

REVIEW ARTICLE

A review of measurement techniques for the thermal expansion coefficient of metals and alloys at elevated temperatures

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Abstract

Metallurgical operations at elevated temperatures, such as those that involve solidification and/or mechanical deformation, can be critically influenced by the thermal stresses and strains that result from expansion and contraction of the material as a function of temperature. With the increasing use of computer-based process models for these operations, there arises a greater need for quantitative data on the thermal expansion coefficient of the relevant alloy at the temperatures involved. After briefly reviewing some existing sources of data for this property, the various techniques for its measurement at elevated temperatures are then described. These include mechanical dilatometry, optical imaging and interference systems, x-ray diffraction methods and electrical pulse heating techniques. Finally the implications, for process modelling, of the available data and measurement techniques are discussed.

Keywords: coefficient of thermal expansion, dilatometer, interferometer, x-ray diffraction, metals processing, high temperature

1. Introduction

Metals and alloys undergo dimensional changes in response to variations in temperature, a phenomenon that has critical implications for industries that process these materials at high temperatures. Solidification practices, such as casting or sprayforming, and hot working procedures in the solid state, such as forging, rolling or extrusion, can be significantly influenced by thermal stresses and strains that arise from temperature variations. If not properly controlled, these effects can have an adverse influence on product quality. Residual stresses and distortion can be introduced and in the worst cases crack-type defects can be formed. Hence the optimization of such processes calls for the use of sophisticated computer models that require comprehensive data on the thermal expansion characteristics of the material. The availability of data for the thermal expansion coefficient at elevated temperatures then becomes a key issue. It is thus appropriate at this time to perform a review of the experimental

techniques for measurement of this property, giving particular emphasis to work at elevated temperatures.

Before presenting the results of this work it is first necessary to examine the alternative definitions of the thermal expansion coefficient, as the exact value can vary according to the definition employed, a fact that must be borne in mind by anyone wishing to employ such data. Some existing sources of data on this property for metals and alloys are presented, with consideration given to values in both published literature and electronic format. A great variety of empirical methods have been employed in the measurement of thermal expansion, and the main part of this paper is then devoted to a review of these techniques, with particular reference to their accuracies and the temperature ranges over which they can be used. Reference is also made to some of the standards in existence for thermal expansion measurement. Finally the implications of these findings are discussed with respect to the modelling of industrial processes.

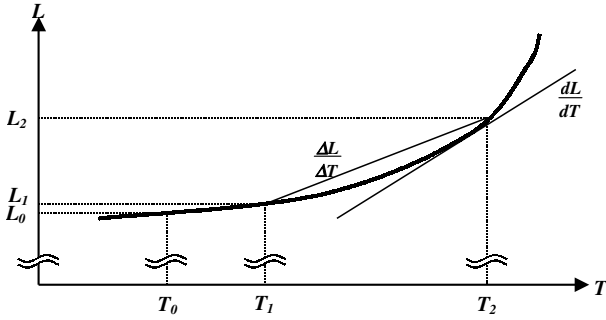


Figure 1. Change in length, L , of a sample of material as a function of temperature, T .

1.1. The coefficient of linear thermal expansion

The expansion of any material with temperature can be quantified in terms of increasing linear dimension, such as the length of a specimen. In its simplest form, the *coefficient of linear thermal expansion* of any material can be defined as the fractional increase in length (linear dimension) per unit rise in temperature. The precise definition is slightly more complex with a number of alternatives as described below. The SI units of this quantity are K^{-1} and typical values for metals and alloys are in the range 10×10^{-6} to $30 \times 10^{-6} \text{K}^{-1}$. Ceramics normally have lower values in the range 1×10^{-6} to $20 \times 10^{-6} \text{K}^{-1}$, and it is generally found that materials with higher melting temperatures have lower thermal expansion coefficients. An approximate rule of thumb for materials that melt above 600°C is that they have a linear expansion of about 2% when heated from room temperature to their melting points [1].

It is important to note that the coefficient of thermal expansion (CTE) is a temperature-dependent property and will, for most metals and alloys, gradually increase with temperature. However in some instances the occurrence of phase changes will cause discontinuities in the value of the coefficient—for example a sharp contraction occurs on heating iron-based materials through the ferrite to austenite transition, as the crystal lattice changes from body-centred cubic to the more compact face-centred cubic structure.

Examination of the literature reveals a number of numerical formulae for calculating the thermal expansion coefficient. These can be grouped into two broad categories, depending on whether the expansion relates to a temperature range or a single temperature. Both of these have further subtle variations and the values of the coefficient can vary significantly according to the definition employed.

1.1.1. Definition over a temperature range. Consider a sample of material with initial length L_0 at temperature T_0 which expands to L_1 at T_1 , and then to L_2 at T_2 , figure 1.

The most general definition is the ‘mean’ or ‘average’ coefficient of expansion as given by ASTM [2–4]:

$$\alpha_m = \frac{(L_2 - L_1)/L_0}{T_2 - T_1} = \frac{1}{L_0} \frac{\Delta L}{\Delta T}. \quad (1)$$

Here α_m is related to the slope of the chord between two points on the curve of length against temperature, and so represents the expansion over the particular temperature *range* from T_1 to T_2 . Furthermore, the fractional increase in length is calculated

by dividing the increase in length by the length at T_0 . It is thus necessary to specify all three temperatures when quoting the value of CTE. Usually T_0 is defined as a reference temperature such as 20°C .

Often the lower limit of the specified temperature range is also used as the reference temperature, leading to a modification of the equation to the following [5, 6]:

$$\alpha_{m*} = \frac{(L_2 - L_1)/L_1}{T_2 - T_1} = \frac{1}{L_1} \frac{\Delta L}{\Delta T}. \quad (2)$$

Sometimes tables are listed with pairs of values for α_{m*} and temperature. This can lead to confusion if it is not made clear that the CTE refers to a temperature range and not to a single temperature.

Another definition involving a temperature range is the *instantaneous* coefficient of thermal expansion [6] at temperature T_m calculated as follows:

$$\alpha_t = \frac{(L_2 - L_1)/L_0}{T_2 - T_1} \quad \text{where} \quad T_m = \frac{T_1 + T_2}{2}. \quad (3)$$

1.1.2. Definition at a single temperature. In contrast to the previous definitions, the *true* coefficient of linear thermal expansion [6] (also referred to as *thermal expansivity* [2]) is related to the derivative dL/dT at a single temperature. This is the slope of the tangent to the curve of length against temperature (figure 1). The true coefficient can thus be defined as follows:

$$\alpha_t = \frac{dL/L}{dT} = \frac{1}{L} \frac{dL}{dT}. \quad (4)$$

This is the limiting case of equation (2) as T_1 and T_2 become closer. It is a more fundamental definition than those described earlier, as only one temperature needs to be specified. However it is sometimes more convenient to use a slightly modified definition which uses the original length L_0 as the denominator for the fractional length change:

$$\alpha_{t*} = \frac{1}{L_0} \frac{dL}{dT}. \quad (5)$$

The relationship between these two values is thus

$$\frac{\alpha_{t*}}{\alpha_t} = \frac{L}{L_0}. \quad (6)$$

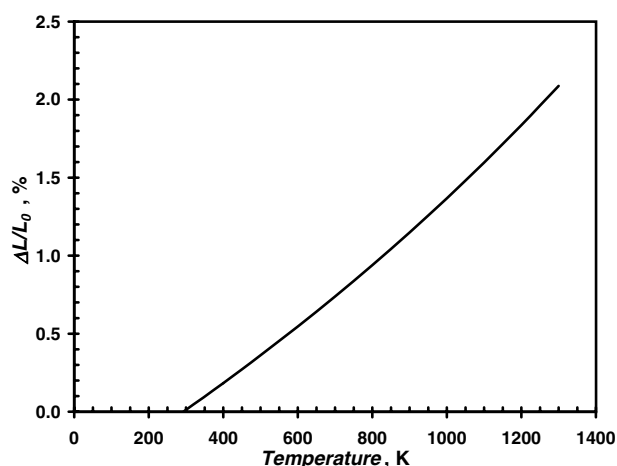
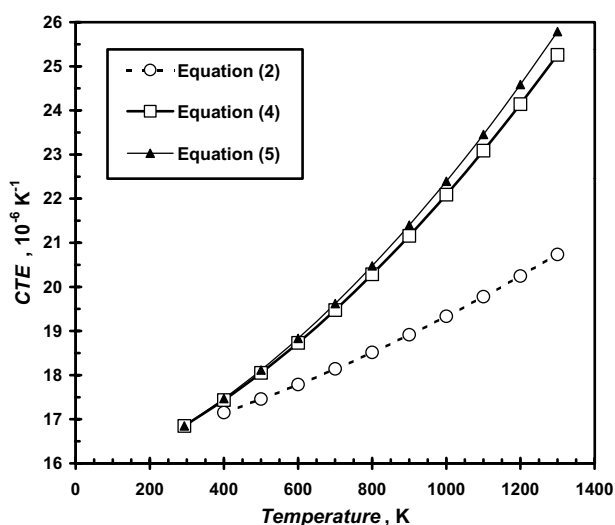
1.1.3. Comparison of values. The difference between the different coefficients can be illustrated by an example, such as the expansion of copper. Recommended literature values for the *thermal expansion*, $\Delta L/L_0$ (as a percentage) for this material have been fitted to the following equation [7] over the range 293 to 1300 K.

$$\begin{aligned} \Delta L/L_0 = & 1.685 \times 10^{-3}(T - 293) \\ & + 2.702 \times 10^{-7}(T - 293)^2 + 1.149 \times 10^{-10}(T - 293)^3. \end{aligned} \quad (7)$$

Here T is absolute temperature and L_0 is defined at 293 K. Values derived from equation (7) are tabulated in table 1 and plotted graphically in figure 2. Using this data for $\Delta L/L_0$, values of CTE defined according to equations (2), (4) and (5) can be calculated. These are also listed in table 1. Note that

Table 1. Values of percentage thermal expansion of copper and three different values of CTE calculated from equations (2), (4) and (5).

