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An apparatus for the measurement of thermal expansion of solids at low temperatures

OM PRAKASH, ASHOK RAO and P N DHEER
Department of Physics and Astrophysics, University of Delhi, Delhi 110007, India

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Abstract. A dilatometer, using the three terminal capacitance technique, suitable for measurement of linear thermal expansion of solids in the temperature range 1·3–300 K is described. The dilatometer is designed such that the mounting system for the specimen does not undergo any significant changes in dimensions when the specimen is heated. The apparatus, therefore, yields in principle absolute values of α, the coefficient of linear thermal expansion. The performance of the apparatus has been checked by measurements on copper in the temperature range of 77–300 K. Some preliminary results on the behaviour of α for $Y_1Ba_2Cu_3O_{6\cdot9}$ compound in the vicinity of superconducting transition temperature, T_c are also described. The system can detect relative changes in length $\Delta l/l_0$ of about 10^{-8}. Attempts are being made to improve the sensitivity.

Keywords. Thermal expansion; high temperature superconductors; critical temperature; three terminal capacitance technique; mounting corrections.

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1. Introduction

Since the discovery of high temperature superconductivity in ceramic materials (Bednorz and Müller 1986; Wu *et al* 1987) there has been a renewed interest in the measurement of linear thermal expansion of solids at low temperatures. The primary aim of such studies is to see if these materials—like conventional superconductors—exhibit a discontinuity (Olsen and Rohrer 1957; White 1962) in the expansion coefficient at the superconducting transition temperature T_c. In addition, of course, such data provide useful information about the lattice dynamics of the solid.

A large number of techniques (Hoydoo *et al* 1991; Kisil *et al* 1989; Hahn 1970; Huzan *et al* 1961; Villar *et al* 1980; Browder and Ballard 1969; Tilford and Swenson 1972; Subrahmanyam and Subramanyam 1986) have been developed for the measurement of linear thermal expansion. The most widely used technique, however, is the three-terminal capacitance bridge technique developed by White (1961). A change in the length of the specimen, caused by heating, produces a change in the capacity of a parallel plate condenser which in turn is measured by the capacitance bridge. In addition to the detection limit of the bridge, the accuracy of the measurements depends on the capacitance of the condenser, noise in the circuit and corrections due to the expansion of the sample mounting system of dilatometer. The expansion of the sample mounting system Δl_m is specially important for measurement on high T_c materials since around liquid nitrogen temperature, the coefficient of thermal expansion (unless materials like invar are used in the dilatometer) is in general

quite large. As a result careful measurements of Δl_m are needed for obtaining reliable values for the expansion Δl of the sample. This work reports the design of a dilatometer in which there is no measurable correction term Δl_m. The only correction necessary is that due to the unavoidable linear expansion of the condenser plate mounted on the specimen. This correction term is however relatively small because of the small thickness of the plate. The performance of the dilatometer has been tested by measuring the linear thermal expansion of copper between 77 K and room temperature. Preliminary results on the behaviour of the expansion coefficient of high T_c compound $Y_1Ba_2Cu_3O_{6\cdot9}$ in the neighbourhood of T_c have also been obtained.

2. The dilatometer

The dilatometer, essentially a modification of one described by Dheer and Surange (1958) is shown in figure 1. Three thick brass rods A, are fixed symmetrically to the top of a brass can C. Two ebonite discs E_1 and E_2 are attached to the rods A as shown in the figure. One of the condenser plates P_1 is fixed to E_1 with the help of a brass bush B. The specimen S, whose thermal expansion is to be measured carries

