

National Institute of Standards & Technology

Certificate

Standard Reference Material 736

Copper-Thermal Expansion

SRM 736 is in the form of a rod, 6.4 mm (1/4 inch) in diameter and is 51 mm (2 inches) long. It is intended for use primarily in calibrating dilatometers or interferometers used in the measurement of thermal expansion. The copper rods have been annealed at 811 K with no significant increase in grain size. The residual resistivity ratio, R_{273K}/R_{4k}, is 62.53 which would indicate a purity of 99.99 atomic percent with about 0.012 atomic percent effective (dissolved, ionized) impurities. This copper is similar to SRM 45d freezing point standard.

Thermal Expansion and Expansivity as a Function of Temperature

T	$\frac{L - L_{293}}{L_{293}}$	$\frac{1}{L_{293}} \frac{dL}{dT}$	T	$\frac{L-L_{293}}{L_{293}}$	$\frac{1}{L_{293}} \frac{dL}{dT}$
20K 30 40 50 60	-3250 x 10 ⁻⁶ -3245 -3229 -3198 -3151	0.27 x 10 ⁻⁶ /K 0.98 2.29 3.87 5.48	340 K 360 380 400 420	793 x 10 ⁻⁶ 1135 1481 1831 2183	17.07 x 10 ⁻⁶ /K 17.22 17.38 17.53 17.68
70	-3089	6.98	440	2538	17.82
80	-3012	8.30	460	2896	17.97
90	-2923	9.46	480	3256	18.11
100	-2823	10.46	500	3620	18.25
110	-2714	11.32	520	3986	18.39
120	-2597	12.05	540	4356	18.53
130	-2474	12.67	560	4728	18.67
140	-2344	13.20	580	5102	18.81
150	-2210	13.64	600	5480	18.95
160	-2072	14.01	620	5860	19.09
180	-1785	14.63	640	6244	19.24
200	-1487	15.14	660	6630	19.38
220	-1180	15.57	680	7019	19.53
240	- 865	15.94	700	7411	19.69
260	- 543	16.24	720	7807	19.84
280	- 215	16.50	740	8205	20.00
293	0	16.64	760	8607	20.16
300	117	16.71	780	9012	20.33
320	453	16.90	800	9420	20.51

The above values of expansion and expansivity were calculated from equations based on a least squares analysis of the expansivity data from five specimens taken from various positions of the stock. These values differ from those in the original certificate for two reasons: 1) new high temperature measurements have been made, and 2) an improved fitting routine has been used in the analysis of the data. A description of the experimental method, fitting procedure, and estimate of uncertainties is given in this certificate.

The technical measurements at NIST leading to certification were performed by R.K. Kirby and T.A. Hahn, both formerly of NIST.

Gaithersburg, MD 20899 October 7, 1990 (Revision of certificates dated 11-19-69 and 8-5-75) William P. Reed, Acting Chief Standard Reference Materials Program This certificate is a revision of the certificate dated August 5, 1975. The changes consist primarily of deletion from the certificate of additional lengths of the copper rods that were made available at its previous issue.

The technical and support aspects involved in the revision, update and issuance of this Standard Reference Material were coordinated through the Standard Reference Materials Program by J.C. Colbert.

The original coordination of certification efforts was performed by R.E. Michaelis.

Procedure

The apparatus³ used for the expansion measurements was a Fizeau interferometer with a 1-cm specimen length. Above room temperature, the measurements were made with the interferometer in a controlled atmosphere furnace using a Pt vs Pt-10%Rh thermocouple. Below room temperature, a cryostat operating with both liquid nitrogen and liquid helium was used with a platinum resistance thermometer. The green spectral line of a mercury light source 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 an OFHC copper ring to form a three point spacer for the interferometer flats. The expansion of each specimen was measured between equilibrium temperatures. With the uncertainties in temperature and fringe measurements, the expansivity was determined with an uncertainty of $\pm 0.03 \times 10^{-6} \text{K}^{-1}$. As a result of measurements that were made on this SRM at the Oak Ridge National Laboratory by Kollie and coworkers⁴ it has been determined that at temperatures above 600 K mechanical creep had occurred during our initial measurements and the expansion values that were given in the original certificate were too low by about 0.4 percent at 800 K. The values given in this revised certificate at temperatures above 600 K are the result of new measurements that were made with an increased area of contact between the specimens and the interferometer flats. When the pressure at the contact was less than 3 x 10⁵ Pa (40 psi) there was no evidence of mechanical creep as determined by taking measurements on both heating and cooling.