Temperature (K)	$\Delta L/L_0$ (%)	CTE equation (2) (10^{-6} K^{-1})	CTE equation (4) (10^{-6} K^{-1})	CTE equation (5) (10^{-6} K^{-1})
293	0.000		16.85	16.85
400	0.184	17.15	17.44	17.47
500	0.361	17.46	18.05	18.12
600	0.546	17.79	18.73	18.83
700	0.738	18.14	19.48	19.62
800	0.939	18.52	20.29	20.48
900	1.148	18.91	21.16	21.40
1000	1.367	19.33	22.09	22.39
1100	1.596	19.78	23.09	23.46
1200	1.836	20.25	24.14	24.59
1300	2.088	20.74	25.26	25.79

**Figure 2.** Percentage thermal expansion of copper as a function of absolute temperature.**Figure 3.** Variation in the value of CTE of copper according to the definition employed.

the temperature corresponding to each value of the mean coefficient (equation (2)) is actually the upper point of the temperature range for which the expansion occurs (the lower point being 20 °C in each case). These data are plotted

graphically in figure 3, which illustrates the differences in the value of CTE depending on how it is defined. Due to the curvature of the $\Delta L/L_0$ versus temperature plot, the CTE defined over a temperature range (equation (2)) is significantly lower than that defined at a single temperature, and this difference becomes greater as the temperature increases. The difference between the coefficients defined by equations (4) and (5) is relatively small. All of these differences have important implications for the way the CTE data is used in engineering situations.

1.2. Volumetric expansion

An alternative means of quantifying expansion is in terms of the fractional increase in volume per unit rise in temperature. Depending on whether this is defined over a temperature range or at a single temperature, the corresponding relationships will be as follows [5, 6]:

$$\beta_{m*} = \frac{(V_2 - V_1)/V_1}{T_2 - T_1} = \frac{1}{V_1} \frac{\Delta V}{\Delta T} \quad (8)$$

$$\beta_t = \frac{1}{V} \left(\frac{\partial V}{\partial T} \right)_p \quad (9)$$

where β is the coefficient of volumetric thermal expansion. Since this definition is often used for the thermal expansion of liquids, the condition of constant pressure is often included in the equation. Strictly speaking this should also be included in the definition of linear expansion.

For an isotropic material the true coefficient of volumetric thermal expansion is equal to three times the true coefficient of linear thermal expansion [6]

$$\beta_t = 3\alpha_t. \quad (10)$$

While equation (10) holds for coefficients defined at a single temperature, the relationship between volumetric and linear coefficients defined over a temperature range is more complex [6].

2. Data available for coefficient of thermal expansion

Some sources of available data for the coefficient of thermal expansion have been briefly reviewed, including both published information and values in electronic databases. Pure metals appear to have been well characterized up to their melting temperatures, but data for engineering alloys at very high temperatures is often limited.

2.1. Published data

A number of standard metallurgical encyclopaedias have data on the coefficient of thermal expansion of metals and alloys. Smithells [8] gives values for *pure* metals at elevated temperatures with references to the sources of the data. For a variety of non-ferrous alloys a single value is given for the range 20–100 °C, but no references. For steels, values are given for a range of temperatures up to 1000 °C.

The *Materials Selector* [9] gives some values for steels up to 1000 °C, and for non-ferrous alloys over the range room temperature to 100 °C. However source references are

not given. The *Metals Databook* by Robb [10] also gives values without sources, mainly in the range up to 300 °C for non-ferrous and up to 500 °C for ferrous alloys.

More detailed data is provided in the *ASM Metals Handbook*, but again without source references. Data on steels [11] are provided up to 1000 °C. These are the average values of the coefficient over the range from 20 °C to the temperature specified. Values for most non-ferrous alloys [12] are given up to 300 °C, with data for some superalloys and refractory alloys to 1100 °C [13].

The most comprehensive published compilation of thermal expansion data consists of two volumes produced by the Thermophysical Properties Research Centre of Purdue University in Indiana, USA. One of these concentrates on non-metallic solids [14], while the other deals specifically with the thermal expansion of metallic elements and alloys [15]. These are part of a 13 volume series entitled *Thermophysical Properties of Matter* edited by Touloukian and Ho [16], which includes corresponding collections of data for thermal conductivity, diffusivity, radiation, specific heat and viscosity. Previously these were produced in a slightly different format [17] with individual volumes dealing with different classes of high-temperature materials.

The volume dealing with expansion of metals [15] covers 64 elements, 94 intermetallic compounds, 125 binary alloys and 70 groups of multiple component alloys, with an extensive index system for locating the appropriate material. In all cases reference is made to the original source article containing the data and, where appropriate, details of the chemical composition, heat treatments etc of the specimen used are included together with an indication of the type of measurement technique employed. The data are presented in a consistent form with graphs and tabulated values of the percent expansion versus temperature from the original experiments, with polynomial fits included. Values of thermal expansion coefficients derived from this data are then presented as a function of temperature. For each material, data from several separate studies are given, and where possible the results have been critically evaluated to enable a set of 'recommended' values to be presented. One of the drawbacks of this data compendium is that it was published in 1975 and thus does not include data acquired over the last 25 years. However there are plans to update this work, possibly in an electronic database format [18]. Examination of the temperature ranges for which thermal expansion values are given reveals that there is very little data available as the melting temperature is approached, except in the case of some pure metals.

Wang and Reeber [19] have published a more up to date review of data for BCC refractory metals such as vanadium, niobium, tantalum, molybdenum and tungsten. This includes thermal expansion values in excess of 2000 °C.

For data on steels, another good source is a publication produced by the British Iron and Steel Research Association [20], based on measurements made at the National Physical Laboratory over the temperature range 0–1100 °C. Thermal expansion—temperature plots are given, which also demonstrate the contraction that occurs on heating through the transformation range. Values of the mean coefficient of thermal expansion are derived from these curves. Details of the chemical composition and heat treatment condition of each steel investigated are included.

2.2. Electronic databases

A number of electronic databases exist with materials property data, and in some cases thermal expansion values are included. Some of these are available freely via the internet, while others require payment either for the complete database on CD-Rom or for individual data values.

Of the freely available databases, *Matweb* [21], an internet service based in the USA, has property data on a vast number of alloys, apparently given by the commercial suppliers of the materials. CTE data are included, but usually only for room temperature, although in some cases values are given up to 500 °C.

Metals Infobase [22] is a CD-Rom subscription service provided by ILI Ltd in the UK. This has CTE data for a diverse range of alloys, but again this is generally limited to room temperature. The Cambridge Materials Selector [23] is another UK-based source of materials property data, which includes values for thermal expansion.

The University of Stuttgart has a database called THERSYST [24] for a variety of alloys, for which fees must be paid for individual sets of data. CTE data is available for elevated temperatures.

ESM software has a collection of databases called *MAPP* [25] for sale—these employ data gathered by ASM International. Apparently this includes thermal expansion data.

JAHM software in the USA promotes a temperature dependent materials property database [26], which includes CTE values. The data comes from a number of sources including the Purdue volumes discussed earlier [15]. The software not only generates tabulated values of the properties, but also can write computer code subroutines for interfacing with e.g. finite element programs.

Densities of solid and liquid alloys, which may be used to calculate CTE data, are contained in the *KINDAS* database [27] available from Aachen University of Technology (RWTH Aachen) in Germany. This Institution has also set up a website listing other databases that are relevant to thermophysical data for casting simulations [28].

3. Experimental measurement techniques

Determination of the thermal expansion coefficient requires the measurement of two physical quantities, displacement and temperature, for a sample of the material that is undergoing an appropriate thermal cycle. Examination of the literature reveals that many different experimental approaches have been employed. Some of these are absolute methods, while others are relative techniques that utilize a reference material.

The sheer diversity of these techniques can be seen from the different methods adopted to measure displacement. A variety of options for mechanically transmitting the displacement to ambient temperature sensors have been used. Non-contact methods include the formation of optical images and diffraction patterns from the specimen surfaces. Beams of x-rays, neutrons or electrons have been diffracted from the crystal lattice planes to measure the increase in atomic spacing. Furthermore strain gauges, capacitance techniques and high-resolution microscopes have also been used to quantify expansion.

These techniques are described below, with particular emphasis on those which enable measurement at elevated temperatures.

3.1. Mechanical dilatometry

One of the oldest and most frequently used methods for thermal expansion measurement is mechanical dilatometry. The displacement that results from a specimen with increasing temperature is mechanically transmitted to a sensor located away from the heat, yielding a plot of displacement against temperature from which the CTE can be calculated.

The most common arrangement is that of the push rod dilatometer [29–31] which has been incorporated into a number of standards. A specimen is enclosed in a furnace or other controlled temperature environment. Two rods in contact with opposite faces of the specimen are aligned side by side, parallel to the direction of expansion, figure 4(a). The rods protrude out from the temperature environment to a displacement sensor, such as a linear variable differential transformer (LVDT), which is maintained at ambient temperature. This measures the relative displacement of the rods as the specimen temperature is changed. A particularly convenient arrangement is for one of the rods to be in the form of a closed tube (figure 4(b)) in which the specimen is located, so that the other rod runs down the axis of the tube and rests against the opposite surface of the specimen. As the specimen expands it pushes this central rod along the axis of the tube so that the relative movement can be measured.

