992 \times 10^{-7} \\ b_1 &= -6.3651146 \times 10^{-7}, \theta_1 = 160 \text{ K} \\ b_2 &= -1.1648859 \times 10^{-7}, \theta_2 = 235 \text{ K} \\ b_3 &= -4.499535 \times 10^{-8}, \theta_3 = 330 \text{ K} \\ b_4 &= -4.8469136 \times 10^{-9}, \theta_4 = 550 \text{ K} \end{aligned}$$

This polynomial has been decomposed into the following third-order spline polynomials which are continuous at the knots, θ_i , as are their first and second derivatives:

$$\begin{aligned} 80 \text{ to } 160 \text{ K}, \frac{1}{L_{293}} \frac{dL}{dT} \times 10^6 &= -2.383 + 8.870 \times 10^{-2} T - 4.4114 \times 10^{-4} T^2 + 8.03 \times 10^{-7} T^3 \\ 160 \text{ to } 235 \text{ K}, \frac{1}{L_{293}} \frac{dL}{dT} \times 10^6 &= 0.225 + 3.982 \times 10^{-2} T - 1.3562 \times 10^{-4} T^2 + 1.667 \times 10^{-7} T^3 \\ 235 \text{ to } 330 \text{ K}, \frac{1}{L_{293}} \frac{dL}{dT} \times 10^6 &= 1.736 + 2.052 \times 10^{-2} T - 5.3494 \times 10^{-5} T^2 + 5.03 \times 10^{-8} T^3 \\ 330 \text{ to } 550 \text{ K}, \frac{1}{L_{293}} \frac{dL}{dT} \times 10^6 &= 3.353 + 5.816 \times 10^{-3} T - 8.9484 \times 10^{-6} T^2 + 5.26 \times 10^{-9} T^3 \\ 550 \text{ to } 1800 \text{ K}, \frac{1}{L_{293}} \frac{dL}{dT} \times 10^6 &= 4.1598 + 1.4179 \times 10^{-3} T - 9.5104 \times 10^{-7} T^2 + 4.176 \times 10^{-10} T^3. \end{aligned}$$

The standard deviation of this fit is 0.026×10^{-6} with 131 data points. These equations and their integrals were used to calculate the values listed in the table. A comparison of the experimental expansion data with values predicted from the equations gives a standard deviation of 5×10^{-6} in the range 80 to 1000 K and 13×10^{-6} in the range 1000 to 1800 K. All of the data for the expansivity were within three standard deviations of the predicted values.

After being heated for the first time to temperatures above 1300 K this SRM was observed to increase in length by about $40 \mu\text{m/m}$. Subsequent heating, even to 2300 K, did not cause any further growth.

It is planned to extend the temperature range covered by this certificate to about 3000 K.

Footnotes

- [1] Hahn, T. A., Thermal Expansion of Copper from 20 to 800 K - Standard Reference Material 736, J. Appl. Phys. **41**, 5096 (1970).
- [2] Rothrock, B. D. and Kirby, R. K., An Apparatus for Measuring Thermal Expansion at Elevated Temperatures, J. Res. NBS **71C**, 85 (1967).