CHAPTER 2

EXPERIMENTAL
2.1 Introduction

Over the years, several experimental methods have been developed to measure the thermal expansion of solids [1]. These can be classified into four groups based on the techniques used: mechanical (push rod dilatometer [2] and scissors dilatometer [3]), optical (interferometric [4-9] and twin-telemicroscopic [10-16]), diffraction (neutron [17] and X-ray [18-21]) and electrical (three terminal capacitance bridge [22-32], variable differential transformer [33]). In recent years the three terminal capacitance bridge technique has been more commonly used for the study of thermal expansion at low temperatures with a high sensitivity. This method was first developed by Professor G.K. White at the National Measurement Laboratory in Sydney, Australia [22]. The basic principle of this technique is to construct a parallel plate capacitor cell in which the sample serves as one of the plates of the cell and a reference material such as copper is used as the second plate. A change in sample dimensions changes the distance between the capacitor plates and the value of the capacitance, which can be measured accurately. Both the plates (terminals) are completely surrounded by a third terminal which is held at ground potential. The capacitance is measured using the ratio transformer bridge developed by Thompson [34]. The balance condition of the bridge is not affected by the capacitance of the parallel plates with respect to ground and by lead capacitances. Several modifications of the capacitance cell have been reported [27-32] to enable absolute measurement of thermal expansion. In all these designs the temperature of the sample is made to change and the other parts of the cell are kept at fixed temperatures.

A new cell design has been reported in the present work which is based on a suggestion by White [35]. The sample is directly placed in between the two plates
of a three terminal parallel plate capacitor, one of the plates of which is spring loaded so as to hold the sample in position between the two plates. A change in sample length alters the distance between the capacitor plates directly and changes its capacitance, which is measured. The details of the cell design are described in the next section.

2.2 Capacitance Cell

The cell design used in the present work is simple in construction and can be used for expansion measurements of small samples. The schematic cell design is shown in Fig.2.1. It consists of two capacitor plates (C1 and C2) made out of high purity oxygen-free copper. The plates are highly polished to get smooth and plane surfaces. C1 is rigidly fixed to the base B using three teflon rods (R). C2 is guided to move freely along the three teflon rods and it rests on three phosphor bronze springs (S1, S2 and S3). This end of the springs are electrically insulated and the other end of the springs are rigidly fixed to the base B. The sample which is insulated at both the ends using thin mylar foils can be inserted between the two plates C1 and C2. by slightly pushing C2 towards the base B. A copper chamber (can) C, encloses the plates and the sample. The sample is grounded so that the capacitance between the plates and the sample does not contribute to the parallel plate capacitance. The can C is also connected to the ground and it serves as the third terminal for the cell. This helps to minimize the effect of stray capacitances. The temperature is controlled by using a non-inductively wound heater H (resistance 25 ohms) around the can C and a platinum resistor sensor (PT103 from Lakeshore) attached to the bottom of the can C Another platinum resistor thermometer (PT2) is fixed on
Fig. 2.1: Schematic diagram of three-terminal capacitance cell used for thermal expansion measurements.
one of the capacitor plates and is used to monitor the sample temperature. Small depressions are made on the edge of the copper capacitor plates and the capacitor leads (ultra miniature co-axial cable of type C1 from Lakeshore cryotronics) are soldered strongly to the plates placing them inside the depressions. Special care has been taken to shield the leads to avoid noise pickups from any outside source.

### 2.3 Insert and Cryostat

The thermal expansion measurements in the temperature range 80-300K are carried out using a homemade experimental set up. The schematic block diagram of the cryostat and the insert are shown in Fig. 2.2. The cryostat arrangement consists of a liquid nitrogen double walled glass dewar and a glass chamber with a provision for evacuating the sample space through the vacuum port P. The glass chamber is fixed vertically on a steel flange (BF) using epoxy adhesive (araldite) which has a hole in its center and the diameter of the hole is same as the inner diameter of the glass chamber. The surfaces of both the walls in the annular space of the glass dewar are silvered to minimize heat radiation. The insert is made up of a thin walled seamless stainless steel tube (ST) with the capacitance cell (CC) attached to its one end. The other end of the tube comes out through the central hole of the top steel flange (TF) and is sealed using a teflon plug and araldite. The steel tube is fixed to the top flange vertically using araldite. The capacitance leads are taken out through the tube and through two small holes drilled in the teflon plug. Other electrical connections to the temperature controller, digital multimeter and current source are taken from the MS connector (MS 3102R-20-27P from Allied Electronics Corporation, Bombay). The MS connector is attached to the insert by holding it on
Fig. 2.2: Schematic block diagram of the cryostat and the insert used for thermal expansion measurements.
a small flange using four screws and an O'-ring and the small flange is connected to another steel tube which is also welded to the top flange of the insert. The electrical connections other than the capacitance leads from the cell are taken through the second tube and are connected to the MS connector. A small hole is drilled on the portion of the tube ST which lies inside the glass chamber to evacuate the inside of the cell. Fig.2.2 also shows the block diagram giving the interconnections of various measurement units.