During specimen expansion, the portions of the rods lying within the furnace also heat up and expand. However, if the temperature profile is identical in both rods, this effect can be neglected except for the amount of expansion in that portion of the longer rod (or tube) that corresponds to the specimen length, distance x in figure 4(a). Addition of this expansion to the measured displacement at the sensor then gives the expansion of the specimen. The amount by which distance x increases can be determined by calibrating the apparatus with a specimen of a reference material, for which the expansion characteristics are well known.

In practice the expansion of the two pull rods is not quite identical. Hence changes in length of the components of this transfer mechanism, which may arise from thermal expansion, phase changes or a response to stress (elastic, plastic or creep deformation) must be taken into consideration. Consequently the accuracy of the apparatus is critically dependent on the material selected for construction of the push rods. One of the most suitable is vitreous silica [31], as in the amorphous state this material has a very low thermal expansion coefficient of the order of $0.5 \times 10^{-6} \text{ K}^{-1}$ (Kikuchi *et al* [32] have studied the marginal differences in thermal expansion behaviour between different types of vitreous silica). The devitrification (i.e. transformation from a glassy state to a crystalline structure) that occurs in this material at higher temperatures places an upper limit on the temperature range for which it can be used in this application, since the crystalline forms have a much higher thermal expansion. This limit has been described by different sources as 700°C [30], 900°C [2] or 1000°C [29, 31]. In some cases however, contamination of the silica by the specimen material can occur at lower temperatures than these and cause

crystallization. High temperature use requires rods made from alumina [33], either in single crystal form (sapphire) or sintered polycrystalline state. Above 1600°C , the extreme conditions necessitate the use of fine-grained isotropic graphite. In all cases the tube and push rod should be produced with an identical orientation from the same batch of material.

The furnace employed should be sufficiently long to produce a uniform temperature within the specimen. A uniform heating rate of less than 3°C min^{-1} is generally recommended [30], and an identical heating ramp should be applied during the calibration run with the reference material. Faster heating rates have been used by some researchers, for example Badini *et al* [34] used $10^\circ\text{C min}^{-1}$ for measuring the thermal expansion of a cast aluminium–saffil fibre composite over the range $30\text{--}300^\circ\text{C}$. Whatever heating ramp is employed, care is required in locating the thermocouples where they will record the actual temperature of the specimen. At high temperatures dilatometers have to employ pyrometers for temperature measurement, and a technique for calibrating these [35] employs a sample with a well defined melting temperature, which results in a sharp change in the dilatometer reading at the melting point. Contact between the push rods/tubes and the specimen is another important factor to be considered in minimizing errors. The contacting surfaces must be either flat or rounded to a large radius. Pointed ends should be avoided as these can lead to local deformation.

Many dilatometers are mounted horizontally, as this gives better temperature uniformity within the furnace. In these cases a small tracking force is applied to the push rod to ensure good contact between the specimen and push rods. This is especially important where measurements are to be made on cooling, as there is a danger of losing contact as the components contract. In contrast, components in a vertical dilatometer can remain in contact under their own weight, which may be at the expense of an inferior thermal gradient in the specimen due to furnace convection currents.

A vertical push-rod dilatometer has been used to measure the expansion of specimens undergoing sintering [36] up to 1500°C with an accuracy of $1 \mu\text{m}$. In some cases this was taken to a temperature where the sample was part molten. A special counterbalance system was employed to prevent the pushrod exerting an excessive force on the sample. Calibration was performed with a single crystal of sapphire to eliminate any contribution from the thermal expansion of the alumina push rod and tube.

Using good practice it is generally possible to measure CTE to an accuracy of around $\pm 1\text{--}2\%$ [31] with dilatometry. However errors will be greater as the melting point of the specimen is approached due to softening effects and potential problems with the sample–push rod contacts.

3.1.1. Special purpose dilatometers. A particular variation on the pushrod dilatometer described above is the *differential dilatometer* [29], figure 4(c). This employs two specimens, one of which is made from a reference material to which the material under investigation is compared. Each specimen has a push rod attached to it and the difference in displacement from the two rods is recorded as the two specimens heat up simultaneously. The thermal expansion of the material under investigation is then calculated from that of the reference

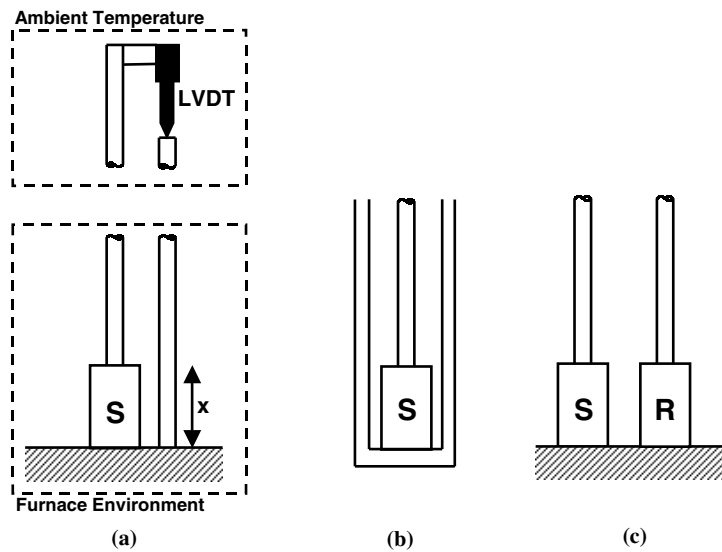


Figure 4. Mechanical dilatometers for thermal expansion measurement of specimen, S: (a) double push rod, (b) tube and push rod, (c) differential with reference specimen, R.

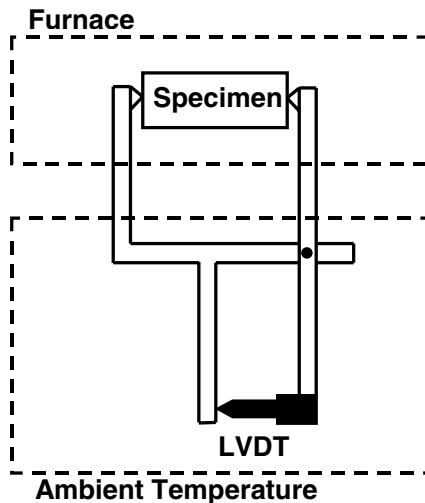


Figure 5. Scissors-type dilatometer with rods exiting the furnace at *right angles* to the measured displacement direction.

material and the relative displacement of the pull rods. Repeating the experiment after interchanging the positions of the two specimens can reduce systematic errors. The reference material should be chosen so that it has similar expansion characteristics to the material under study. This technique is especially useful for quality control purposes where comparison can be made between samples of different batches of the same material.

For any dilatometer, the critical part of the displacement mechanism is the portion that varies from the specimen temperature to ambient temperature. In the arrangements described thus far, the push rods are aligned parallel to the displacement direction. A number of modifications exist [37–39] in which the rods are taken out from the furnace at *right angles* to the displacement direction. These include a Baudran or scissors-type dilatometer [37], in which a pivoting mechanism transfers the displacement to an LVDT, figure 5. It has been claimed that these methods represent

absolute techniques since the expansion of the rods should not contribute to the measured displacement. However, this system does not seem to be employed in commercial equipment currently available. There is though a standard [39] for expansion measurement of investment casting shell moulds, which recommends this method, albeit with the aid of a reference material for calibration.

A technique for measuring bending effects during expansion has been patented by Bassily [38]. This employs spatial-position members perpendicular to the specimen ends to which they are attached. These protrude out of opposite sides of a controlled temperature enclosure, and on each side two quartz rods connect to a displacement transducer. Signals from the two transducers then give a measure both of the overall specimen expansion and of the bending that takes place.

For temperatures near ambient, it is possible to keep the reference structure that transmits the displacement at a stationary temperature while the specimen heats up. Gilmore and Lloyd [40] patented a technique in which the specimen temperature is controlled by reservoirs of hot and cold water, with push rods made out of low thermal expansion, low thermal conductivity material. This was for the purpose of measuring materials with CTE values below about $0.4 \times 10^{-6} \text{ K}^{-1}$. It would be difficult to employ this technique for high temperatures due to the tendency for the displacement mechanism itself to heat up.

Chen [41] has patented a technique for studying phase transformations in steel specimens. As well as measuring thermal expansion it is claimed that the apparatus can measure variations in internal friction and magnetic properties. A long, vertically mounted specimen is heated by either an induction coil or a resistance wire and the increase in length measured by a counterbalanced lever mechanism that connects to a linear variable differential transformer (LVDT). A jet of nitrogen also provides cooling. It is not clear from the description what type of temperature profile is attained, since only one thermocouple is inserted into the bottom end of the specimen.

Dilatometers can also be used for measuring thermal expansion of molten metals (alternative methods include

hydrostatic weighing of an immersed object or measurement of liquid emerging from a capillary [42, 43]). A sealed volume of the liquid is enclosed by a ceramic piston moving within a cylinder that is closed at one end [30]. The increase in liquid volume pushes the piston along the cylinder and this relative motion between piston and cylinder can be recorded in the same way as for the push-rod dilatometers described above. The volume expansion of the liquid can then be calculated after taking into account the expansion of the internal diameter of the cylinder. A variation on this is to use two pistons at either end of an open cylinder [44, 45].

If data are to be recorded by heating from below to above the melting point great care is required. Data analysis procedures must take account of the transition from linear expansion to volumetric expansion measurement. Furthermore, the initial diameter of the sample must be such that it completely fills the cylinder when the melting temperature is reached. If it is too small there will still be a gap between its outer surface and the inside of the cylinder on melting, resulting in an apparent contraction as the piston is pushed into the cylinder so that the liquid can take up the shape of the enclosed volume. On the other hand, if the specimen is initially too large, then as it heats up and expands it is liable to split the cylinder before the melting point is attained.

