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Review on the Methods for the Measurements of Thermal Expansion Coefficient

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Abstract: *The thermal expansion of solids is a basic physical property representing the dimensional change in a solid induced by a change in temperature. It is of technical importance as it determines the thermal stability of crystal. The thermal shock resistance of crystal depends on thermal expansion. In modern epitaxial device technology lattice mismatch is an important factor this related to thermal expansion behavior. In nuclear fuel technology, the thermal expansions a deciding factor in the choice of container The thermal expansion coefficient is related to several other physical properties. Knowledge of thermal expansion is necessary in experimental determination of temperature variation of elastic constant, refractive index, dielectric constant and photo elastic constants. Data on thermal expansion is required in conversion of C_p into C_v. This demand has led to the development of number of experimental techniques for measurement of thermal expansion of crystals. The various methods may be grouped as (1) Optical method (ii) Capacitance method (iii) Diffraction method (iv) and other methods. Several of these have been discussed by Krishnan (1958) Yates (1952)and Krishnan et al91977). Some of the important methods will be briefly discussed before describing the techniques*

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I. Introduction

The co efficient of thermal expansion coefficient of most of solid materials is in the range $[10 - 60 \times (10^{-6}\ k^{-1})$. the crystal sample used is generally a few mm thick. As thermal expansion is temperature dependent, measurement have to n=be over small temperature intervals of few degrees at a time. Thus as sensitivity ($\underline{\Delta}1/1$) of order of 10^{-4} to 10^{-6} is generally required. Some time for detecting small changes a higher sensitivity is required. This demand has led to the development of number of experimental techniques for measurement of thermal expansion of crystals. The various methods may be grouped as (1) Optical method (ii) Capacitance method (iii) Diffraction method (iv) and other methods. Several of these have been discussed by Krishnan (1958) Yates (1952)and Krishnan et al91977). Some of the important methods will be briefly discussed before describing the techniques.

II. Optical method

Most of the optical methods make use of the phenomenon of Interference. Fizau (1864, 1866) was first to setup an interferometer for the measurement of thermal expansion. The sample was a crystal block with one face polished. A glass plate was supported above this face. A beam of approximately monochromatic light travelled down the system and reflection occurred from the lower surface of the glass plate and upper surface of the specimen. When these two faces were at a small angle, the two reflected beams resulted in a set of straight fringes. From the shift of fringes thermal expansion can be calculated. Fizeau not only devised this method but made measurements on dozen of crystals. It should said to the credit of Fizeau that although several refined techniques have developed since his days a century ago, most of his data remained uncontradicted. Several modifications of Fizeau interferometers have appeared (Enck and co-workers, 1965; Kirham and Yates 1968; Waterhouse and Yates, 1968; Baily and Yates 1967; Pojur and Yates1970). The main difference between these versions and that of Fizeau the use of quartz or glass optical flats, use of partially metalized plates, use of three small pieces of the sample or a cylindrical sample, use of laser to give sharp fringes and the use of camera or other recording system.

Fraser and Hollis Hallett (1965) and Miencke and Graham (1975) employed a Fabry – perrot etalon for the measurement of thermal expansion. The specimen was in the form of a cylinder two inches long and one inch in diameter with an axial hole half inch in diameter. The ends were in the form of three feet with polished co-planar optically flat surfaces. This sample was sandwitched between two half silvered optical flats. A beam of monochromatic light passes through the etalon to the opposite side resulting in a fringe pattern. From a

continuous monitoring of the intensity of the central interference fringe as the etalon is warmed, the thermal expansion coeffienct can be obtained.

Kirby (1967) modified an Abbey Pulfrich interferometer for thermal expansion measurement. He devised a holder such that a large single crystal is held inside an etalon. The design of the holder is such that experiments can be done on the same crystal sample placed in different orientations in the etalon, thus enabling the measurement of thermal expansion in different directions. Kirby used his apparatus to measure the thermal expansion of a rutile crystal in different directions.

In yet another modification, Aurora et al., (1984) used a Jamin interferometer in conjunction with a laser for measuring the thermal expansion of some superionic conductors.

In a novel method, Heflinger et al., (1973) used double exposure holographic interferometry. Basically, this involves the superposition of holograms of a surface at two different temperatures. The expansion co-efficient is easily determined from the read-out photographs by a simple formula.

III. Capacitance methods

In this method a heterodyne oscillator is used. The plate separation of one capacitance is controlled by the sample thickness and the other capacitance is variable. When the sample expands, it results in the change in the capacitance of the first condenser which in turn alterts the frequency. Either the change in frequency Δf is measured, or the change in capacitance (Δc) in the second condenser necessary to restore the original frequency is measured. It can be shown that $2\Delta f/f = \Delta c/c = \Delta l/l$. hence the expansion coefficient can be calculated.

Bijl and Pullan (1955) were the first to use this method. Several capacitance dilatometers with minor modifications have been proposed. (Dheer and Surange, 1958: Madaiah and Graham, 1964).

