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A practical measurement system for the accurate determination of linear thermal expansion coefficients

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Abstract. An accurate and versatile system for the measurement of linear thermal expansion coefficient is described. The system consists of a double-path optical heterodyne laser interferometer and a parallel spring strip supporting system which provides both versatility and accuracy of measurement.

The interferometer has a resolution of 1 nm and the parallel strip supporting system is used to accommodate various shapes and dimensions of specimens to be measured.

From the measurements of twelve industrial materials including two standard reference materials at room temperatures, the measurement accuracy is estimated to be within 3 to $7 \times 10^{-8}\,K^{-1}$ on materials having corresponding expansion coefficients of between -1 and $23 \times 10^{-6}\,K^{-1}$.

1. Introduction

Generally, when an interferometric dilatometer is used, specimens are required to be polished flat and parallel to very fine tolerances in order to obtain high visibility of optical fringes and to provide stable optical contact between the specimen and mirror. The flatness and parallelism of the specimen ends are usually required to be within 0,05 μm and 5 seconds of arc, respectively.

At NRLM, we have developed several types of dilatometers with supporting systems of specimens in order to obtain more efficient measuring methods and shorten the measuring time. Good results have been obtained (Okaji *et al* 1981a, b). However, the accuracy of the measurement was limited by an optical misalignment of the interferometer caused by thermal deformations on the measuring system. We have therefore designed and developed multi-fold path interferometers which compensate the optical misalignment by the use of rectangular prisms, and have very high sensitivity in length measurement (Okaji and Imai 1983).

At present, the two-fold path interferometer and parallel spring specimen holder provide a method of achieving both accurate and versatile linear thermal expansion measurements. The twelve various kinds of specimens including two standard reference materials (copper SRM 736 and tungsten SRM 737), supplied by NBS (USA), have been measured at near room temperatures (15 ~ 25 °C) to confirm the performance of this dilatometric system.

2. Apparatus

2.1. Interferometer

A two-fold path interferometer has been developed for measuring linear thermal expansion coefficients – it is shown schematically in figure 1. This interferometer involves a self-compensation of optical misalignment and a wide tolerance of nonparallelism between two mirrors but it has a simpler construction and fewer optical elements than an earlier version (Okaji and Imai 1983). In addition, it has a simpler configuration than similar double-path interferometers previously reported by Bennett (1977) and Tanaka (1983). The interferometer is insensitive to atmosphere refractive index changes and mechanical drift owing to its symmetrical form and common path configuration.

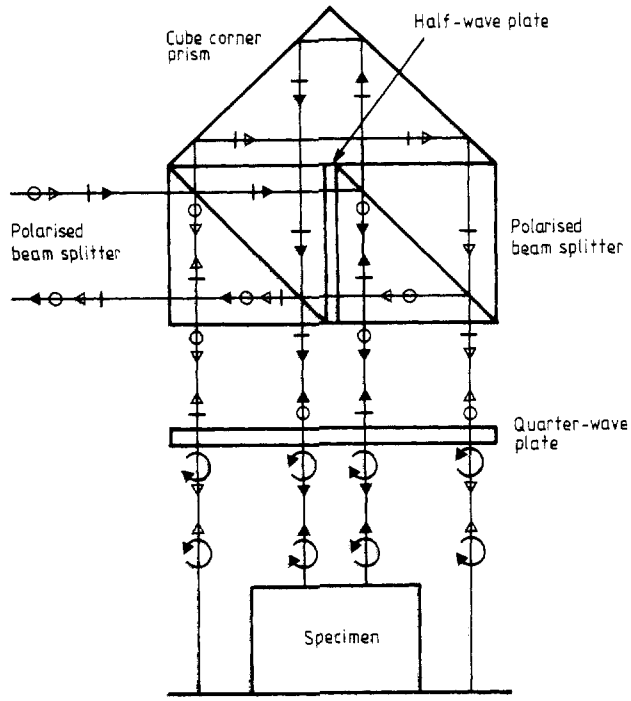

Figure 1. The two-fold path interferometer.

The incident light beam from a stabilised Zeeman laser has two components which are characterised by linearly polarising planes orthogonal to each other. As is seen in figure 1, the two components are divided and made parallel by a double beam splitter which consists of two polarising beam splitters separated by a half-wave plate. Each beam, now having the same polarising plane, is reflected by a front mirror or back mirror respectively. The quarter-wave plate rotates the polarisation of the returning beam through 90° and it is therefore transmitted by the original beam splitter and folded through exactly 180° by a cube corner prism. Then, the beams trace the second transmission/reflection paths before finally being recombined by the same double beam splitter.