The situation becomes even more complex for an alloy that melts over a certain temperature range. This case has been examined at the National Physical Laboratory by Morrell and Queded [30, 46] with an arrangement that employed two pistons. Tin and aluminium alloys were tested with pistons/cylinders made from a variety of materials. The clearance between piston and cylinder was found to have a critical effect—if this was too large the molten material could leak out of the enclosed volume, while too small a clearance led to excessive friction. These problems were minimized for aluminium alloys by using boron nitride pistons and cylinder, where a combination of the non-wetting characteristics of BN and the oxide skin on molten aluminium prevented escape of liquid past the piston. Specific problems were associated with the mushy zone temperature range. It was found that, on heating, the specimen retained considerable rigidity beyond the solidus temperature and thus did not completely fill the cylinder until a point well into the semi-solid condition. A further problem occurred when measurements were subsequently taken on the cooling cycle—as the temperature dropped to this ‘deform point’ the contraction ceased to be homogeneous as problems with feeding of liquid into the semi-solid mush started to occur.

Henderson *et al* [44, 45] have also studied thermal expansion in the mushy zone with a similar two-piston arrangement. The difference between the inside diameter of the cylinder and the outside diameter of the pistons was kept to below 10 μm so as to prevent escape of liquid. Graphite was used to fabricate the cylinder and pistons for tests on aluminium alloys, and sapphire for tests on Inconel 718. These container materials were chosen so as to prevent reaction with the specimen. Furthermore since the thermal expansions of these materials were well known, it made it possible to correct for the volume increase of the cylinder–piston arrangement.

3.2. Optical methods

The dimensional changes in a test-piece with increasing temperature can be measured optically with techniques that can be divided into three main categories. The first involves the creation of an image of a specimen or of relevant parts of it, and determining the spatial movement either of the ends or of some clearly defined marks along the length. This generally requires the optical path to be perpendicular to the displacement direction. Examples of this technique include the so-called *twin telemicroscope* method and various optical comparators described below. The second group is based on optical *interference* measurements, and differs fundamentally from the first in that the optical path is parallel to the direction of displacement that is being measured. By calculating the path difference between beams reflected from opposite surfaces of the specimen, the displacement can be determined in terms of the wavelength of the light used. The third group employs *speckle interferometry*, and thus relies on the formation of an interference pattern on the surface of the specimen itself, changes in this pattern then enable the calculation of displacement.

3.2.1. Optical imaging. One of the simplest optical techniques is to employ a specimen with suitable targets that are displaced relative to one another as the specimen heats up. Images of these targets are then viewed in a direction perpendicular to the displacement being measured. This has been referred to as an *optical comparator* or the *twin telemicroscope* technique [47–49].

Specimens, which are typically cylindrical in shape, are heated in a conventional furnace. If the specimen is conducting, the passage of an electrical current directly through the specimen [50] may also be used for heating purposes. Suitable viewing ports must be incorporated both in the furnace and in any environmental chamber that surrounds the specimen, so that the targets remain visible for the duration of the test.

A variety of different targets have been employed including indentations, grooves and holes machined into the specimen, wires or pins attached to the specimen surface, or even the two ends of the specimen itself. In all cases efforts should be made to ensure that the targets are as sharp as possible. Illumination of the targets can either be from the front, so that the reflected light forms the image, or from the rear, so that a silhouette effect is produced. At higher temperatures the radiant light emitted by the specimen itself can be viewed against a dark background—this has enabled measurements to be made up to 3000 °C [49].

Measurement of the displacement of the targets can be carried out with two telemicroscopes of high magnification (e.g. 50 \times) connected via a frame of low expansion material such as Invar, which is maintained at constant temperature outside the furnace. The displacement can then be determined with micrometer eyepieces, having first measured the initial separation of the targets prior to heating. Alternatively, the microscope frame can be rotated to view a constant temperature reference scale located outside the furnace, so that the absolute length at each temperature can be recorded. Variations on this technique include the use of photodiodes [51] or a CCD

array [52] onto which an image of the targets is projected so as to automate the procedure.

The best results are obtained when the portion of the specimen between the targets is at a uniform temperature, or when the temperature distribution in this region can be quantified. Since it is not dependent on comparison to the thermal expansion of a reference material, this method represents an absolute technique for measurement of coefficient of thermal expansion. The accuracy of displacement has been estimated at $\pm 2 \mu\text{m}$, thus for a 50 mm long specimen expanding by 2% of its length (i.e. 1 mm), the uncertainty would be $\pm 0.2\%$ of the change in length [49].

3.2.2. Optical interference. The distance between two points on a specimen can be measured in terms of the number of wavelengths of light travelling parallel to the direction of displacement. Optical interference techniques for measurement of thermal expansion include Fizeau, Fabry–Pérot and laser-based polarizing interferometers [48, 53, 54]. Although the underlying concepts are fairly straightforward, the equipment required is nevertheless elaborate and costly. However, by employing sophisticated instrumentation great accuracy is possible with these absolute techniques. The precision can be considerably better than mechanical dilatometry [3].

A typical arrangement is shown in figure 6(a). The specimen, S, is placed between two optical flats, A and B, which move apart as the specimen expands. Coherent monochromatic light is reflected from the bottom surface of the upper flat (which is transparent) and the top surface of the lower flat, and these two rays interfere either constructively or destructively depending on the separation x . An alternative arrangement, figure 6(b), allows a relative technique which employs a reference sample, R, which acts as the lower reflection surface, while the optical flat is supported either by a ring shaped specimen or three separate specimens of the material under investigation. In this case the change in distance x that is measured by interference is the difference of expansion of this material in relation to the reference.

The specimen and optical flat system are positioned inside a suitable heating system such as a furnace (or cryostat if low temperature properties are required). Use of a vacuum chamber means that the system will give absolute measures of displacement, as no correction for the effect of the refractive index of the atmosphere on the wavelength of light is required. On the other hand the presence of a low pressure gas will aid the transfer of heat from the walls of the furnace to the specimen, thus allowing faster heating rates.

The geometry of the optical system has a number of variations. In a Fizeau interferometer, a point source of light is collimated by a lens to give rays normal to the reflecting surfaces. A pattern of interference fringes can then be seen, with each fringe corresponding to an equal thickness in the distance x between the reflecting surfaces. This pattern will consist of straight bands if one of the reflecting surfaces is very slightly inclined, or a set of concentric rings if, for example, the lower surface of A in figure 6(b) is slightly convex. As the specimen expands the ridges move and if a reference mark is positioned on one of the surfaces the number of ridges passing this point can be counted. A detailed description of

the detectors and associated instrumentation for performing the fringe counting operation has been provided by Ruffino [55]. A Fabry–Pérot interferometer uses optical flats that have highly reflective coatings on them to give multiple reflections, which result in much sharper fringes and the potential for greater accuracy. The flats are kept parallel, and, by having a light source that is not collimated, the angle of incidence will vary slightly so that concentric fringes are created. Each fringe then corresponds to a particular angle of incidence. Whilst this technique works well for low temperatures, deterioration of the special optical coating occurs on heating, making this system unsuitable for high temperature work.

Use of polarized light from a laser beam can give greater accuracy in determining fractions of fringe spacings. This type of arrangement, often referred to as a Michelson interferometer, has been used in a number of systems. Work by Bennett [56] at NPL employed such a technique. Laser light was passed through a beam splitter to create two separate beams, one of which was directed onto the polished top surface of the specimen, the other onto the reflective base on which the specimen sat. A cube-corner reflector allowed each beam to have two passes of the appropriate distance before the two beams were recombined. A spatial fringe pattern was not produced with this technique. Instead an intensity–time plot was recorded from the recombined beam. Bennett's paper described expansion measurements in the range 0–500 °C for a variety of materials. As an indication of the high accuracy of this technique, the expansion coefficient of fused silica (of the order $0.5 \times 10^{-6} \text{ K}^{-1}$) could be determined with an uncertainty of around 1% over a 50 K temperature range for a 50 mm specimen. A similar arrangement to that due to Bennett is included in the double-pass Michelson interferometer described in ASTM E 289-99 (along with a Fizeau technique). This recommends the use of interferometry up to 700 °C.

For the measurement of the thermal expansion of standard length bars close to room temperature, interferometry is accepted as the most accurate technique. For example, length bars in the range 100–1500 mm have been measured at the National Physical Laboratory [57] with an estimated uncertainty of length measurement of $\pm 30 \text{ nm} \pm 6.2 \times 10^{-8} L$, where L is the length of the bar. This corresponds to an uncertainty of between $\pm 0.22 \times 10^{-6} \text{ K}^{-1}$ and $\pm 0.012 \times 10^{-6} \text{ K}^{-1}$ for the CTE, depending on the length of the bar and the temperature increments employed. Suska and Tschirnich [58] have also described an interferometer for length standards, which gives an uncertainty for the CTE of less than $\pm 0.02 \times 10^{-6} \text{ K}^{-1}$ for 1000 mm long bars.

Some researchers have reported very high accuracies, for example Okaji and Yamada discuss a sub-nanometre accuracy for the path length when using a double beam interferometer for the range 0–50 °C [59]. In this case a single crystal specimen of silicon was heated in a vacuum thermal bath with precise temperature measurement facilitated with platinum resistance thermometers. The same equipment was also used to measure the expansion of 100 mm gauge blocks of steel and partially stabilized zirconia over a similar temperature interval [60], leading to an uncertainty of $\pm 0.007 \times 10^{-6} \text{ K}^{-1}$ for the CTE.