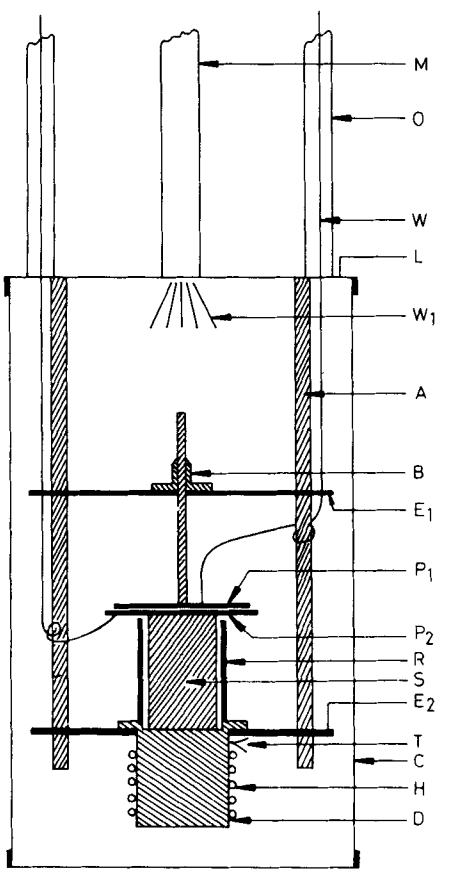

Figure 1. The dilatometer.

the second condenser plate P_2. S is attached (soldered or glued) to a copper base, D, which in turn is firmly screwed to the ebonite disc E_2.

A heating coil H and a thermometer T (a copper-constantan thermocouple or a doped Ge-resistor) are fixed on the copper base in order to facilitate quick replacement of the sample. A copper radiation shield R is soldered to D to ensure uniformity of the sample temperature.

The fixing of heating coil H and thermometer T on the base D, though quite satisfactory for metallic specimens, was found to be rather inconvenient for materials like the high T_c superconducting compound $Y_1Ba_2Cu_3O_y$ (which are relatively poor thermal conductors), mainly because of the long time required for attaining thermal equilibrium. For such materials it was found that the equilibrium time could be substantially reduced if H and T are attached directly to the specimen. It is also advisable to use two thermometers, one at each end, to ensure absence of any thermal gradients along the sample. In the present investigation the temperature difference between the two ends of the specimen, measured with a differential copper-constantan thermocouple, was always less than 5 mK.

The can assembly is suspended in a dewar containing liquid nitrogen (or liquid helium) with the help of tubes O and M. The central tube M is also used for pumping the system and for taking out electrical leads (W_1) from the heater and the thermometer. The leads (W) from the condenser plates P_1 and P_2 are taken out through the side tubes O.

The dilatometer is evacuated, filled with helium gas at low pressure and then cooled by filling the dewar with liquid nitrogen (or liquid helium). When the specimen attains the bath temperature, the dilatometer is evacuated to a pressure of about 10^{-6} Torr. Measurements of linear thermal expansion are then carried out by heating the specimen to successively higher temperatures T, and measuring the corresponding values of capacitance C between the plates P_1 and P_2.

Let (C_0, l_0) denote the capacitance C and the length of the specimen at liquid nitrogen (77 K) temperature. Let (C_T, l_T) denote the same quantities at temperature $T(>77\,\text{K})$. Assuming that P_1, P_2 form an ideal parallel-plate condenser of area A, it is easy to show that

$$\frac{\Delta l}{l_0} = \frac{l_T - l_0}{l_0} = \frac{\varepsilon_0 A}{l_0}\left(\frac{1}{C_T} - \frac{1}{C_0}\right) \tag{1a}$$

where ε_0 is the permitivity of free space. Thus measurement of C_T as a function of T enables determination of $\Delta l/l_0$ and hence that of α, the coefficient of linear thermal expansion, as a function of temperature.

If, as it sometimes happens, $\Delta C = C_T - C_0 \ll C_0$ expression (1a) can be approximated as

$$\frac{\Delta l}{l_0} = -\frac{\varepsilon_0 A}{l_0}\frac{\Delta C}{C^2}. \tag{1b}$$

3. Capacitance measurements

The capacitance C between the plates P_1 and P_2 is measured by using GR-1620A capacitance measuring assembly in three terminal configuration; the three terminals

being the two leads from the plates P_1, P_2 and the ground. In this configuration the bridge measures directly the capacity C (without any contribution of the lead capacitance). With $C \sim 100\,\text{pF}$ and $A \sim 20\,\text{cm}^2$ changes in capacitance $C \sim 10^{-4}\,\text{pF}$ could be detected. Substitution of these values in (1b) shows that the system has at present a sensitivity $\Delta l/l_0 \sim 10^{-8}$. It is however, hoped that the sensitivity can be further improved by reducing the noise level of the system.