In this system, very strict parallelism between two polarising planes of the double beam splitter is required. That condition is obtained by using a stable precision rotating and tilting table as a mechanical holder of one of the polarising beam splitters. High sensitivity and easy fringe detection have been achieved by optical heterodyne interferometry. The same detection system described in the previous work (Okaji and Imai 1983) is used in the present work, and sub-nanometre resolution in length measurement can be also obtained.

2.2. Specimen holder and temperature enclosure

In the previous papers, we have shown the utility of the parallel spring strip both as a holder of a specimen and a support for two plane mirrors (Okaji *et al* 1981b, c). In the present work, the same type of parallel spring specimen holder is also used with a

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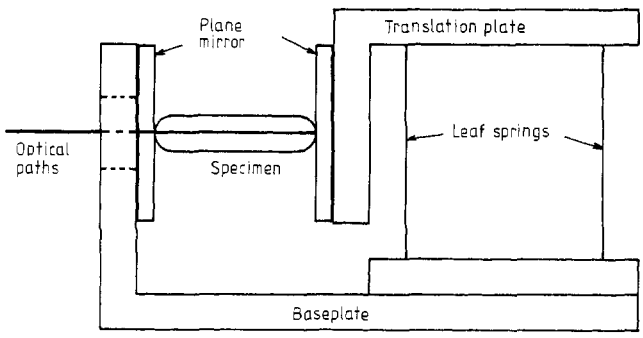

Figure 2. Schematic diagram of the parallel spring specimen holder.

few modifications. As is shown in figure 2, the specimen is attached directly to two mirrors, one of which is fixed to the baseplate and the other is fixed to the translation plate of the spring component. The specimen can be supported by the force of the springs.

The dimensional uncertainties between the two plates and the four leaf springs of the present spring component are within 10 μm over 50 mm long plates and springs respectively. Therefore, nonparallel movements of the spring component can be suppressed to be within 0.2 seconds pitching and 0.02 seconds yawing over a 100 μm translation. Pitching and yawing have been reduced by one tenth of that in our previous system (Okaji *et al* 1981b). Each part of the specimen holder is made by carbon steel blocks (S55C which contains 0.55% carbon) and 0.6 mm thick 18Cr–8Ni stainless steel springs.

The specimen holder is set up in a simple temperature-controlled enclosure which has a 10 mm thick styrofoam and 5 mm thick acrylate resin wall and has a thermal regulator using the Peltier effect with a small fan to produce rapid temperature changes between 0 °C and 40 °C with a uniform temperature distribution of about 15 mK. The enclosure is set up on a heavy iron plate on which the interferometric system is also positioned.

3. Performance

To provide measurement versatility, the system should be used in air, so that a correction of a change in refractive index of the air is required. This introduces a small increase in the overall uncertainties (see table 1). Under these conditions, very quick temperature equilibrium in the enclosure is possible, and a typical time interval for a measurement can be shortened to about 30 min.

3.1. Uncertainties in length and temperature measurement

The linear thermal expansion coefficient is generally determined by

$$\alpha = \mathrm{d}l/(l\mathrm{d}T) \tag{1}$$

where, $\mathrm{d}l(=l_1-l_2)$ is a length change of a specimen, l is a specimen length, and $\mathrm{d}T(=T_1-T_2)$ is a temperature interval. Subscripts 1 and 2 represent the initial and final states of the measurement, respectively.

The measurement uncertainties were estimated with following determinations.

$$\delta\alpha_{\mathrm{total}} = (\delta l/l)_{\mathrm{sp}}\alpha + [\delta(\delta l/l)]_{\mathrm{L}}/\mathrm{d}T + [\delta(\mathrm{d}T)/\mathrm{d}T]_{\mathrm{T}}\alpha \tag{2}$$

Each subscript means that each term is associated with the uncertainty of the measurement of specimen length, length change, and temperature change, respectively. Each term is represented by

$$(\delta l/l)_{\mathrm{sp}},$$

$$[\delta(\mathrm{d}l)/l]_{\mathrm{L}} = [\delta(\mathrm{d}l)/l]_{\mathrm{las}} + [\delta(\mathrm{d}l)/l]_{\mathrm{fri}} + [\delta(\mathrm{d}l)/l]_{\mathrm{air}} = \sum_i [\delta(\mathrm{d}l)/l]_i, \qquad i = 1, 2, 3,$$

$$[\delta(\mathrm{d}T)/\mathrm{d}T]_{\mathrm{T}} = [\delta(\mathrm{d}T)/\mathrm{d}T]_{\mathrm{res}} + [\delta(\mathrm{d}T)/\mathrm{d}T]_{\mathrm{flc}} + [\delta(\mathrm{d}T)/\mathrm{d}T]_{\mathrm{cal}} = \sum_j [\delta(\mathrm{d}T)/T]_j, \qquad j = 1, 2, 3.$$