Further work in Japan is taking place with regards to interferometric techniques at elevated temperatures, including

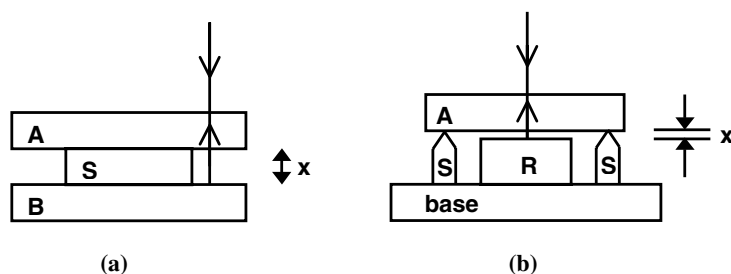


Figure 6. Typical arrangements for optical interferometers (a) with specimen, S, between two optical flats, A and B, (b) relative method employing reference sample, R. In both cases the increase in distance x is measured as a function of temperature.

work by the company Sinku Riko. For example Oikawa *et al* [61] have employed a modified Michelson interferometer to study the expansion of quartz up to 700 °C. Specifically this employs a dual path for a He–Ne laser beam—reflected twice from each mirror. A 10–15 mm long 4–7 mm diameter specimen with rounded ends was heated at 2 °C min⁻¹ in a furnace. Temperature was measured with a thermocouple near the specimen (the system was previously calibrated with a thermocouple in contact with a dummy specimen). The resolution reported was 2 nm, with a precision of 20 nm. Masuda *et al* [62] have estimated a sensitivity of 6 nm (1/100 of the wavelength of a He–Ne laser) for expansion measurement of corundum up to 727 °C. Their technique involved the formation of a Lissajous figure from the signals from horizontal and vertical polarized beams, which were initially 90° out of phase.

To combat any slight variations in the wavelength of laser light used for interferometry, Escalona and Rosi [63] have proposed a technique of frequency modulation continuous wave interferometry (FMCW) for thermal expansion measurement. In addition to the main interferometer measuring the specimen expansion, this method also employs a second interferometer which does not have the specimen in its path and thus acts as a reference unit.

Use of interferometry at higher temperatures yields numerous problems that include the effect of radiant light emitted from the specimen and the deterioration of the optical reflective surface with time. However innovative work by Miiller and Cezairliyan [64] in the USA has resulted in a special pulse heating technique being developed. Tubular specimens were heated by a pulse of electric current of duration less than one second. Temperature was monitored by a pyrometer and the expansion across the tube diameter recorded by a modified Michelson interferometer arrangement. A laser beam was reflected onto opposite sides of the specimen before recombination with a reference beam. Using this technique, results for the thermal expansion of niobium, molybdenum and tantalum above 2500 °C have been reported as well as tungsten up to 3300 °C.

A similar principle has been used by Righini *et al* [65–67]. Again a tubular specimen was heated by a short pulse of electric current and a pyrometer recorded temperature. However, in this case, interferometry was used to record the relative displacement of clamps attached to the *ends* of the specimen, which remained near ambient temperature. A computational technique then determined CTE from the total displacement and the longitudinal temperature profile of the specimen, which

was recorded at several intervals during the brief heating ramp. The thermal expansion of niobium to above 2000 °C has been studied with this method.

A device which combines aspects of push-rod dilatometry with optical interference has been proposed by Arndt *et al* [68], for thermal expansion measurement up to 2000 °C. A 6 mm square specimen with height 30 mm is heated by means of an electron beam furnace. A ceramic rod rests on top of the specimen and transmits the displacement to a mirror. A second mirror is mounted on top of a water-cooled Invar frame, the bottom of which is attached to the specimen stage. The whole of this is then enclosed in a vacuum chamber with a viewing window to allow a two or four beam laser interferometry system to measure the relative movement of the two mirrors. Whilst this system makes use of the high sensitivity of optical interferometry, it is not clear what error may be introduced by the ceramic rod, as its expansion is not compensated for.

3.2.3. Speckle pattern interferometry. When two coherent, collimated laser beams are directed onto a surface a special type of interference pattern is created on the surface itself. This is known as the ‘speckle’ effect. It has been shown [69] that this pattern changes when the surface on which it is created is deformed in its own plane. Lokberg *et al* [70] used speckle patterns to qualitatively study deformation on specimens up to 1700 °C. A TV camera recorded the patterns at different temperatures and subtracted the original pattern obtained at ambient temperature.

The measurement of thermal expansion coefficients by this technique has been reported by Kim *et al* [71]. In this work the pattern formed by two laser beams on the surface of an Inconel specimen was recorded by a CCD camera, while the specimen was heated by the passage of an electric current. At each temperature the pattern obtained from the specimen was digitally subtracted from the initial reference pattern. The fringes in the resulting pattern were then used to calculate the thermal expansion. Despite the experiment being carried out in air, expansion data was recorded up to 1200 °C.

3.2.4. Other optical techniques. Some other optical methods, which do not fit into the above categories, have been reported. A recently proposed technique by Vikram [72] aims to measure changes in the area of a sample as it expands thermally. The total amount of incident light scattered from a flat sample onto a photodetector was measured, and the CTE of a 25 mm square specimen of aluminium was accurately determined to 95 °C with this technique. By obscuring part of the specimen

area from the light source the sensitivity was increased, and procedures were suggested for corrections due to changes in specimen reflectance with temperature.

Fan *et al* [73] attached two thin blades to a specimen of stainless steel which was heated by an electric current at approximately $20\text{--}30\text{ }^{\circ}\text{C s}^{-1}$. The gap between the blades was narrow enough to form an optical slit which projected a diffraction pattern when illuminated with a laser source. Expansion of the specimen caused an increase in slit width, which altered the diffraction pattern. Measurement of the diffraction intensities with a photodetector allowed the CTE to be computed.

Another electrical pulse heating system developed by Cezairliyan [74] employed a rectangular aperture in front of an optical radiation source, with a pyrometer for measuring the intensity of radiation that passed through. A specimen in the form of a thin strip was then placed in front of the aperture so as to obscure the majority of radiation reaching the pyrometer. As the specimen was rapidly heated by the passage of a direct current, the increase in its width caused a further reduction in radiation from the source, and this change was used to calculate the amount of thermal expansion. To take account of radiation emitted from the specimen itself, a rotary chopper was used to rapidly turn the source radiation on and off at 600 Hz. In this way the specimen radiation could be measured and subtracted from the total intensity recorded.

In Austria, Pottlacher and Kaschnitz have also used an electrical pulse heating technique [75–78]. Here the specimens were in the form of 50 mm long wires up to 0.5 mm in diameter. These were heated by the discharge of a capacitor circuit at around 7000 V. Heating rates around 10^8 to 10^9 K s^{-1} were thus produced causing the specimen to be raised to a temperature of several thousand kelvin. Temperature was recorded by pyrometry and a high-speed CCD camera was used to record optical images of the specimens during heating. A variety of metallic materials have been investigated with this technique and density/thermal expansion data generated for the solid and liquid. In particular, results for a range of iron-nickel alloys [78] were compared directly to results from a conventional push-rod dilatometer, which employed much slower heating rates. Good agreement between the two techniques was reported, except for high-iron alloys, where the rapid heating appeared to suppress the BCC to FCC transition.

3.3. Diffraction techniques

Measurements of the crystal lattice parameter of a material at different temperatures can be used for calculation of thermal expansion coefficient. This can be effected by diffraction of waves from the crystal planes, and the bulk of these studies have been based on the diffraction of x-rays [79, 80]. The specimen, in the form of powder, polycrystalline wire or rotating crystal, is enclosed in a suitable furnace. A beam of monochromatic x-rays is then directed on to the specimen and the angles at which the beam is scattered recorded either by means of a cylindrical film surrounding the specimen (Debye–Scherrer camera) or by measuring the intensity of the beam as a function of angle using a diffractometer. The spacing of the crystal planes can then be calculated from the angle and wavelength using the Bragg relation. By repeating this

at a different temperature the expansion of the crystal lattice can thus be calculated. Some researchers [81] have reported measurements at temperatures approaching $1200\text{ }^{\circ}\text{C}$ with x-ray diffraction.

This technique is particularly suitable for small samples and where the material to be studied is very weak. For anisotropic materials the variation in expansion with different directions within the crystal lattice can also be determined [81–84]. A potential drawback is the fact that the technique does not directly measure the increase in lattice parameter, but rather relies on the subtraction of two separate measurements of this quantity. As these two measurements will be very close, the percentage error in the thermal expansion will be much greater than that for the lattice parameter. Accurate measurement of the temperature of the small sample and the elimination of temperature gradients is also difficult. Some workers have attempted to measure the temperature by performing diffraction on a reference material with known expansion characteristics [85], which can be mixed in with the material under investigation if it is in powder form.

It should also be noted that measurements of the expansion of the lattice parameter do not necessarily correspond to the macroscopic expansion of the bulk material. When more than one phase is present the macroscopic expansion may be due to changes in the *proportions* of phases present as well as changes in the crystal lattice of the phases as recorded by the x-ray pattern. When liquid is present then clearly no diffraction will take place from this phase due to the lack of a regular atomic structure. Furthermore, at high temperatures the macroscopic expansion will be influenced not only by increases in lattice parameter, but also by the increase in equilibrium vacancy concentration. For example, the difference between the thermal expansion coefficient of the bulk and that of the lattice has been estimated to be approximately $1.5 \times 10^{-6}\text{ K}^{-1}$ for copper at its melting point [86], which would represent a percentage error of around 6%.

A recently developed technique [87] utilizes rapid heating via an infrared spot heater, which is directed onto a polycrystalline specimen held in a specially designed stage. With this system it is claimed that only the specimen, and not the stage, heats up and expands, thereby maintaining better alignment of the specimen in the x-ray beam.