4. Sources of errors

The average temperature of the brass rods A was found to increase by only 0·08 K when the specimen was heated to 300 K. Thus heating of the specimen causes relatively

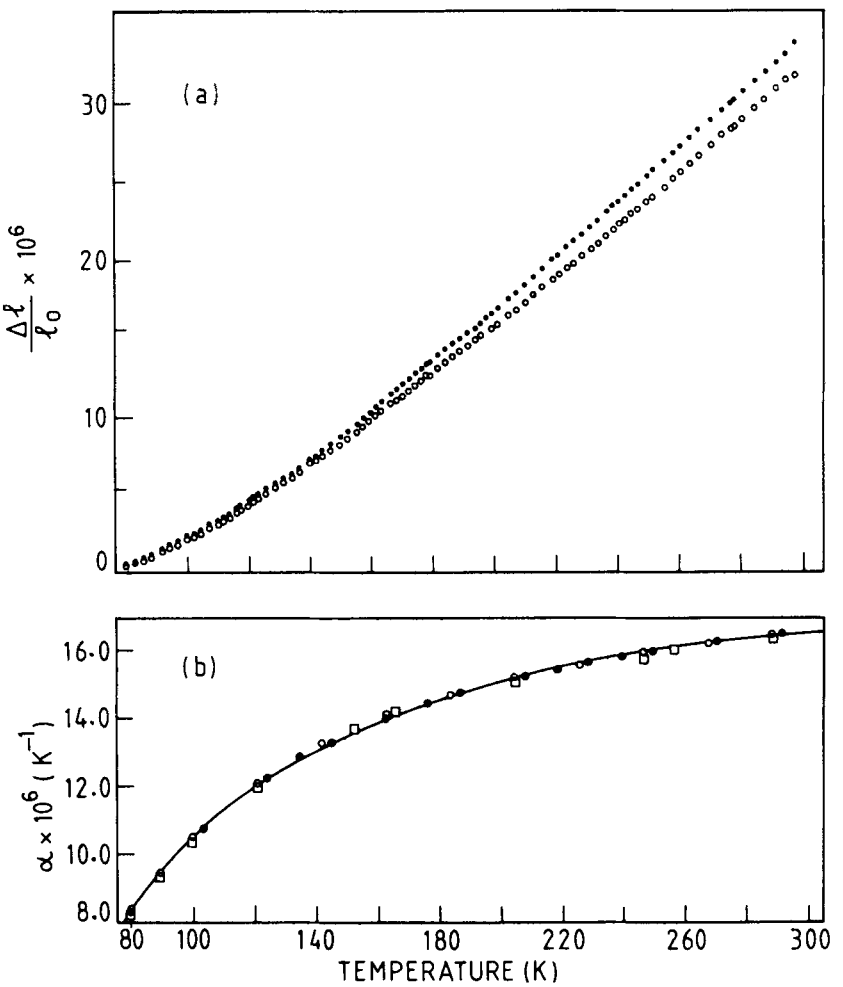

Figure 2(a). Temperature dependence of thermal strain $\Delta l/l_0$. ● Measured thermal strain; ○ after subtracting contribution due to expansion of condenser plate P_2 (thickness 2 mm) and buckling of supports. (**b**) temperature dependence of the linear thermal coefficient of expansion of copper. ———— Present work; ○, Kroeger and Swenson (1977); ●, Rubin *et al* (1954); □, Hahn (1970).

small changes in the temperature and hence the dimensions of these rods. The expansion of ebonite disc E_2 and copper base does not significantly affect the results since both are clamped at the upper end. Thus the sample mounting system does not undergo any significant changes in dimensions when the specimen is heated. However, errors in the measurement can still arise from (1) buckling of the disc E_2 (2) linear thermal expansion of condenser plate P_2, (3) non parallelism of P_1 and P_2, (4) surface irregularities of P_1 and P_2 and (5) edge effects.

The correction $\Delta l'$ due to (1) and (2) has been determined by removing the sample and fixing P_2 directly on D and measuring changes $\Delta C'$ in the capacitance as a function of temperature. It is satisfying to note that $\Delta l'$ thus determined agrees closely with that expected from the expansion of P_2 (i.e. buckling of D appears to be insignificant). For a thickness of P_2, about 1 mm, $\Delta l'$ is less than 3% of Δl for copper (see figure 2 where for the sake of clarity, results obtained for 2 mm thickness are shown). $\Delta l'$ is about 5% of Δl for type II superconductors.