2.4 Temperature and Capacitance Measurements

The glass chamber which houses the insert is first pumped through the port P and then liquid nitrogen (LN) is poured into the dewar. After waiting for about three hours for the insert to cool down to the liquid nitrogen bath temperature, the cell temperature is set to 78 K and controlled for half an hour before taking the first capacitance reading. The sample temperature is then scanned at the rate of 2 \( \frac{mK}{sec} \) using a Lakeshore model DRC91C temperature controller. Temperature values are recorded at the intervals of 4 sec using a Model 2500A automatic capacitance bridge from Andeen Hagerling. This bridge is designed to take three terminal capacitance measurement at a fixed frequency of 1 KHz with a true resolution of 0.5 attofarad (1 attofarad = \( 10^{-18} \) F). An average of fifty readings were taken to get the capacitance variation with temperature at 0.4 K intervals. To improve the quality of the data the bridge can be programmed to certain internal features. To avoid random noise peakup the bridge is programmed to sense the capacitance values at every 0.05 sec and average it out internally for 1 sec. To reject noise from external sources which generate signals at the same frequency and phase as the 1 KHz sine wave that the
instrument uses, the bridge is programmed to periodically alternate the applied signal to make it distinguishable from the synchronous noise signal. Also for rapidly changing capacitance values a track mode is used in which the bridge uses a high precision algorithm to take fast measurements. The measurement is carried out with an applied rms voltage of 15 volts on the capacitor plates.

The sample temperature measurement is carried out by sending a current of 1 mA through the PT2 sensor using a model 120A constant current source from Lakeshore Cryotronics and monitoring the voltage drop across the sensor using a model 195A digital multimeter from Keithley.

Mechanical vibrations generate a lot of scatter in the capacitance data due to microphonic effects. Care is taken to reduce those effects. The temperature setting, scanning and monitoring as well as capacitance measurements are fully automated using a 2AT6 computer. A program is written to automate the experiment which uses a IEEE-488 card.

### 2.5 Calibration and Standardisation of Data

For an ideal parallel plate capacitor the capacitance $C$ is given by, $C = \varepsilon A / L$, where $A$ is the area of the capacitor plate, $L$ is the distance between the two capacitor plates and $\varepsilon$ is the permittivity of the medium. If the medium does not change, then the change in the distance between the capacitor plates ($\Delta L$) is related to the change in capacitance ($\Delta C$) and the change in area ($\Delta A$) of the capacitor plates as,

$$\frac{\Delta L}{L} = \frac{\Delta A}{A} - \frac{\Delta C}{C} \quad (2.1)$$
In the present cell design the sample is inserted between the capacitor plates and the length of the sample at any temperature is equal to the distance between the two plates at that temperature. Hence the fractional length change of the sample at any temperature $T$ relative to the length at a reference temperature $T_0$ 
\[
\left( \frac{\Delta L(T)}{L(T_0)} \right) = \frac{L(T) - L(T_0)}{L(T_0)}
\]
is given in terms of the fractional area change, \([\Delta A(T)/A(T_0)]'\) of the copper capacitor plates of the capacitance cell whose thermal expansion is known, as:

\[
\left( \frac{\Delta L(T)}{L(T_0)} \right) = \left( \frac{\Delta A(T)}{A(T_0)} \right)^P - \left( \frac{\Delta C(T)}{C(T_0)} \right) = 2 \left( \frac{\Delta L(T)}{L(T_0)} \right)^{C_u} - \left( \frac{\Delta C(T)}{C(T_0)} \right) \tag{2.2}
\]

The fractional length change data of any sample can thus be obtained from the measured fractional capacitance change data \((\Delta C(T)/C(T_0))\) as a function of temperature.