Where, $i = 1, 2, 3$, refers to a laser stability, a fringe determination, and a correction of refractive index of air, respectively; and $j = 1, 2, 3$ refers to a resolution of temperature measurement, a fluctuation of temperature, and a thermocouple calibration, respectively. All terms except for terms of specimen length and thermocouple calibration are associated with random error so that an amount of total uncertainty can be determined by

$$\alpha_{\mathrm{total}} = \sqrt{\sum_{i,k}^{3,2} (\delta l/l)^2_{i,k}/(\mathrm{d}T)^2 + \sum_{j,k}^{2,2} [\delta(\mathrm{d}T)/(\mathrm{d}T]^2_{j,k}\alpha^2} + \{(\delta l/l)_{\mathrm{sp}} + [\delta(\mathrm{d}T)/\mathrm{d}T]_{j=3}\}|\alpha|. \tag{3}$$

where, $|\alpha|$ is an absolute value of expansion coefficient, and $k = 1, 2$ represents initial and final state of the measurement, respectively.

The specimen length is measured to be within 10 μm uncertainty with a micrometer calliper. This uncertainty contributes $2 \times 10^{-4} \times \alpha$ to the expansion coefficient of a 50 mm long specimen.

Uncertainties in length measurement are due to the long-term stability of the laser wavelength, the fringe determination ambiguity, and uncertainties associated with corrections of refractive index of the air. The long-term stability of the laser (STZL-1, stabilised transverse Zeeman laser, product of Asahi Spectra Co. Ltd) is 1×10^{-9}, and fringe determination is estimated to be within $\lambda/600$ (about 1 nm) by an AC fringe-detection method. The temperature, pressure, and humidity were measured to determine the refractive index of the air through which the laser beams pass. The uncertainties in their determinations are 10 mK, 10 Pa, and 3%RH, respectively. The respective uncertainty contributions of all these factors are 0.1, 2, 0.9, 2.7 and $2 \times 10^{-9}\,\mathrm{K}^{-1}$ to the expansion coefficient of a 50 mm long specimen over a 10 K interval. These uncertainties are independent of the magnitude of the thermal expansion coefficient.

The uncertainties of the temperature interval measurement are associated with the resolution of temperature measurement, the temperature variation close to the specimen, and the calibration uncertainty of the thermocouples. The voltage of copper–constantan thermocouples used in the present experiment can be measured with a digital voltmeter with a 0.1 μV resolution (which corresponds to about 2.5 mK). The temperature stability in the enclosure is within 15 mK, and the calibration uncertainty of the thermocouples was within 0.2%. Therefore each uncertainty represents $2.5 \times 10^{-4} \times \alpha$, $1.5 \times 10^{-3} \times \alpha$, and $2 \times 10^{-3} \times \alpha$ respectively, under the same experimental conditions.

3.2. Uncertainties associated with the specimen supporting system

There are many possible sources of uncertainties in the parallel

spring movement, such as pitching, yawing, and rolling of the movement due to temperature changes of the specimen and the holder. Such uncertainties are caused by errors in the parallelism of the spring movement, and by differences of expansion coefficient of the specimen holder component materials.

It is not necessary to consider the effect of rolling in the spring strip hinge because its axis coincides with the measuring axis. Also the effect of yawing introduces small uncertainties which are compensated by the interferometer. Pitching uncertainty remains if the specimen axis is offset from the plane of measurement. Suppose the amount of offset is 1 mm, the error in length (Abbe's error) becomes 1×10^{-9} m over a 100 μm translation of the component. Hence, if a specimen which has an expansion coefficient of 20×10^{-6} K^{-1} is measured, a relative translation of the strip hinge is 4.5 μm which is estimated by a determination of $\Delta a \times l \times \Delta T$, where $\Delta\alpha$ is a difference of expansion coefficient between the specimen and plate of the specimen holder, l is the specimen length, and ΔT is the temperature interval. Then, the uncertainty of the length measurement is $4.5 \times 10^{-11}(1 \times 10^{-9} \times (4.5/100)]$ m. The uncertainty of the expansion coefficient introduced by pitching is, therefore, 0.1×10^{-9} K^{-1} in this case.