A freshly cleaved thin mica sheet of about 0·1 mm thickness, was used to adjust the gap between the condenser plates P_1 and P_2. It was found that with this simple technique the departures from the parallelism were not more than 5%. The consequent

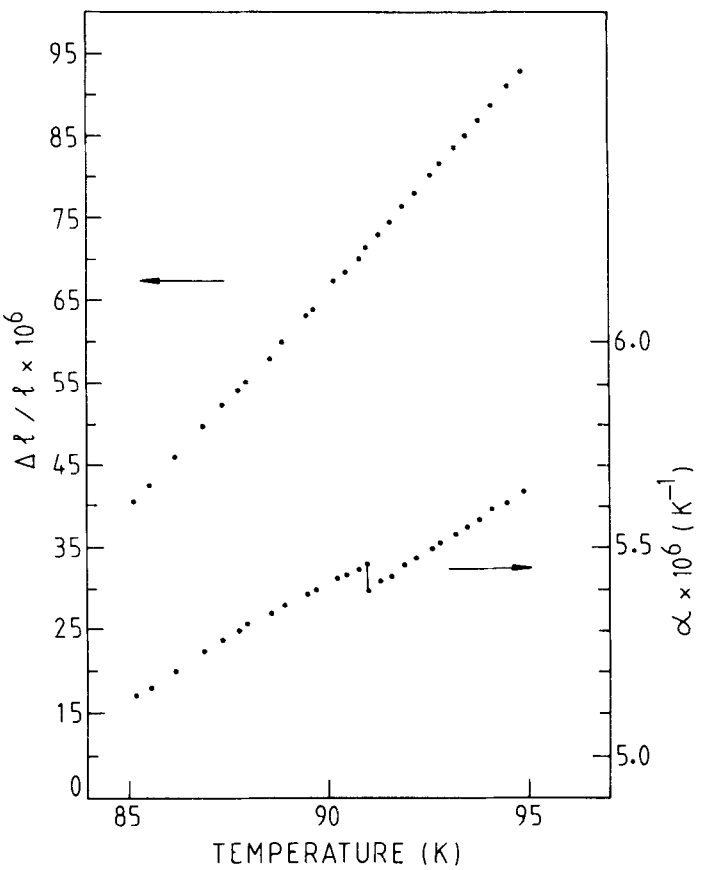

Figure 3. Thermal strain $\Delta l/l_0$ and linear thermal coefficient, α, of $Y_1Ba_2Cu_3O_{6\cdot9}$ in the vicinity of superconducting critical temperature T_c.

systematic errors in calculation of α are therefore not likely to exceed 0·01%. The condenser plates were finely polished and errors due to surface unevenness are estimated to be less than 0·3%.

It may be seen from figure 1 that the diameter of P_1 is kept slightly less than that of P_2. This has two advantages. First, the edge effects—due to the distortion of electric field near the periphery of the condenser plates—are considerably reduced and errors due to this can be assumed negligible. Secondly, since P_1 is always maintained at the bath temperature (by thermal anchoring of the electrical leads to rods A) the capacitance and hence the expansion data is unaffected by a real thermal expansion of P_2.

5. Performance of the dilatometer

The performance of the apparatus has been tested by the measurement of the linear thermal expansion of a copper rod (commercial grade) and the results are shown in figure 2. The figure also shows the results obtained by Kroeger and Swenson (1977), Rubin *et al* (1954) and Hahn (1970). It is evident that the present results are in good agreement with those reported earlier. Preliminary results have also been obtained on the behaviour of α, the coefficient of linear expansion of high T_c superconducting compound $Y_1Ba_2C_3O_{6\cdot9}$ in the vicinity of T_c. The results shown in figure 3 clearly demonstrate the existence of a jump $\Delta\alpha \sim 6\cdot0 \times 10^{-8}$ at the transition temperature. Detailed measurements of α in the temperature range $77 < T < 300$ K for this and other high T_c superconducting compounds are now being carried out and will be reported soon. Although the performance of the apparatus has been tested for $T > 77$ K, the dilatometer can be used without any modification, for measurements at lower temperatures.

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