Intercomparison of Interferometric Dilatometers at NRLM and NPL

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M. Okaji and K. P. Birch

Abstract. Interferometric dilatometers at the National Research Laboratory of Metrology (NRLM) in Japan and the National Physical Laboratory (NPL) in England been intercompared by measuring the thermal expansion coefficients of pure polycrystalline silicon, a low expansion ceramic (Miraclon) and fused silica (SRM 739). The dilatometers, two from each laboratory, were intercompared over their respective operating temperature ranges of 250 to 375 K and 290 to 800 K. The results presented in this paper show that the agreement for each material is within the combined uncertainties in the measurement of each thermal coefficient of expansion.

1. Introduction

The determination of the linear thermal expansion coefficient of materials is one of the most fundamental requirements of those industries that use accurate length and temperature measurement. Length measurement is normally realised by mechanical, capacitive, or, for high accuracy, by interferometric means which allow nanometric resolutions to be achieved. For a number of years NRLM and NPL have applied these techniques to realise interferometric dilatometers and have used them to determine expansion data for a wide range of materials.

Substantial industrial interest has recently concentrated on the development of ultra-low expansion ceramics with thermal expansion coefficients of, in some cases, $0{,}01 \times 10^{-6}\,K^{-1}$. This development has produced a corresponding demand to characterise the thermal expansion performance of these materials. To ensure adequate measurement uncertainties for this purpose at NRLM and NPL an intercomparison was made using three materials with different expansion coefficients. The measurements were made on the same samples using two dilatometers from each laboratory which operated over the different temperature ranges 250 to 375 K and 290 to 800 K. Curve fits obtained from the thermal expansion data derived from these measurements were compared for each material.

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2. Dilatometer Descriptions

2.1 The first NRLM dilatometer (NRLM/1)

The first NRLM dilatometer [1, 2] consists of a double path heterodyne interferometer and a focused image furnace with a quartz vacuum chamber. The design of the interferometer is shown in Fig. 1 and consists of two polarising prisms, a cube-corner reflector, a quarter-wave plate, and a half-wave plate. Each optical element is mounted on a fine adjustable stage which allows easy optical alignment. This system combines an alignment versatility with a short term stability which is adequate for this type of dilatometer. The length change of the specimen is monitored by comparing the phase of the heterodyne signal from the interferometer with that from a reference signal. A commercial Zeeman-beat stabilised laser (λ = 633 nm) is used as the wavelength source. The specimen is wrung on a fused silica or silicon baseplate which is enclosed in an evacuated vacuum chamber. The chamber can be heated or cooled at a rate of 4 K/min from ambient to 1 000 K. Calibrated thermocouples J (type R) are attached to the specimen for temperature measurement.

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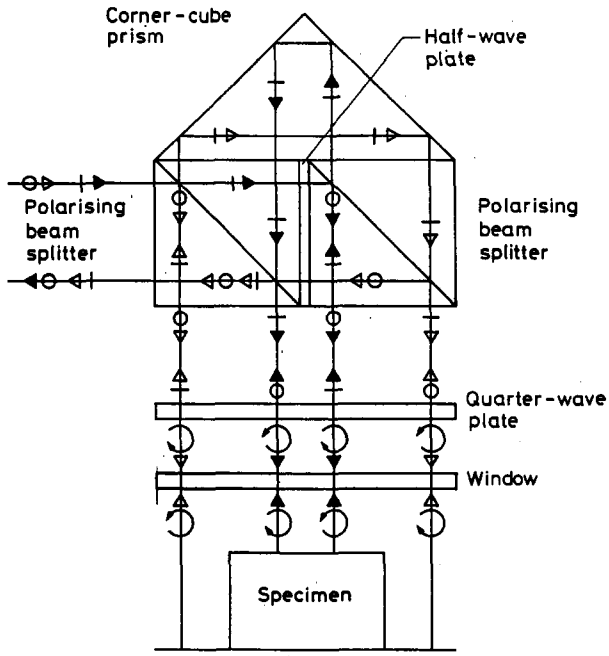

Figure 1. The NRLM interferometer configuration.

2.2 The second NRLM dilatometer (NRLM/2)

The second NRLM dilatometer [3] uses the same interferometer system as NRLM/1 with an evacuated aluminium specimen chamber. The chamber incorporates a jacket that can be heated or cooled by air for temperature cycling the specimen. The dilatometer, which, when compared with NRLM/1, has both an improved temperature gradient and an improved temperature measuring system uses calibrated thermocouples (type T) for measuring the temperature of the specimen.