Since 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 five specimens indicated that no significant differences existed between them. All of the data were pooled and, as suggested by J.R. Rosenblatt of the Center for Computing and Applied Mathemantics at NIST, a polynomial of the form

$$\frac{1}{L_{293}} \frac{\Delta L}{\Delta T} \times 10^6 = a_0 + a_1 T + a_2 T^2 + a_3 T^3 + b_1 (T - \theta_1)_+^3 + \ldots + b_6 (T - \theta_6)_+^3,$$

where

$$(T-\theta_t)_+^3 = (T-\theta_t)^3 \text{ if } T > \theta_t,$$

$$(T-\theta_t)_+^3 = 0 \text{ if } T \le \theta_t,$$

was fit to them by the method of least squares using an Omnitab routine. The values of θ_1 , θ_2and θ_6 were determined by the shape of the second derivative of the polynominal and minimizing the residual standard deviation. This procedure is considerably different from that used in the original certificate dated 11-19-69 and results in a better fit to the data at all temperatures. After the initial fit to the measured values was made, corrections for finite ΔT 's were applied to give the instantaneous coefficients of expansion defined by

$$\frac{1}{L_{293}} \frac{dL}{dT} = \frac{1}{L_{293}} \frac{\Delta L}{\Delta L} - \frac{1}{24} \left(\frac{1}{L_{293}} \frac{\Delta L}{\Delta T} \right)^{"} (\Delta T)^{2}$$

where values of the second derivatives, $\left(\frac{1}{L_{293}}\frac{\Delta L}{\Delta T}\right)^{\prime\prime}$, were obtained from the initial fit. A final fit to the corrected data was then made which resulted in the following values:

$$a_0 = 3.6766548 \times 10^{-2}$$

 $a_1 = +9.4571214 \times 10^{-3}$
 $a_2 = -7.6957895 \times 10^{-4}$

$$a_3 = +5.3009542 \times 10^{-5}$$
 $b_1 = 1.2206897 \times 10^{-4}, \theta_1 = 28 \text{ K}$
 $b_2 = +3.9954573 \times 10^{-5}, \theta_2 = 41 \text{ K}$
 $b_3 = +3.0265158 \times 10^{-5}, \theta_3 = 62.5 \text{ K}$
 $b_4 = +1.4101666 \times 10^{-6}, \theta_4 = 90 \text{ K}$
 $b_5 = -2.3610759 \times 10^{-6}, \theta_5 = 178 \text{ K}$
 $b_6 = -2.0050557 \times 10^{-7}, \theta_6 = 350 \text{ K}$

This polynominal 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:

20 to 28 K,
$$\frac{1}{L_{293}} \frac{dL}{dT} \times 10^{6} = -0.037 + 9.46 \times 10^{-3} \text{T}$$

$$-7.70 \times 10^{-4} \text{T}^{2} + 5.301 \times 10^{-5} \text{T}^{3}$$
28 to 41 K,
$$\frac{1}{L_{293}} \frac{dL}{dT} \times 10^{6} = 2.643 - 2.7765 \times 10^{-1} \text{T}$$

$$+ 9.4842 \times 10^{-3} \text{T}^{2} - 6.906 \times 10^{-5} \text{T}^{3}$$
41 to 62.5 K,
$$\frac{1}{L_{293}} \frac{dL}{dT} \times 10^{6} = -0.111 - 7.616 \times 10^{-2} \text{T}$$

$$+ 4.5698 \times 10^{-3} \text{T}^{2} - 2.9105 \times 10^{-5} \text{T}^{3}$$
62.5 to 90 K,
$$\frac{1}{L_{293}} \frac{dL}{dT} \times 10^{6} = -7.500 + 2.7851 \times 10^{-1} \text{T}$$

$$-1.1049 \times 10^{-3} \text{T}^{2} + 1.160 \times 10^{-6} \text{T}^{3}$$
90 to 178 K,
$$\frac{1}{L_{293}} \frac{dL}{dT} \times 10^{6} = -8.528 + 3.12779 \times 10^{-1} \text{T}$$

$$-1.48566 \times 10^{-3} \text{T}^{2} + 2.5705 \times 10^{-6} \text{T}^{3}$$
178 to 350 K,
$$\frac{1}{L_{293}} \frac{dL}{dT} \times 10^{6} = 4.788 + 8.8354 \times 10^{-2} \text{T}$$

$$-2.24845 \times 10^{-4} \text{T}^{2} + 2.0939 \times 10^{-7} \text{T}^{3}$$
350 to 800 K,
$$\frac{1}{L_{293}} \frac{dL}{dT} \times 10^{6} = 13.385 + 1.4668 \times 10^{-2} \text{T}$$

$$-1.4314 \times 10^{-5} \text{T}^{2} + 8.883 \times 10^{-9} \text{T}^{3}$$

The standard deviation of this fit is 0.059×10^{-6} with 141 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 6×10^{-6} . All of the data for both the expansion and expansivity were within two standard deviations of the values predicted by the equations.

Notes

- 1. Vacuum annealing (G.E. Hicho).
- 2. Residual resistivity ratios (V.A. Deason and R.L. Powell).
- 3. Hahn, T.A., Thermal Expansion of Copper from 20 to 800 K Standard Reference Material 736, J. Appl. Phys. 41, 5096 (1970).
- 4. Kollie, T.G., McElroy, D.L. Hutton, J.T., and Ewing, W.M., A Computer Operated Fused Quartz Differential Dilatometer, AIP Conf. Proc., No. 17 Thermal Expansion, p. 129, AIP, New York (1974).