In this case of Fizeau or Fabry–Perot interferometry, very stable optical contacts are usually obtained between the specimen and baseplate reference. Conversely, in this case, the length of a specimen is not so well defined due to reduced rigidity and stability. However, these uncertainties are difficult to estimate. Sources of uncertainty associated with the differences of expansion coefficients between each part of a specimen holder are also hard to estimate quantitatively, owing to differing thermal responses of its component parts. The magnitudes of these uncertainties were, therefore, estimated from the specimen measurements.

3.3. Total uncertainty

The magnitudes of uncertainties discussed are listed in table 1. The amount of total uncertainty depends on the magnitude of the expansion coefficient of the specimen. An estimate of the total uncertainty is generally represented from the equation (3) as,

$$\sqrt{0.345 \times 10^{-16} + 2.31 \times 10^{-6} \times \alpha^2} + 2.2 \times 10^{-3}|\alpha| \quad (\mathrm{K}^{-1}). \qquad (4)$$

The terms of the square root represent the total amount of uncertainties associated with random errors and the second with systematic errors.

4. Experimental results and discussions

4.1. Specimens

In order to determine the accuracy of the measuring system, various kinds of specimens such as low-expansion materials, sintered hard alloys which are difficult to machine, metals having high expansivities, industrial materials, and standard reference materials were used.

Twelve kinds of specimens were measured. Shapes and dimensions of specimens were 5 mm diameter or 5 mm square and 50 mm long rods, the ends of which were rounded. The first three were low-expansion materials: Super-Invar alloy, Invar alloy, and a glass-ceramic (product of Hoya Glass Co.). Two were sintered tungsten carbide containing 7 and 12% cobalt, respectively. One was elastic Invar (Nickel span C). Two were carbon steel, one was a 50 mm long bar gauge, and the other was S55C (contains 0.55% carbon). Two were aluminium cast and aluminium alloy which have relatively high expansion coefficients. The last two were standard reference materials, copper SRM 736 and tungsten SRM 737, supplied by NBS.

4.2. Results and discussions

The measurements were carried out based on the factorial design to analyse the instrument performance. Each specimen was inserted into the strip hinge support and the linear thermal expansion coefficient was determined four times over the temperature range 15–25 °C before the specimen was removed and relocated in the holder. This procedure was repeated three times for each specimen resulting in twelve determinations. Since there were twelve different materials measured, a total of 144

Table 1. Sources of uncertainty and estimated amount for a 50 mm long specimen over a 10 K interval of temperature.

Source	Contribution to expansion coefficient (K^{-1})
Random error	
Laser stability	0.1×10^{-9}
Fringe determination	2×10^{-9}
Refractive index correction	
Temperature	1.4×10^{-9}
Pressure	2.7×10^{-9}
Humidity	2×10^{-9}
Temperature determination	$2.5 \times 10^{-4} \times \alpha$
Temperature fluctuation	$1.5 \times 10^{-3} \times \alpha$
Relocation of the specimen	not estimated
Systematic error	
Determination of specimen length	$2 \times 10^{-4} \times \alpha$
Thermocouple calibration	$2 \times 10^{-3} \times \alpha$
Excess motion of components	
Pitching effect	0.1×10^{-9}†
Thermal response	not estimated

† Maximum value

Table 2. Linear thermal expansion coefficients and standard deviations of twelve specimens at 20 °C.

Specimen	Thermal expansion coefficient ($\times 10^{-6}$ K^{-1})	Standard deviation ($\times 10^{-6}$ K^{-1})
Super-Invar	−0.812	0.034
Glass-ceramics	0.000	0.029
Invar	0.351	0.011
Tungsten carbide†	4.049	0.027
Tungsten carbide‡	4.228	0.032
Tungsten SRM 737	4.413	0.026
Elastic Invar	7.245	0.026
Carbon steel§	10.984	0.035
Carbon steel‖	11.314	0.020
Copper SRM 736	16.556	0.033
Aluminium diecast	21.238	0.068
Aluminium alloy	23.129	0.070

† containing 12% of cobalt.
‡ containing 7% of cobalt.
§ bar gauge.
‖ containing 0.55% of carbon (S55C).

Table 3. Analysis of variance table for the estimation of measurement error.

Source of variation	Degrees of freedom	Expectation of mean square	Sum of squares $(\times 10^{-6}\,K^{-1})^2$	Mean square $(\times 10^{-6}\,K^{-1})^2$	F-test
Material A	11	$\sigma_{e_2}^2 + 4\sigma_{e_1}^2 + 12\sigma_A^2$	8843.6	804.0	†
Replication R	2	$\sigma_{e_2}^2 + 4\sigma_{e_1}^2 + 48\sigma_R^2$	0.1858×10^{-1}	0.9292×10^{-2}	
Interaction e_1(A × R)	22	$\sigma_{e_2}^2 + 4\sigma_{e_1}^2$	0.9326×10^{-1}	0.4239×10^{-2} (V_{e_1})	†
Error e_2	108	$\sigma_{e_2}^2$	0.7658×10^{-1}	0.7091×10^{-3} (V_{e_2})	
Total	143		8843.8		

† significant at 1% level

determinations were made. Factors to be considered were type of material, replication of the measurement, their interaction, and random error.