2.3 The first NPL dilatometer (NPL/1)

The first NPL dilatometer [4] uses a commercial wavelength-stabilised He-Ne laser (λ = 633 nm) to illuminate the interferometer as shown in Fig. 2. The double-path interferometer consists of a single, cemented unit which comprises three beam-splitters and a cube-corner reflector. Electrical outputs produced by detectors placed in the two interferometer output beams are recorded continuously on a chart recorder. The recorded traces vary sinusoidally by 360° as the interferometer path length changes by $\lambda/4$ and hence provide a permanent record of the length changes induced in the sample.

The specimen to be measured is wrung on a CERVIT baseplate which is enclosed in an evacuable chamber that can be heated from ambient to 800 K. Calibrated thermocouples (type K) are attached to the specimen for temperature measurement. The performance of this dilatometer had been degraded by regular use for several years and this showed as scatter in some of the results obtained. This was not the case in the results obtained by Bennett [4] when the dilatometer was first commissioned.

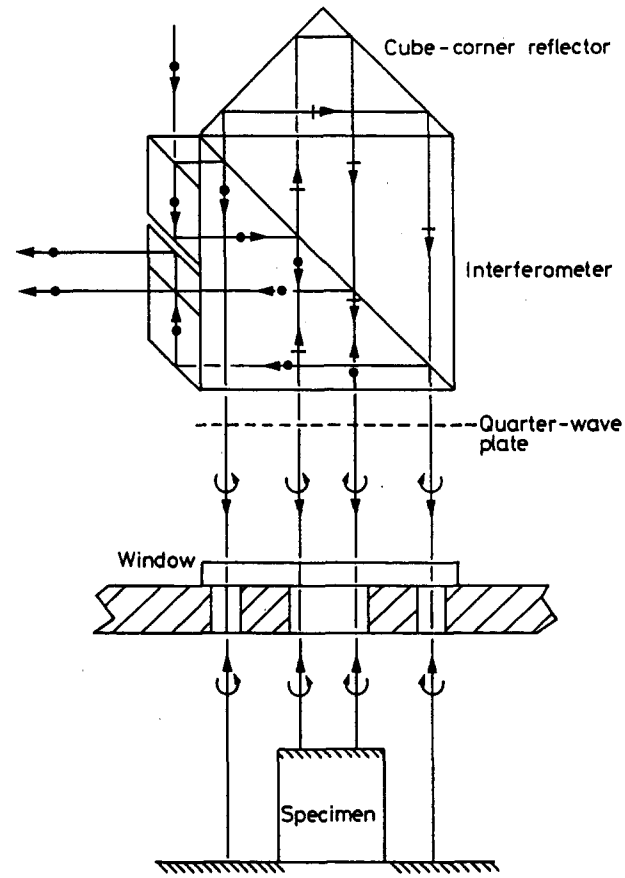

Figure 2. The NPL interferometer configuration.

2.4 The second NPL dilatometer (NPL/2)

The second NPL dilatometer [5] is similar to those previously described where the specimen is contacted on a Zerodur baseplate located in a vacuum chamber. The chamber, which can be temperature cycled from 245 to 375 K, incorporates calibrated thermocouples (type T) which are attached to the specimen to provide temperature measurement. The system uses the same type of interferometer as that in the NRLM/1 dilatometer except that the support arrangement is different [6]. This dilatometer, which has a higher accuracy than NPL/1, operates under computer control and thermal expansion data is processed automatically.

3. Samples and their Preparation

The dilatometers all measure the mean linear thermal coefficient of expansion of a material, $\bar{\alpha}$, which is given by

$$\bar{\alpha} = \frac{\Delta \ell}{\ell \Delta T}, \quad (1)$$

where $\Delta \ell$ is the change in the length of the material induced by a change in the material temperature ΔT at a particular temperature T, and ℓ is the length of the material. For materials with a high thermal expansion coefficient (e.g. metals) the main source of uncertainty is the measurement of ΔT whilst for low expansion materials the main source is the measurement of $\Delta \ell$.

Interferometeric dilatometers are normally used to measure low expansion materials because sub-nanometric sample length changes can be resolved. Therefore stable materials with a range of low thermal coefficients of expansion were chosen in order to intercompare the dilatometers.