Some work has also been carried out with neutron diffraction [88, 89], which has the advantage of a greater depth of penetration compared to x-rays, so that a sample of $\sim 1\text{ cm}^3$ in size can be employed. Diffraction experiments have even been carried out with a 4.5 GeV beam of positrons from a synchrotron radiation source [90], in this case the thermal expansion of boron nitride was measured up to $1527\text{ }^{\circ}\text{C}$ under a hydrostatic pressure of 7 GPa.

Electron diffraction [91] has been used to study thermal expansion in the surface layers of crystalline materials, which can differ from that in the bulk lattice. Termed reflection high-energy electron diffraction (RHEED), the technique employs an electron beam at a glancing angle of around 3° to the surface, so that only the first few atomic layers are investigated.

While RHEED gives thermal expansion perpendicular to the surface, helium atom scattering [92] has been used for expansion measurements in directions parallel to the surface. The use of a beam of helium atoms results in scattering only

from the topmost layer of atoms in the surface, so that the resulting diffraction patterns relate to atomic spacings within this layer.

3.4. Other specialized techniques

A number of other systems, which do not fall into the above categories, have been employed for thermal expansion measurement, and these are described in this section.

Thermal expansion measurements at the National Physical Laboratory have been carried out using a miniature thermo-mechanical system termed ETMT [93]. In this set-up, metallic specimens having a cross-sectional area of a few mm² are heated by the passage of a dc electric current of up to 200 A. The specimen ends are maintained at ambient temperature so that a parabolic temperature profile develops along the specimen. The increase in length of the specimen is recorded as a function of temperature at the centre. The CTE can then be calculated by assuming it is a second order polynomial function of temperature and integrating the contributions to length extension from the parabolic temperature distribution of the specimen.

A Japanese patent [94] describes an electrical pulse heating technique for rapidly heating samples of conducting material. The thermal expansion is then measured with reference to the potential difference of voltage probes attached to the electrodes on the specimen ends.

Recently Cannaearts *et al* [95] and Igeta *et al* [96] have measured the expansion of gold wires by employing an atomic force microscope. As the heating of the wire is by an ac electrical current the technique is referred to as a *scanning Joule expansion microscope* (SJEM). In these particular cases the researchers were attempting to measure temperature from the expansion data, and the technique is limited at present to electronic components. However, the high sensitivity (some researchers [97,98] have quoted 1 pm (10⁻¹² m) for the resolution of vertical displacement) may give potential in the future for thermal expansion measurement.

Moelle *et al* [99] have calculated thermal expansion behaviour from measurements of specific heat capacity. This work was carried out for polycrystalline diamond over the temperature range from 27 to 577 °C and literature values of the bulk modulus and Grüneisen constant were also used in the calculation.

The capacitance of a parallel plate capacitor is critically dependent on the gap between the plates. Thus by creating a situation in which a thermally expanding specimen alters the gap between two plates it is possible to measure thermal expansion, and a number of such techniques have been developed. However, these have been aimed primarily at cryogenic temperatures where high sensitivities have been reported [100]. One example of high temperature use has been mentioned by Wassermann *et al* [101], who used a differential capacitance technique up to 1200 K for Ni-Ti shape memory alloys.

Electrical resistance strain gauges, bonded to specimen surfaces, have been employed to measure thermal expansion [102–104]. This method is relatively quick and, by arranging a number of gauges in various orientations, can be used to investigate expansion anisotropy [105]. However these studies have been restricted to low and near ambient temperature

ranges. Similar considerations apply to other surface mounted sensors, such as in-fibre Bragg gratings [106], which rely on the wavelength change of spectral peaks reflected from a white light source.

Chung *et al* [107] have employed an electrostatically levitated sample to measure the thermal expansion of nickel. The sample, a few mm in diameter, is first fully melted and then allowed to cool to solid from the fully molten state. CCD cameras record the dimensions of the drop and a pyrometer takes the temperature readings. By calculating the volume of the drop the density can be computed and hence an estimate made of the thermal expansion.

A number of techniques have been developed specifically for measurement of CTE of thin films of material deposited on a substrate, generally for the electronics industry. Reviewed by Wolff [108], these include such methods as measurement of the change in curvature with temperature of a specimen which comprises the film and substrate. Microfabricated cantilevers of films have been employed by Fang *et al* [109]. Measurement of the capacitance of the film has also been used [110], as well as spectroscopic ellipsometry [111], interferometry and diffraction methods.

Maznev *et al* have studied the time taken for thermal expansion to take place in response to a temperature increase [112]. A laser pulse, of the order of 10⁻¹³ s duration, was used to heat thin films of gold. The 'rise time' for the thermal expansion of the surface was then found to be of the order of 10⁻¹⁰ seconds.

Specific problems can be encountered in measuring the CTE of laminates because of the tendency towards specimen bending [113] as a result of the asymmetry of the structure (intentional or unintentional). For a mechanical dilatometer the bending will cause an error in the measured length of the sample, which can be corrected for if the curvature can be separately measured or estimated. In the case of a Michelson interferometer the bending will also cause the beams to become misaligned so that the recorded amplitude from the photodetectors can be used in a complex calculation of the error produced.

4. Standards

It would be appropriate at this stage to present a brief overview of a number of standards that exist for measurement of CTE, which are based on some of the experimental techniques described above.

For materials having a CTE above 5 × 10⁻⁶ K⁻¹, two ASTM standards describe mechanical dilatometer type techniques. The first of these, termed thermo-mechanical analysis [114], covers the range -120 to 600 °C and is aimed at specimens up to 10 mm in length, which are heated at a constant rate of 5 °C min⁻¹. Vitreous silica is used for the push rods and specimen holder. The mean coefficient of linear thermal expansion is then calculated from the plot of expansion versus temperature.

For higher temperatures and greater accuracy ASTM E 228-95 [2] describes a more detailed method, which employs specimens at least 25 mm in length. Push rods made out of vitreous silica enable measurement over the range -180 to 900 °C, and this can be extended to 1600 °C with alumina and

up to 2500 °C with graphite. In addition to the mean coefficient of linear thermal expansion over a temperature range, this method also allows for calculation of the ‘thermal expansivity’ at a particular temperature. An appendix also discusses the use of a differential dilatometer in conjunction with a reference material.

For highly accurate measurement of thermal expansion, especially for materials with CTE less than $5 \times 10^{-6} \text{ K}^{-1}$, ASTM E 289-99 [3] describes interferometric techniques for solids in the range -150 to 700 °C. Various geometries are possible for the specimen, which either has polished reflective ends or is placed between two flat reflecting surfaces. Interference of monochromatic light reflected from the opposite surfaces then creates a fringe pattern, changes in which can be monitored as the specimen is heated. Sophisticated instrumentation allows changes in length corresponding to a fraction of a fringe to be detected and this enables very small displacements to be recorded. Accuracy approaching $\pm 0.04 \times 10^{-6} \text{ K}^{-1}$ can thus be obtained for the CTE. When the specimen is maintained in a vacuum the technique is an absolute one and its accuracy of measurement is greatest. When an atmosphere is present, corrections are necessary for the change in wavelength of light. However the presence of an atmosphere assists heat transfer and is thus more favourable to accurate measurement and control of temperature. Two geometrical configurations are described in this standard: a Fizeau interferometer with a point light source and a Michelson interferometer employing a laser source, with either a single or double pass of the beam depending on the required accuracy.

A number of standards exist for thermal expansion measurement in ceramics and refractories. BS1902 has three techniques for refractory materials. The first is based on the horizontal push-rod dilatometer and employs a bar or rod shaped specimen up to 10 mm in diameter with silica push rods up to 1100 °C [115]. The second technique is a vertical dilatometer [116] usable to 1600 °C for larger specimens up to 50 mm across which have a central hole bored in them. In this case two concentric alumina tubes transfer the displacement from discs resting on the upper and lower surfaces of the specimen. Lastly, for testing investment casting shell moulds a recently added technique [39] can be used in which two alumina arms, connected to the specimen ends, protrude from the furnace at right angles to the direction of expansion measurement. One of the arms is fixed while the other is pivoted to act as a lever so that a linear variable differential transformer (LVDT) can measure a displacement. Expansion of the specimen can then be calculated with the aid of calibration data from a reference material.

BS EN 821-1 [117] deals with a dilatometer for testing monolithic ceramics up to 1500 °C. The specified accuracy in the CTE with this procedure can be $\pm 0.1 \times 10^{-6} \text{ K}^{-1}$ when measured over a 100 K temperature interval. The standard gives two configuration options: either as a conventional push-rod dilatometer with a single specimen, or as a differential dilatometer with a reference specimen. A detailed description of the specimen holder is provided, which incorporates a specially shaped test-piece support running on silica balls to minimize friction. Above 1000 °C alumina is the recommended material for constructing the apparatus.

Emphasis is also placed on recording the temperature distribution along the specimen by means of differential thermocouples. A similar procedure has been described in BS 7134 [118] for engineering ceramics. For the thermal expansion of glass a push-rod dilatometer is described in ISO 7991/BS 7030 [119].

5. Discussion

From consultations that the authors have had with a variety of industrialists it is clear that computer models of production processes for metals and alloys are continually becoming more sophisticated. There is a trend, for example, in models of the casting process to include the analysis of stresses and deformations that arise during cooling. Consequently the accuracy of these, and models of other high-temperature metallurgical processes such as forging, will be critically dependent on the provision of thermal expansion data at the appropriate temperature.

As there is a significant variation in thermal expansion behaviour with temperature for many materials, the use of room temperature CTE data in such models could lead to substantial errors, as could the application of values of the coefficient that do not have the appropriate definition. It is most likely that process models will employ some form of temperature increment or decrement δT over which the proportional increase $\delta L/L$ in a linear dimension of a grid element is calculated by an equation of the form

$$\frac{\delta L}{L} = \frac{1}{L} \frac{dL}{dT} \delta T \quad (11)$$

which is equivalent to

$$\frac{\delta L}{L} = \alpha_t \delta T. \quad (12)$$

The most appropriate definition for the coefficient of thermal expansion would thus be the true coefficient α_t as defined by equation (4). Care should thus be taken to either use data calculated on the basis of this definition or to convert values from other definitions to α_t .