The mean and standard deviation of the linear thermal expansion coefficients are listed in table 2. Table 3 shows the result of variance analysis for 144 measured data. Significant difference in the factors of material and interaction are revealed. It is obvious that the type of material factor should be significant. The significant difference of interaction (first-order error) means that there is significant uncertainty due to the interaction between material and replication of measurement. Thus, this interaction can be considered to be a measurement error σ_{e_1} and is estimated as follows from the figures in table 3.

$$\sigma_{e_1} = \sqrt{(V_{e_1} - V_{e_2})/4} = \sqrt{(0.004239 - 0.000709)/4} = 0.030.$$

This value is nearly equal to the mean of standard deviations in table 2. As is shown in figure 3, the amount of the measured standard deviations are larger than those of theoretically estimated ones. However, these uncertainties are not excessive, and the increased uncertainty for such a versatile system does not exceed $4 \times 10^{-6}\,K^{-1}$. It corresponds to only 0.02 μm in terms of the uncertainty in length measurement, and is sufficiently small for practical determinations.

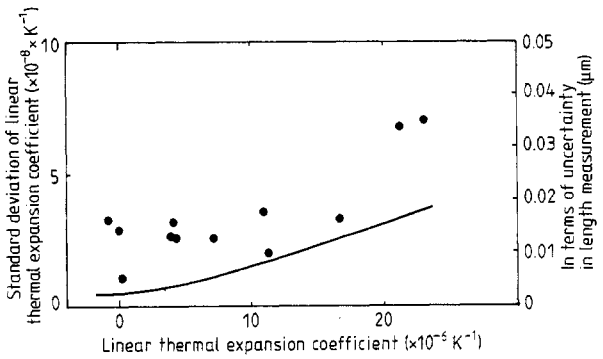

Figure 3. Standard deviations of measured and estimated expansion coefficients of twelve specimens: ●, standard deviation, ——, estimated total random error.

In order to determine an absolute measurement accuracy, two standard reference materials, copper SRM 736 and tungsten SRM 737 were measured to compare with NBS calibrations (Kirby and Hahn 1975, 1976). The comparisons are shown in figure 4. Here, error bars representing standard deviations on

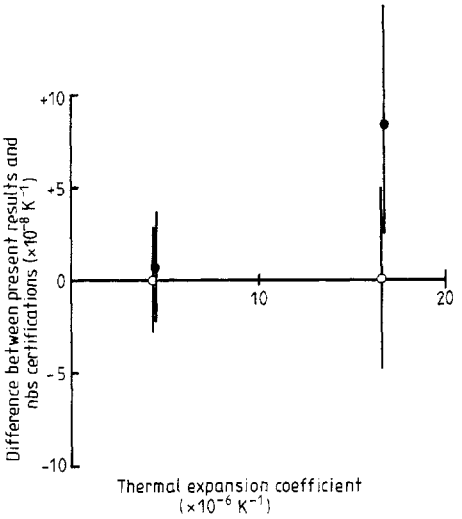

Figure 4. Differences between present (○) and NBS (●) calibrations of SRM 736 (copper) and SRM 737 (tungsten).

every twelve observations are shown. There is no significant difference between these results and NBS calibrations of both materials. The differences represent only 0.16% and 0.51% of the expansion coefficient, respectively.

5. Conclusion

A thermal expansion measuring method for obtaining both high accuracy and versatility has been described. A system providing self-compensation of optical misalignment and high sensitivity in fringe determination has been described using a double-path optical heterodyne laser interferometer and a specimen holder using a parallel spring component. An uncertainty of determination in length measurement is estimated to be within $\lambda/600$ by an AC fringe-counting method, and the flexible specimen supporting system does not add any excess uncertainty to the measurement. A highly reliable and rapid measurement in air at room temperatures can be achieved with a simple temperature-controlled enclosure. The total uncertainty of the measurement is estimated from measurements of twelve kinds of specimens including two standard reference materials to be $3 \sim 7 \times 10^{-8}\,K^{-1}$ on materials which have linear expansion coefficients of $-1 \sim 23 \times 10^{-6}\,K^{-1}$.

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