These materials were (in decreasing order of thermal coefficient of expansion) polycrystalline silicon, a low expansion ceramic (Miraclon) and fused silica (in the form of an NIST standard reference material SRM 739). There are materials with lower coefficients of expansion than fused silica (e.g. Zerodur), but these were not used because of concerns over their dimensional stability [7] with respect to crystalline changes and stress relief. Instead an 8 mm length of fused silica was chosen to produce the same order of length change as that found in a typical 80 mm long sample of ultra-low expansion ceramic. The lengths of the silicon and Miraclon specimens were 35,6 mm and 22,6 mm respectively.

Two surfaces of each of the specimens were polished flat to 0,05 μm and mutually parallel to $2{,}5 \times 10^{-5}$ rad. One polished face of each piece of fused silica and Miraclon was gold coated in order to enhance the reflectivity of the surface. The distance between the two surfaces of each specimen was determined using a calibrated comparator which measured the difference in length between the specimen and a material standard of known length.

4. Measurement Results

4.1. Procedure

Measurements were made by temperature cycling each specimen over the operating temperature range of each dilatometer. Each cycle of measurements involved wringing individual samples onto the dilatometer baseplate following which the mean linear thermal coefficient of expansion for the material was determined at equal temperature intervals. These measurement cycles were repeated several times and the temperature intervals used, together with the number of determinations made, are given below:

Dilatometer	Temperature Interval/K	Number of determinations		
		Silicon	Miraclon	Fused silica
NRLM 1	50/30*	37	63*	36
NRLM 2	30	20	19	20
NPL 1	50	30	30	42
NPL 2	30	16	14	16

Individual sets of measurement data, i.e. for one material and one dilatometer, were used to produce polynomial expressions for the instantaneous coefficient of thermal expansion (α). The technique used was described by Bennett [4] who showed that if the mean linear thermal coefficient of expansion is determined over a number of equal temperature intervals (ΔT), the values obtained may be used to derive a polynomial expression for $\bar{\alpha}$ in terms of the mean temperature (T_m) of the form

$$\bar{\alpha} = A + BT_m + CT_m^2 + DT_m^3 + ET_m^4 + FT_m^5 + \ldots \quad (2)$$

This may be compared with the instantaneous thermal expansion polynomial which may be written as

$$\alpha = a + bT + cT^2 + dT^3 + eT^4 + fT^5 + \ldots \quad (3)$$

Since $\Delta T = T_2 - T_1$ ($T_1 < T_2$) and $T_m = (T_2 + T_1)/2$ are readily measured, comparison of equations (2) and (3) enables the polynomial coefficients in (3) to be obtained. Bennett showed how the coefficients for a third order fit could be obtained. These were found to be adequate for the NRLM/2 and NPL/2 results, however, for the NRLM/1 and NPL/1 results, fifth order fits were required. The coefficients in this case can be shown to be:

$$a = A - C\Delta T^2/12 + 7E\Delta T^4/240 \quad (4)$$
$$b = B - D\Delta T^2/4 + 7F\Delta T^4/48 \quad (5)$$
$$c = C - E\Delta T^2/2 \quad (6)$$
$$d = D - 5F\Delta T^2/6 \quad (7)$$
$$e = E \quad (8)$$
$$f = F \quad (9)$$

Curves were plotted of each derived polynomial expression and these are shown for individual materials in Figs. 3(*a*), 4(*a*) and 5(*a*). Figs. 3(*b*), 4(*b*) and 5(*b*) show the curves replotted as differences from the average value of the NRLM/1 and NPL/1 polynomial expression. This also shows the residual variations at the 3σ level.

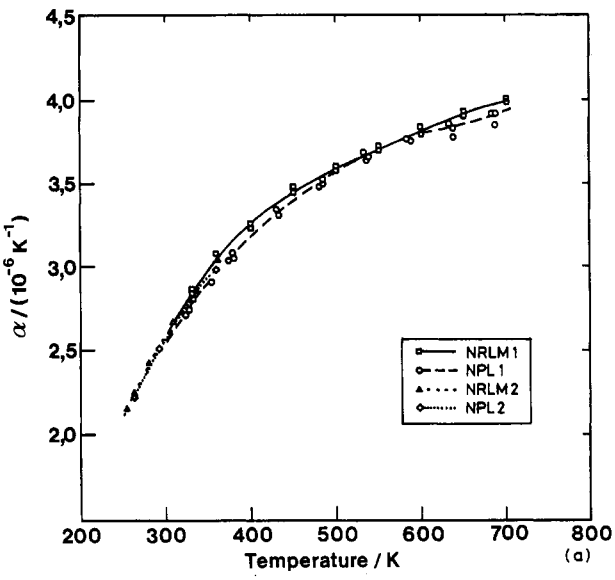

Figure 3a. Variation of the thermal expansion coefficient of silicon with temperature.