A major refinement was introduced by Carr et al., (1964), who designed a three terminal capacitance cell. Two such capacitances are connected in a transformer bridge and the small changes in capacitance are measured by means of an AC bridge. The overall sensitivity of this method is very high ($\Delta l/l \sim 10^{-10}$). White (1965) has used this set-up for the measurement of thermal expansion of a number of solids at very low temperature. Jones and Richards (1973) discuss transducer designs for sensitive capacitance micrometry. Ema et al., (1975) have designed a capacitance dilometer with a sensitivity of $\Delta c/c \sim 10^{-7}$): they used this arrangement to measure the changes in the expansion coefficient of sodium nitrite through the transition.

IV. Diffraction Methods

Bragg's law of diffraction is

$$\mathbf{d}= \frac{\lambda}{2} sin\theta$$

Where $\lambda$the wavelength is θ is the angle of diffraction and d is interplanar spacing. This law provides the basis for determining lattice constant and thermal expansion. Achange in temperature of the crystal cause a change in the value of d, which is observed as a change in θ. Thus from change in the Bragg angle, the expansion coefficients can be obtained. However, actually, instead of finding changes in in he d value of the individual lines, the lattice constants are determined accurately by using all the available lines and thermal expansion is calculated from temperature variation of lattice constants.

The accurate determination of lattice parameters from x-ray diffraction is now highly developed field. Excellent treatment of this topic is available is available in number of treatises. (Klug and Alexander 1954; Pieser et al., 1955 Kaelble 1967). Single crystal methods are used rarely. The powder method is is more commonly used . anumber of cameras based on different geometries have been proposed. The geometries employed In powder cameras are (i) flat film camera (ii) cylindrical camera and (iii) focusing cameras.

Cameras with low temperature facilities have been designed by many workers and are also commercially available. The well known Unicam high temperature camera with 19 cm diameter has been employed by many workers for lattice parameter studies up to 100^0 c. the lattice parameter can be determined, generally, with an accuracy of ± 0.002 A.this accuracy has been enhanced in some cases by using special techniques (Kaelble 1967)..

V. Other Methods:

Nielsen and Leipold (1963) measured the thermal expansion of magnesium oxide with the help of tele-microscopes. A three inch long specimen was heated in an induction furnace. Through a window in the furnace, the displacement of the tip of the sample was measured with a tele microscope having a filer micrometer eye piece.

Mantysalo (1965) proposed an ultrasonic method for the measurement of thermal expansion. In this method an audio signal modulated a UHF signal. The amplitude variation of the UHF signal is traced on record. The output of the modulated oscillator produces standing waves in acrystal. When the crystal expands an oscillating curve of nthe ultrasonic attenuation is obtained as the function of temperature. The period of this

oscillation is given by $\Delta t=\lambda/2a\beta$ where (λ) is the ultrasonic wave length, (a) the length of the crystal and (β) the thermal expansion coefficient.

Jaakkola et al., (1968) designed a hydrostatic weighing apparatus for precision measurement of volumes. The solid to be studied formed one arm of a sensitive analytical balance. The suspendes solid was kept in an enclosure which was thermally insulated and vibration free. Further, a temperature control of 0.002 K was achieved. The heating was done by immersing the solid in a liquidwhose density is known. With this method the authors were able to detect the difference between the thermal expansion of NH_4Br and ND_4Br.

Foster and Finnie (1968) developed a method suitable for crystal with very small coefficients of expansion. This method employs a single frequency He-Ne laser. The beam from the laser is split. One beam passes through an interferometer which includes the sample. The laser length is modulated by a signal which modulates the output frequency. By mixing this with the unmodulated beam the change is calibrated in terms of the laser length. In this actual experiment, the laser length changes because of the expansion of the crystal. The expansion is obtained from the change in frequency combined with the calibration. Foster and Finnie used this method for measurement of thermal expansion of Invar.

Fused quartz dilatometers are simple and comparatively inexpensive. In a typical dilatometer the sample is enclosed in a fused quartz tube, with the end of the sample just projecting out of the tube. This end of the sample is in contact with a sensitive dial guage. The composite holder (quartz tube and sample) I placed in a heater and length changes and directly read on the dial gauge. Jansson and Sjoblom (1970) describe such a dilatometer which was used by them for the measurement of thermal expansion of some alkali sulphates.

Shrivastava and Joshi (1972) proposed a method for the measurement of thermal expansion from knowledge of a stress coefficient of electrical resistance. Two identical wires are used in the experiment, one is stretched and the other is heated. The change in resistance of both the wires is measured and the change in length of the heated wire is calculated in terms of the change in length due to stress. Obviously, this method is useful for conducting materials available in the form of wires.

A new technique for the thermal expansion of isotropic solids was proposed by Drotning (1979). This method utilizes Υ-ray attenuation to measure thermal expansion. A Υ-ray beam passes through the sample and is detected, with a scintillation detector. The change in attenuation with temperature is related to the change in length of the sample. The unique features of this method are that it is a non-contacting probe and it can be used for measuring the thermal expansion of a sample in the solid state as well as the molten state through the melting temperature.

VI. Comparison Of Methods

To compare the various methods, the optical method require large and well-polished crystals. The capacitance methods also require large crystals. Further, in both these methods, thermal expansion in different directions can be determined by making measurements in different directions. In the diffraction methods, very small crystals, in fact, small quantities of powder material are enough. Further, in the case of low symmetry crystals, temperature changes in a number of directions can be determined in a single experiment. Accuracy – wise, the three methods are comparable.

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