U.S. Department of Commerce
Juanita M. Kreps
Secretary
National Bureau of Standards
Ambler, Acting Director

National Bureau of Standards

Certificate

Standard Reference Material 732

Single Crystal Sapphire - Thermal Expansion

T. A. Hahn

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

T	$\frac{L-L_{293}}{L_{293}}$	$\alpha = \frac{1}{L_{293}} \frac{dL}{dT}$	T	$\frac{L-L_{293}}{L_{293}}$	$\alpha = \frac{1}{L_{293}} \frac{dL}{dT}$
293 K	0×10^{-6}	5.38×10^{-6} K^{-1}	900 K	4608×10^{-6}	8.79×10^{-6} K^{-1}
300	38	5.48	1000	5500	9.05
350	328	6.12	1100	6417	9.29
400	647	6.61	1200	7357	9.52
450	987	7.00	1300	8320	9.74
500	1346	7.32	1400	9306	9.97
550	1719	7.59	1500	10314	10.20
600	2104	7.82	1600	11345	10.42
650	2500	8.02	1700	12399	10.66
700	2906	8.20	1800	13477	10.89
750	3320	8.37	1900	14578	11.14
800	3743	8.52	2000	15704	11.38

This single-crystal sapphire was grown from the melt, cut into 20 cm lengths, and centerless ground to 6.4 mm diameter. Back reflection Laue photographs of each rod gave an average orientation of the c-axis of the crystal relative to the rod axis equal to 59°. Several rods were checked for variations in orientation along their lengths, but none were found. Three specimens that were thought to have the greatest difference in orientation, ~1°, were selected for the thermal expansion measurements. The expansion of these three specimens from room temperature to 1000 K, however, did not differ by more than $4 \cdot 10^{-6}$. This is well within the precision of the measurement system, and indicates that no significant difference exists between any of the rods. The tabulated values were calculated from an equation whose parameters were determined by a least squares analysis of the pooled expansion data. Three-point interpolation of the tabulated data may be used to obtain values not listed. Descriptions of the experimental methods, the equation used to fit the data, and estimates of uncertainties are given in this certificate and in reference [1].

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

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J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

This SRM is available in rods 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.

Procedure

In the temperature range below 1000 K, expansion measurements were made with a Fizeau interferometer [2]. Interference fringes were formed between fused-silica optical flats using the green spectral line of a mercury lamp. Fringe motion was measured with a filar-micrometer eyepiece. Each test specimen was made by grinding three 1-cm rods to fit a sample holder to form a three-point spacer for the interferometer flats. Temperatures were measured using a calibrated Pt vs. Pt-10% Rh thermocouple fastened to the sample holder. The specimens were heated in a low-pressure helium atmosphere and the expansion measured between equilibrium temperatures. Measurements on a variety of different materials with this system generally give a reproducibility on the order of $\pm 5 \times 10^{-6}$ in $\Delta L/L$ values.

In the temperature range above 1000 K, expansion measurements on one specimen were made with a twin-microscope technique [3] using a tungsten-mesh vacuum furnace. Knife edges machined into the specimen 10 cm apart defined its length. The change in length on heating between equilibrium temperatures was measured with filar-micrometer eyepieces. The temperatures were measured with W vs Re and Pt-30% Rh vs Pt-6% Rh thermocouples. Deviations in the expansion measurements result in an expected reproducibility of $\pm 15 \times 10^{-6}$ in $\Delta L/L$ values.

The tabulated values given in this certificate were calculated from a variation of Gruneisen's equation given by

$$\frac{L - L_{293}}{L_{293}} = \frac{E(\frac{\theta}{T})}{3a\left[Q_o - kE(\frac{\theta}{T})\right]} + \frac{1-a}{a}$$

where

$$E(\frac{\theta}{T}) = \frac{3nR\theta}{4}\left[2\,(c^{\frac{\theta}{T}}-1)^{-1} + (c^{\frac{\theta}{2T}}-1)^{-1}\right]$$

R is the gas constant ($8.3143\ J\cdot K^{-1}\cdot mol^{-1}$), n the number of atoms per molecule(5), and $a = L_{293}/L_0$. The values θ=927 K, $Q_0 = 5\times10^6\ J\cdot mol^{-1}$, k = 3.6, and a = 1.000674 were obtained by successive least squares approximations described by Wachtman, et al. [4] to minimize the standard deviation of the fit. The residual standard deviation of the fit is 16×10^{-6} using all 113 data points. The standard deviation of the data below 1000 K is 5×10^{-6} while above 1000 K it is 25×10^{-6}. All of the data are within approximately two standard deviations in the respective temperature intervals.

Expansivities were calculated from the derivative of the expansion equation:

$$\alpha = \frac{1}{L_{293}}\frac{dL}{dT} = \frac{Q_o C_v(\frac{\theta}{T})}{3a\left[Q_o - kE(\frac{\theta}{T})\right]^2}$$

where

$$C_v(\frac{\theta}{T}) = \frac{3nR}{32}(\frac{\theta}{T})^2\left[4\mathrm{csch}^2\left(\frac{\theta}{2T}\right) + \mathrm{csch}^2\left(\frac{\theta}{4T}\right)\right]$$

Values of expansivity calculated from this equation were compared to values calculated from the experimental data resulting in a standard deviation of $0.04 \times 10^{-6} K^{-1}$ below 1000 K and $0.18 \times 10^{-6} K^{-1}$ above 1000 K. Examination of the deviations of the data from the above equations for both the expansion and expansivity did not indicate any systematic variations.

References

1. T. A. Hahn, Proceedings of the 1977 International Symposium on Thermal Expansion (in press).
2. T. A. Hahn, J. Appl. Phy. **41,** 5096 (1970).
3. B. D. Rothrock and R. K. Kirby, J. Res. NBS **71C,**85 (1967).
4. J. B. Wachtman, Jr., T. G. Scuderi, and G. W. Cleek, J. Am. Ceram. Soc. **45,** 319 (1962).