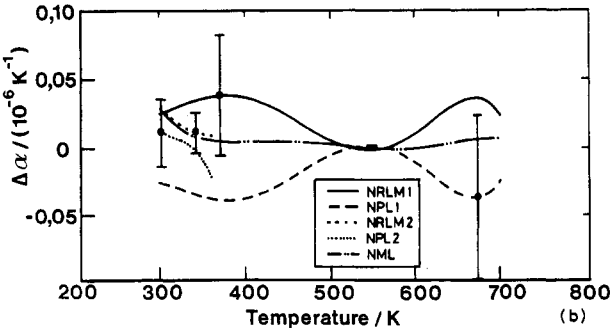

Figure 3b. Differences of individual measurements on silicon from the average of the NRLM/1 and NPL/1 polynomial expressions.

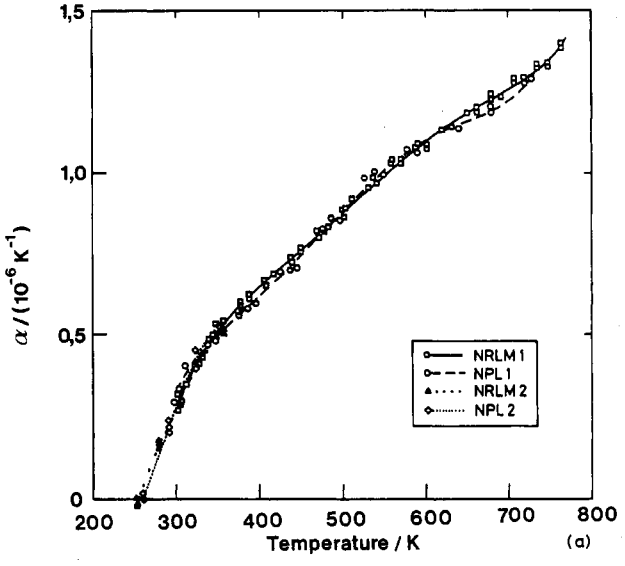

Figure 4a. Variation of the thermal expansion coefficient of Miraclon with temperature.

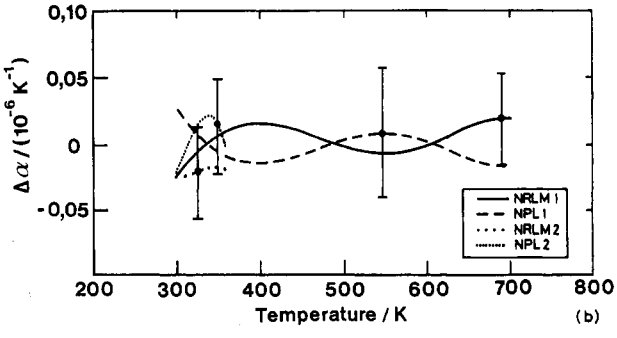

Figure 4b. Differences of individual measurements on Miraclon from the average of the NRLM/1 and NPL/1 polynomial expressions.

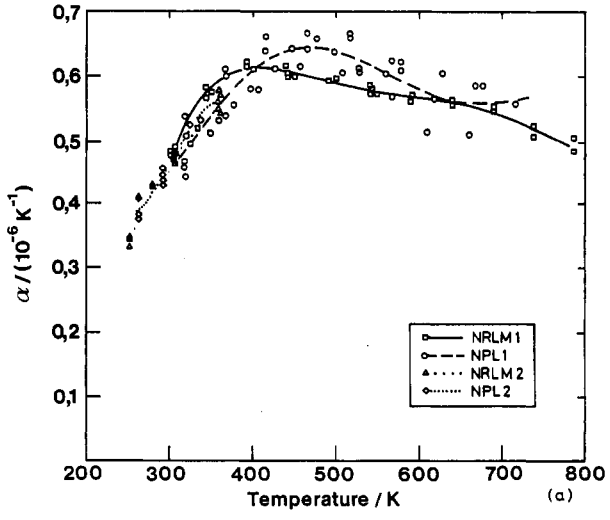

Figure 5a. Variation of the thermal expansion coefficient of fused silica with temperature.