It is recommended that data for CTE at high temperatures be taken from traceable sources such as the literature references in this paper or one of the commercially available databases, after first verifying the reliability of the data. A cautionary note might be appropriate at this point for anyone contemplating using electronic databases, especially those displayed on the World Wide Web. It is essential to fully investigate the reliability of the data, as it is all too easy for an individual or organization to set up a website with inappropriate data. One cannot deny the convenience of data in electronic format, especially with the opportunities that exist for effective searching of records in this format. However, it should be borne in mind that this information may not have been subjected to such a rigorous peer review process as, for example, data in scientific journals. It would be desirable if some form of system could be set up to accredit information on websites and other commercial databases. Indeed there may well be a role for learned societies or professional bodies to become involved in such activities in the future.

If thermal expansion data corresponding to the composition of the alloy and the required temperature range cannot be found in any reliable sources, then experimental measurement of the property will be required. Since new alloy compositions are continually being developed, it can therefore be anticipated that there will be a greater future need for thermal expansion measurement at high temperatures, especially those approaching and exceeding the solidus of the material concerned.

For each individual case consideration should be given as to whether the empirical conditions under which the data are generated are appropriate to the metallurgical conditions of the industrial process. Particular attention should be paid to phase changes in the material. In the vicinity of such transformations the dimensional changes will be influenced not just by the thermal expansion of the phases concerned but also by changes in the relative proportions of the phases. These in turn will be influenced by the thermal and mechanical history to which the material has been subjected. Indeed, different behaviour may be exhibited on heating and cooling, and the rate of change of temperature can also have an effect.

These issues have important implications for the future development of CTE measurement techniques. There may, for example, be an increased emphasis on methods that can apply a user-defined thermal cycle, which may involve rapid heating and/or cooling. This would have definite advantages over some of the techniques described earlier that require relatively slow temperature changes in order to achieve temperature uniformity within the specimen.

Extension of the upper temperature limits of the current range of the experimental techniques would also be of considerable benefit. Of particular use would be the development of empirical techniques for measurements above the solidus temperature in the partially molten condition. Great care would be required in this case to ensure that the microstructure of the sample under investigation corresponds as closely as possible to that present in the industrial process. Of the experimental techniques described in this report, the only method to receive extensive attention in the mushy zone is mechanical dilatometry. Since problems can occur with the weak nature of material containing a significant proportion of liquid, it would be desirable to develop a non-contact technique for the semi-solid case. As mentioned earlier, x-ray techniques for thermal expansion are unlikely to be appropriate, as diffraction does not occur from atoms in the amorphous liquid phase. The most convenient technique could thus be of the *optical* imaging type. This would have an advantage over optical *interferometry*, as it does not require part of the specimen surface to remain in an optically reflective condition at the extreme temperatures concerned. The optimum technique would probably involve a compromise between the desired goals of high temperature operation, speed of temperature change, accuracy of displacement measurement and specimen uniformity.

In conclusion it can be stated that there is likely to be a greater need for accurate thermal expansion data for metals and alloys at the elevated temperatures that correspond to conditions of industrial processing. Due to the relative scarcity of currently available data there will thus be a requirement for more empirical measurements of this property, by means of existing methods, modifications to these and also entirely new techniques.

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References

- [1] Touloukian Y S, Kirby R K, Taylor R E and Desai P D 1975 *Thermophysical Properties of Matter, Volume 12—Thermal Expansion, Metallic Elements and Alloys* (New York: IFI-Plenum) p 11a
- [2] American Society for Testing and Materials 1995 ASTM E 228-95
- [3] American Society for Testing and Materials 1999 ASTM E 289-99
- [4] Kirby R K 1992 *Compendium of Thermophysical Property Measurement Methods* vol 2, ed K D Maglic, A Cezairliyan and V E Peletsky (New York: Plenum) ch 19, pp 549–67
- [5] Barron T H K 1998 *Cindas Data Series on Material Properties, Volume 1-4—Thermal Expansion of Solids* ed R E Taylor (Materials Park, OH: ASM International) ch 1, p 5
- [6] Touloukian Y S, Kirby R K, Taylor R E and Desai P D 1975 *Thermophysical Properties of Matter, Volume 12—Thermal Expansion, Metallic Elements and Alloys* (New York: IFI-Plenum) p 4a
- [7] Touloukian Y S, Kirby R K, Taylor R E and Desai P D 1975 *Thermophysical Properties of Matter, Volume 12—Thermal Expansion, Metallic Elements and Alloys* (New York: IFI-Plenum) pp 77–8
- [8] Smithells C J 1976 *Metals Reference Book* 5th edn (London: Butterworth) pp 940–1
- [9] Waterman N A and Ashby M F 1997 *The Materials Selector* vol 2 (London: Chapman and Hall)
- [10] Robb C 1987 *Metals Databook* (London: Institute of Metals)
- [11] 1979 *Metals Handbook* 9th edn, vol 1 (ASM) pp 146–7
- [12] 1979 *Metals Handbook* 9th edn, vol 2 (ASM)
- [13] 1979 *Metals Handbook* 9th edn, vol 3 (ASM)
- [14] Touloukian Y S, Kirby R K, Taylor R E and Desai P D 1977 *Thermophysical Properties of Matter, Volume 13—Thermal Expansion, Nonmetallic Solids* (New York: IFI-Plenum)
- [15] Touloukian Y S, Kirby R K, Taylor R E and Desai P D 1975 *Thermophysical Properties of Matter, Volume 12—Thermal Expansion, Metallic Elements and Alloys* (New York: IFI-Plenum)
- [16] Touloukian Y S and Ho C Y (eds) 1975–7 *Thermophysical Properties of Matter* (New York: IFI-Plenum)
- [17] Touloukian Y S (ed) 1967 *Thermophysical Properties of High Temperature Solid Materials* vol 1–6 (New York: MacMillan)
- [18] 1999 Private communication with Purdue University, IN, USA
- [19] Wang K and Reeber R R 1998 *Mater Sci. Eng. R* **23** 101–37
- [20] The British Iron and Steel Research Association (ed) 1953 *Physical Constants of Some Commercial Steels at Elevated Temperatures* (London: Butterworth)
- [21] <http://www.matweb.com>