Equ.(2.2) holds for an ideal parallel plate capacitor and requires that the plates be perfectly parallel and flat. However in practice it is not possible to get a perfectly smooth finish of the plate surfaces and there can be some small non-parallelism between the plates. To correct for these effects experimentally, we first measure the thermal expansion of a standard copper sample of cylindrical cross section of diameter 3 mm and length 3 mm and compare the experimentally observed fractional length change data with the reported standard data of Kroeger and Swenson [29] which are obtained using an absolute expansion cell. Fig.2.3 shows a plot of 
\[
\left( \frac{\Delta L(T)}{L(T_0)} \right)_{\text{reported}} \text{ versus } \left( \frac{\Delta L(T)}{L(T_0)} \right)_{\text{measured}}
\]
for the standard copper sample. The plot is fitted to a straight line

\[
\left( \frac{\Delta L(T)}{L(T_0)} \right)_{\text{standard}}^{C_u} = a \left( \frac{\Delta L(T)}{L(T_0)} \right)_{\text{measured}}^{C_u} + b
\]
Fig. 2.3: Experimental fractional length change data of a standard copper sample obtained using our cell is plotted with the standard fractional length change data of Cu given by Kroeger and Swenson [29]. The solid line shows the linear fit to the plot.
giving $a = 0.9367$ and $b = -1.8 \times 10^{-8}$. These values of $a$ and $b$ are taken to be the cell constants for our capacitance cell.

To test the performance of the cell, we have measured the thermal expansion of several other materials such as, aluminium (Al), silver (Ag), nickel (Ni) and iron (Fe) using our set up. These metal samples were annealed at 400° C for 24 hours to remove vacancies if any before recording the data. The experimentally observed thermal expansion data for these materials are found to be in excellent agreement with the data reported by other workers [1,36] and are compared in Fig.2.4a and Fig.2.4b.

2.6 Error Analysis

The resolution of the Model 2500A Amdoén Hagerling three terminal capacitance bridge is 0.5 aF as given by the manufacturer. However due to the imperfect shielding of the capacitor cables and the microphonic noises there are fluctuations in the measured capacitance values which are more than the ultimate resolution that the instrument can provide. To estimate the experimental error in the capacitance measurements repeated observations of the capacitance values are carried out for the capacitance cell maintaining it at a fixed temperature with a stability of ± 10 mK. The standard deviation obtained from the distribution of the observed data is $\sigma_C = \pm 1$ aF. This is then taken to be the error in the capacitance measurement.

Using the error propagation formulae [37], the error in the measurement of $\Delta C(T)/C(T_0) = (C(T) - C(T_0))/C(T_0)$ for a value of $C(T_0) = 5$ pF (typical value of the capacitance at $T_0 = 293$ K in our measurements) is $3 \times 10^{-7}$. From eqn.(2.2) the total error in $\Delta L(T)/L(T_0)$ comes from the error in the standard data of the
Fig. 2.4a: The fractional length change data obtained from our experiments for Ag and Al (open data points) are plotted with the data taken from literature (filled data points) [1].
Fig. 2.4b: The fractional length change data obtained from our experiments for Ni and Fe (open data points) are plotted with the data taken from literature (filled data points) [36].
Fig. 2.5: Linear thermal expansion coefficient \( \alpha \) obtained from the numerical three point differentiation of the measured fractional length change data obtained from our experiments for Al, Ag, Ni and Fe samples.
fractional length change for Cu reported by Kroeger and Swenson [29] and from the error in \( \Delta C(T)/C(T_0) \) measurement done in our bridge. Thus the error in the fractional length change \( \Delta L(T)/L(T_0) \) comes out to be \( 3.5 \times 10^{-7} \) from our experiments.

The coefficient of thermal expansion \( \alpha = \frac{d \ln L}{dT} \) is evaluated from the temperature dependence of the fractional length change \( \Delta L(T)/L(T_0) \) by numerical three point differentiation. Fig.2.5 shows the temperature dependence of \( \alpha \) for some of the test materials. The temperature stability of the cell is \( \pm 10 \text{ mK} \) and the experimental error in \( \alpha \) is calculated to be \( \sigma_\alpha = 4 \times 10^{-8} \text{ K}^{-1} \).
References