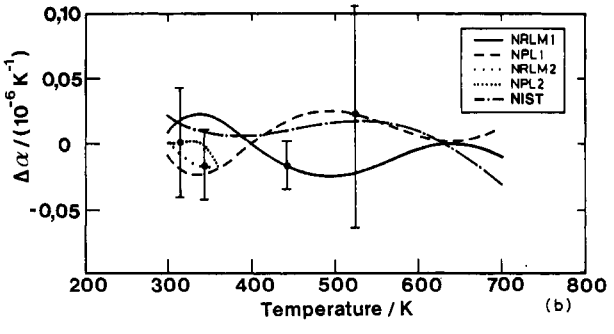

Figure 5b. Differences of individual measurements on fused silica from the average NRLM/1 and NPL/1 polynomial expressions.

Table 1. Estimated uncertainties (3σ).

Source of uncertainty	NRLM 1	NRLM 2	NPL 1	NPL 2
1. *Path length measurement*				
Laser wavelength stability	$1{,}5 \times 10^{-9}$	$1{,}5 \times 10^{-9}$	3×10^{-9}	$1{,}5 \times 10^{-9}$
Long term stability/nm	3	3	3	6
Zero drift reproducibility/nm	1,5	1,5	2	1,6
2. *Temperature measurement*				
Temperature determination/K	0,03	0,01	0,15	0,05
Temperature distribution/K	0,25	0,06	1,0	0,3
Thermocouple calibration/K	0,3	0,03	0,45	0,15

4.2. Uncertainties in the Measurements

Estimates of the uncertainties are summarised by dilatometer in Table 1 which also lists the contribution by source. In order to estimate the total uncertainty that these contributions make in the determination of $\bar{\alpha}$ for each material, the conditions shown in Table 2 were used to calculate the uncertainties for the specimens listed in Table 3(*a*).

Table 2. Values of ΔT/K, $\alpha/10^{-6}\,K^{-1}$ and ℓ_0/mm used in estimating the uncertainty in α.

Specimen	Variable	NRLM 1	NRLM 2	NPL 1	NPL 2
Silicon	ΔT	50	25	50	30
	α	3,3	2,65	3,3	2,65
	ℓ_0	35,58	35,58	35,58	35,58
Miraclon	ΔT	30	25	50	30
	α	0,9	0,3	0,9	0,3
	ℓ_0	22,63	22,63	22,63	22,63
Fused silica	ΔT	50	25	50	30
	α	0,55	0,5	0,55	0,5
	ℓ_0	10,0	10,0	8,2	8,2

Table 3 (a). Estimated uncertainties expressed as $3\sigma/10^{-6}\,K^{-1}$.

Specimen	NRLM 1	NRLM 2	NPL 1	NPL 2
Silicon	0,037	0,012	0,073	0,030
Miraclon	0,019	0,011	0,021	0,010
Fused silica	0,014	0,026	0,026	0,022

Table 3 (b). Residual variations expressed as $3\sigma/10^{-6}\,K^{-1}$.

Specimen	NRLM 1	NRLM 2	NPL 1	NPL 2
Silicon	0,045	0,016	0,063	0,024
Miraclon	0,036	0,036	0,050	0,039
Fused silica	0,019	0,028	0,087	0,042

From the polynomial expressions discussed above, the residual variations from each expression, at the 3σ level, were also determined. These are summarised in Table 3(*b*).

4.3. Silicon

The results for silicon are shown in Figs. 3 (*a*) and 3 (*b*) which show the four curves derived from the NRLM and NPL data. Included for comparison is the curve obtained for pure silicon from NML [8], which shows good agreement with the NRLM and NPL results. The lower temperature (260 to 360 K) and the higher temperature (300 to 700 K) curve fits show deviations ranging, respectively, up to $0{,}03 \times 10^{-6}\,K^{-1}$ and $0{,}07 \times 10^{-6}\,K^{-1}$. The differences between the curves are all within the range of uncertainties listed in Table 3(*a*) however, over both temperature ranges, the NRLM curve was always higher than that of the NPL. Over the common temperature interval for all dilatometers (300 K to 360 K) the curve fits agree to within $\pm\ 0{,}04 \times 10^{-6}\,K^{-1}$. Since silicon has a relatively high thermal coefficient of expansion, these systematic differences imply that there are temperature measuring anomalies in the low and high temperature dilatometers of about 0,2 K and 0,5 K respectively.