- [22] ILI Ltd, Index House, Ascot, Berkshire SL5 7EU, UK
- [23] Granta Design Ltd, Trumpington Mews, 40B High Street, Trumpington, Cambridge CB2 2LS, UK
- [24] Neuer G, Brandt R, Jaroma-Weiland G and Pflugfelder G 1989 *Int. J. Thermophys.* **10** 749–63
- [25] ESM Software, 2234 Wade Court, Hamilton, OH 45013, USA
- [26] JAHM Software, 103 Hill Street, Suite 9, Stoneham, MA 02180, USA
- [27] Friedrichs H A and Ronkow L W 1995 *Steel Res.* **66** 110–12
- [28] <http://www.gi.rwth-aachen.de/mebsp-b/main.html>
- [29] Gaal P S 1998 *Cindas Data Series on Material Properties, Volume I-4—Thermal Expansion of Solids* ed R E Taylor (Materials Park, OH: ASM International) pp 165–80
- [30] Morrell R 1999 Private communication
- [31] Valentich J 1979 *J. Mater. Sci.* **14** 371–8
- [32] Kikuchi Y, Sudo H and Kuzuu N 1997 *J. Appl. Phys.* **82** 4121–3
- [33] Valentich J 1977 *ISA Trans.* **16** 81–9
- [34] Badini C, Fino P, Mussob M and Dinardoc P 2000 *Mater. Chem. Phys.* **64** 247–55
- [35] Hurst E P II and Gaal P S 1999 *Thermal Conductivity 24—Thermal Expansion 12* ed P S Gaal and D A Apostolescu (Lancaster, PA: Technomic) pp 331–5
- [36] Upadhyaya A, Griffo A and German R M 1999 *Thermal Conductivity 24—Thermal Expansion 12* ed P S Gaal and D A Apostolescu (Lancaster, PA: Technomic) pp 393–404
- [37] Touloukian Y S, Kirby R K, Taylor R E and Desai P D 1975 *Thermophysical Properties of Matter, Volume 12—Thermal Expansion, Metallic Elements and Alloys* (New York: IFI-Plenum) p 18a
- [38] Bassily S F 1999 *US Patent* 5 936 165
- [39] 1992 *British Standard* 1902, section 5.14
1998 Amendment No 1
- [40] Gilmore J F and Lloyd C A 1990 *US Patent* 4 923 307
- [41] Chen H-B 1990 *GB Patent* 2 220 270 A
- [42] Shpil'rain E E, Yakimovitch K A and Mozgovoi A G 1992 *Compendium of Thermophysical Property Measurement Methods* vol 2, ed K D Maglic, A Cezairliyan and V E Peletsky (New York: Plenum) ch 21, pp 601–24
- [43] Sommer K-D and Poziemski J 1994 *Metrologia* **30** 665–8
- [44] Blumm J and Henderson J B 2000 *High Temp.—High Pressures* **32** 109–13
- [45] Blumm J, Henderson J B and Hagemann L 1998 *High Temp.—High Pressures* **30** 153–7
- [46] Morrell R and Quested P 1998 Dilatometric measurement of melting behaviour of metal alloys *NPL Report CMMT (A) 106* (Teddington: National Physical Laboratory)
- [47] Rothrock B D and Kirby R K 1967 *J. Res. Natl Bureau Standards C* **71** 85–91
- [48] Touloukian Y S, Kirby R K, Taylor R E and Desai P D 1975 *Thermophysical Properties of Matter, Volume 12—Thermal Expansion, Metallic Elements and Alloys* (New York: IFI-Plenum) p 19a
- [49] Taylor R E, Rothrock B D and Kirby R K 1998 *Cindas Data Series on Material Properties, Volume I-4—Thermal Expansion of Solids* ed R E Taylor (Materials Park, OH: ASM International) ch 9, pp 225–42
- [50] Taylor R E and Groot H 1980 *High Temp.—High Pressures* **12** 147–60
- [51] 1993 *Patent* SU 1768042
- [52] Wang H *et al* 1999 *Int. J. Thermophys.* **20** 743–53
- [53] Hahn T A 1998 *Cindas Data Series on Material Properties, Volume I-4—Thermal Expansion of Solids* ed R E Taylor (Materials Park, OH: ASM International) ch 6, pp 181–92
- [54] Ruffino G 1984 *Compendium of Thermophysical Property Measurement Methods* vol 1, ed K D Maglic, A Cezairliyan and V E Peletsky (New York: Plenum) ch 18, pp 689–706
- [55] Ruffino G 1992 *Compendium of Thermophysical Property Measurement Methods* vol 2, ed K D Maglic, A Cezairliyan and V E Peletsky (New York: Plenum) ch 20, pp 569–99
- [56] Bennett S J 1977 *J. Phys. E: Sci. Instrum.* **10** 525–30
- [57] Lewis A 1994 *Meas. Sci. Technol.* **5** 694–703
- [58] Suska J and Tschirmich J 1999 *Meas. Sci. Technol.* **10** N55–9
- [59] Okaji M and Yamada N 1999 *Thermal Conductivity 24—Thermal Expansion 12* ed P S Gaal and D A Apostolescu (Lancaster, PA: Technomic) pp 336–43
- [60] Okaji M, Yamada N and Moriyama H 2000 *Metrologia* **37** 165–71
- [61] Oikawa N, Maesono A and Tye R P 1999 *Thermal Conductivity 24 Thermal Expansion 12* ed P S Gaal and D A Apostolescu (Lancaster, PA: Technomic) pp 405–14
- [62] Masuda K, Erskine D and Anderson O L 2000 *Am. Mineral.* **85** 279–82
- [63] Escalona R and Rosi C 1996 *Proc. SPIE* **2730** 414–17
- [64] Müller A P and Cezairliyan A 1998 *Cindas Data Series on Material Properties, Vol I-4—Thermal Expansion of Solids* ed R E Taylor (Materials Park, OH: ASM International) ch 10, pp 243–68
- [65] Righini F, Rosso A and Cibrario A 1985 *High Temp.—High Pressure* **17** 153–60
- [66] Righini F, Roberts R B, Rosso A and Cresto P C 1986 *High Temp.—High Pressure* **18** 561–71
- [67] Righini F, Roberts R B and Rosso A 1986 *High Temp.—High Pressure* **18** 573–83
- [68] Arndt H, Heichler W, Leisering R and Schmidt G 1990 *Patent* DD 276 918 A1
- [69] Leendertz J A 1970 *J. Phys. E: Sci. Instrum.* **3** 214–18
- [70] Lokberg O J, Malmo J T and Slettemoen G A 1985 *Appl. Opt.* **24** 3167–72
- [71] Kim S, Kim J H, Lee J K and Jarng S S 1997 *J. Mater. Sci. Lett.* **16** 1753–6
- [72] Vikram C S 1997 *Meas. Sci. Technol.* **8** 1156–9
- [73] Fan H T, Shen C H, Liu Y and Wang J 1989 *Int. J. Thermophys.* **10** 1085–92
- [74] Cezairliyan A 1971 *Rev. Sci. Instrum.* **42** 540–1
- [75] Pottlacher G, Kaschnitz E and Jäger H 1993 *J. Non-Cryst. Solids* **156–158** 374–8
- [76] Beutl M, Pottlacher G and Jäger H 1994 *Int. J. Thermophys.* **15** 1323–31
- [77] Otter C, Pottlacher G and Jäger H 1996 *Int. J. Thermophys.* **17** 987–1000
- [78] Seifert A, Pottlacher G, Jäger H, Groboth G and Kaschnitz E 1998 *Phys. Chem.—Chem. Phys.* **102** 1266–71
- [79] McKinstry H A, Huang C Y and McKinstry S T 1998 *Cindas Data Series on Material Properties, Volume I-4—Thermal Expansion of Solids* ed R E Taylor (Materials Park, OH: ASM International) ch 7, pp 193–206
- [80] Touloukian Y S, Kirby R K, Taylor R E and Desai P D 1975 *Thermophysical Properties of Matter, Volume 12—Thermal Expansion, Metallic Elements and Alloys* (New York: IFI-Plenum) pp 21a–23a
- [81] Antii M-L, Babushkin O, Shen Z, Nygren M and Warren R 1999 *Key Eng. Mater.* **164–165** 279–82
- [82] Suzuki N *et al* 1998 *J. Alloys Compounds* **281** 77–80
- [83] Halvarsson M, Langer V and Vuorinen S 1995 *Surf. Coatings Technol.* **76–77** 358–62
- [84] Liu Q, An S and Qiu W 1999 *Solid State Ion.* **121** 61–5
- [85] Merryman R G and Kempter C P 1965 *J. Am. Ceram. Soc.* **48** 202–5
- [86] Touloukian Y S, Kirby R K, Taylor R E and Desai P D 1975 *Thermophysical Properties of Matter, Volume 12—Thermal Expansion, Metallic Elements and Alloys* (New York: IFI-Plenum) p 14a
- [87] Langelaan G and Saimoto S 1999 *Rev. Sci. Instrum.* **70** 3413–17
- [88] Bowman A L, Krikorian N H and Neveson N G 1972 *American Institute of Physics Conf. Proc. No 3—Thermal Expansion* (New York: American Institute of Physics) pp 119–30
- [89] Bittorf Ch, Matthies S, Priesmeyer H G and Wagner R 1999 *Intermetallics* **7** 251–8
- [90] Solozhenko V L and Peun T 1997 *J. Phys. Chem. Solids* **58** 1321–3
- [91] Zeng X and Elsayed-Ali H E 1999 *Surf. Sci.* **422** L977–82

- [92] Miyake T, Oodake I and Petek H 1999 *Surf. Sci.* **427–428** 39–43
- [93] Roebuck B, Gee M G, Brooks M, Cox D and Reed R 1999 *COMPASS 99—Proc. 1st Int. Conf. on Component Optimisation (Swansea)* ed W J Evans, R W Evans and M R Bache (Cradley Heath: Engineering Materials Advisory Services) pp 233–40
- [94] 1996 *Patent* JP 08128977
- [95] Cannaeerts M, Seynaeve E, Rens G, Volodin A and Van Haesendonck C 2000 *Appl. Surf. Sci.* **157** 308–13
- [96] Igeta M, Inoue T, Varesi J and Majumdar A 1999 *JSME Int. J. B* **42** 723–30
- [97] Majumdar A and Varesi J 1998 *J. Heat Transfer, Trans. ASME* **120** 297–305
- [98] Varesi J and Majumdar A 1998 *Appl. Phys. Lett.* **72** 37–9
- [99] Moelle C, Klose S, Szücs F, Fecht H J, Johnston C, Chalker P R and Werner M 1997 *Diamond Relat. Mater.* **6** 839–42
- [100] Swenson C A 1998 *Cindas Data Series on Material Properties, Volume I-4—Thermal Expansion of Solids* ed R E Taylor (Materials Park, OH: ASM International) ch 8, pp 207–24
- [101] Somsen Ch, Zahres H, Kastner J, Wassermann E F, Kakeshita T and Saburi T 1999 *Mater. Sci. Eng. A* **273–275** 310–14
- [102] Finke T E and Heberling T G 1978 *Exp. Mech.* **18** 155–8
- [103] Poore M W and Kesterson K 1978 *J. Testing Evaluation* **6** 98–102
- [104] Fox J N 1990 *Am. J. Phys.* **58** 875–7
- [105] Kinan A M 1973 *American Institute of Physics Conf. Proc.* No 17, pp 37–44
- [106] Yu-Lung L and Han-Sheng C 1998 *Meas. Sci. Technol.* **9** 1543–7
- [107] Chung S K, Thiessen D B and Rhim W K 1996 *Rev. Sci. Instrum.* **67** 3175–81
- [108] Wolff E G 1999 *Thermal Conductivity 24—Thermal Expansion 12* ed P S Gaal and D A Apostolescu (Lancaster, PA: Technomic) pp 368–87
- [109] Fang W, Tsai H-C and Lo C-Y 1999 *Sensors Actuators A* **77** 21–7
- [110] Snyder C R and Mopsik F I 1998 *Rev. Sci. Instrum.* **69** 3889–95
- [111] Rafla-Yuan H, Hichwa B P and Allen T H 1998 *J. Vac. Sci. Technol. A* **16** 3119–22
- [112] Maznev A A, Hohlfeld J and Gütde J 1997 *J. Appl. Phys.* **82** 5082–5
- [113] Wolff E G 1999 *Thermal Conductivity 24—Thermal Expansion 12* ed P S Gaal and D A Apostolescu (Lancaster, PA: Technomic) pp 351–67
- [114] American Society for Testing and Materials 1993 *ASTM E 831–93*
- [115] 1990 *British Standard* 1902, section 5.3
- [116] 1989 *British Standard* 1902, section 5.4
- [117] 1995 *British Standard* EN 821-1, part 1
- [118] 1990 *British Standard* 7134, section 4.1
- [119] 1988 *British Standard* 7030 (ISO 7991:1987)