4.4. Miraclon

The curves derived for Miraclon are shown in Figs. 4 (*a*) and 4 (*b*). The lower and the higher temperature curves show deviations ranging, respectively, up to $0{,}04 \times 10^{-6}\,K^{-1}$ and $0{,}03 \times 10^{-6}\,K^{-1}$. There were no significant systematic differences between any of the curves and over the common temperature intervals, for all the dilatometers, the differences in curve fits ranged up to $0{,}05 \times 10^{-6}\,K^{-1}$. The differences between the higher temperature curves were within the range of uncertainties given in Table 3(*a*).

4.5. *Fused silica (SRM 739)*

As discussed in Sect. 3, 8 mm lengths of fused silica were chosen to assess the limiting uncertainty in the thermal expansion measurement of low expansion materials since an expansion coefficient variation of $0{,}01 \times 10^{-6}\,K^{-1}$ in this length of material corresponds to only a 3 nm change in length of the material for $\Delta T = 30$ K. The curves derived for fused silica are shown in Figs. 5 (*a*) and 5 (*b*). Included for comparison is the corresponding curve obtained by the NIST [9] which is in good agreement with the NRLM and NPL results. The low and high temperature curve fits show deviations ranging, respectively, up to $0{,}02 \times 10^{-6}\,K^{-1}$ and $0{,}04 \times 10^{-6}\,K^{-1}$. These are all within the combined uncertainties listed in Table 3(*a*) for this material. Over the common temperature range for all the dilatometers the curve fits agreed to within approximately $\pm\ 0{,}02 \times 10^{-6}\,K^{-1}$. As expected the results show more scatter in the data obtained from the NPL/1 dilatometer due to the relatively low resolution method of measuring sample length changes. A further contribution arises from drift in the detecting electronics of the interferometer.

4.6. *Discussion*

Tables 3 (*a*) and 3 (*b*) show that the estimated uncertainties for each material were generally lower than the residual variations determined from the polynomial fits. These differences imply that the individual uncertainties listed in Table 1 are too low. Of particular significance are the large differences for Miraclon: this is discussed further below.

The results from the NRLM 1 and NPL 1 dilatometers were found to be within the uncertainties estimated in Table 3(*a*) for all three materials. The systematic differences between the curves for silicon are significant and, since the same trend did not appear in the Miraclon curves, this implies that a temperature measuring anomaly existed only when the silicon measurements were made. The measurement results for fused silica are known to be limited by the low resolution method of measuring sample length changes in NPL 1.

Agreement of the measurements from the NRLM 2 and NPL 2 dilatometers was within the uncertainties given in Table 3(*a*) for silicon and fused silica, however, the curves obtained from the NPL 2 and NRLM 2 data for Miraclon showed significantly larger variations than expected from the calculated uncertainties. There are also large differences between the uncertainties estimated for Miraclon and the corresponding residual variations given in Tables 3 (*a*) and 3 (*b*) for the NRLM 2 and NPL 2 results. A similar trend may be seen in the NRLM 1 and NPL 1 data for Miraclon given in Tables 3 (*a*) and 3 (*b*).

It was concluded therefore that an additional source of uncertainty may lie in the relative dimensions of the Miraclon specimen. This specimen has a square cross section of 5 mm × 5 mm and a length of 22 mm, which gives it a relatively small area of contact on the dilatometer baseplates and may have lead to sample movement during measurement. A further source of variation may be the length stability of the material.

5. Conclusion

An intercomparison of the measurement of linear thermal expansion coefficients for several materials has been carried out by using four different types of interferometric dilatometer. The measurements for silicon, Miraclon, and fused silica (SRM 739) show good agreement, and confirm that measurement uncertainties of between $0{,}02 \times 10^{-6}K^{-1}$ and $0{,}09 \times 10^{-6}K^{-1}$, at the 3σ level, may be obtained with the dilatometers developed at NRLM and NPL. The intercomparison of a number of different instruments has enabled several sources of measurement uncertainty to be identified which, following their correction, will enable an enhanced measurement performance to be obtained.

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