

# High Sensitivity Technique for Measurement of Thin Film Out-of-Plane Expansion. II. Conducting and Semiconducting Samples

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**Abstract.** This paper describes the construction, calibration, and use of a precision capacitance-based metrology for the measurement of the thermal and hygrothermal (swelling) expansion of thin films. It is demonstrated that with this version of our capacitance cell, materials ranging in electronic properties from insulator to conductors can be measured. The results of measurements on *p*-type <100> oriented single crystal silicon are compared to the recommended standard reference values from the literature and are shown to be in excellent agreement.

## INTRODUCTION

It has been well recognized by the microelectronics industries that thermal stress modeling is critical for the prevention of device failure. The key parameter for this modeling is the coefficient of thermal expansion (CTE). For thin films (thicknesses of less than 100  $\mu\text{m}$ ) the standard test methods for characterization of the CTE involve one of two techniques: (1) the measurement of thick (>0.5 cm) samples - assuming that the thin films will behave identically to these "bulk-like" samples or (2) the measurement of the in-plane expansion of 1 cm strips of the thin films. The first method is known to have several problems, and the latter method is based upon two *a priori* assumptions: (1) that the material being measured is isotropic and (2) that the thin film can be handled for performing these measurements [1-6]. For many polymeric films produced by spin casting on a substrate, both of these assumptions are unfounded. In light of these limitations, the 1999 International Technology Roadmap for Semiconductors (ITRS) cites a need for the areas of assembly and packaging modeling of "[m]easurement, collection, and dissemination of materials properties of packaging materials for the sizes, thicknesses, and temperatures of interest." [7] In order to address this metrology limitation, we have developed a capacitance-based

technique for measurement of the coefficient of thermal expansion of films with thicknesses ranging from 2  $\mu\text{m}$  to 1 cm.

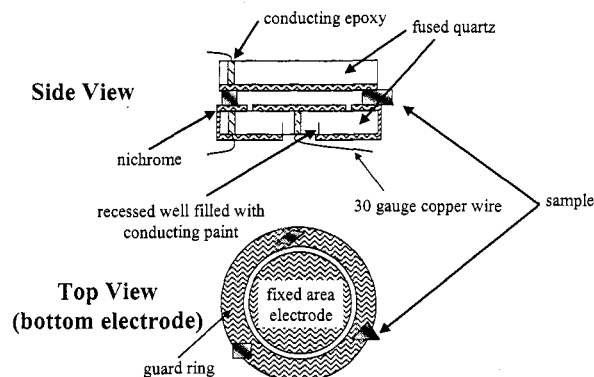
## EARLY ATTEMPTS AND CLAIMS

With high precision automated capacitance bridges, a relative resolution of the capacitance of 1 nF/F is easily obtainable. This fact combined with the simple relationship relating plate separation ( $d$ ) to capacitance ( $C$ ) for a parallel plate capacitor

$$C = \frac{\epsilon\epsilon_0 A}{d} \quad (1)$$

has driven many experimenters in the past to develop capacitance-based metrologies for determination of the coefficient of thermal expansion [8-10]. (In the above equation  $\epsilon$  is the dielectric constant of the medium separating the plates,  $\epsilon_0 = 8.854 \text{ pF/m}$  is the permittivity of free space, and  $A$  is the effective area of the electrode.) Several of the problems inherent in these previous attempts included: misalignment of the electrodes, expansion of the electrodes or the entire apparatus, specimen geometry requirements, stray capacitances and other fluctuations. Our earlier capacitance cell design eliminated almost all of these problems.

The previous design was based on a three terminal guarded electrode configuration shown in Fig. 1 [12-15]. It took advantage of the fact that a guarded electrode's electrical field lines are perpendicular to the electrode and therefore if the non-guarded electrode has a larger area, it is only the guarded electrode area ( $A$ ) that enters into Eqn. (1). Because of this, the appropriate choice for  $\epsilon$  is the dielectric constant of air, a well-defined quantity rather than the dielectric constant of the sample, which can display complicated behavior as a function of temperature or time due to phase transitions, physical aging, ion transport, *etc.* This earlier design was capable of measuring the coefficient of thin polymer films down to the order of  $5 \mu\text{m}$  with a relative reproducibility on thermal cycling of  $\pm 2 \mu\text{m}/\text{m}$ , excluding creep effects [11]. However, the design was limited to non-conducting samples, as a conducting sample would short the high terminal to ground.



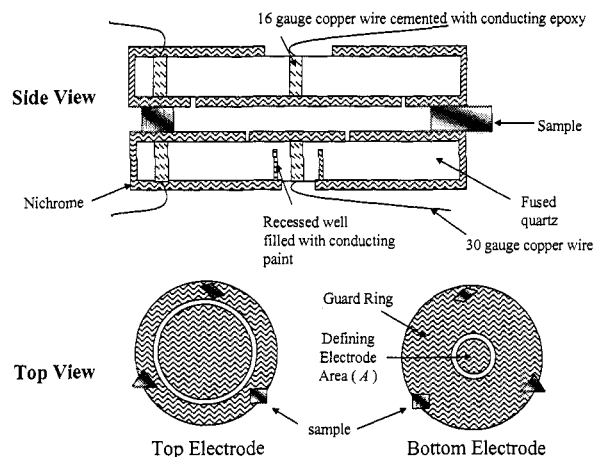
**FIGURE 1.** Schematic of previous design of the capacitance cell.

To verify the ability of the previous capacitance cell to provide accurate CTE values, measurements were attempted on single crystal silicon. However, after several attempts, it was realized that the silicon/nichrome interface was a Schottky junction and acted as both a voltage and frequency multiplier. The result was that the 1 kHz measurement frequency was multiplied into the ultrasonic region at which point the conducting epoxy contacts were shaken loose and the cell was damaged. Therefore, verification of the calibration was performed using single crystal  $\langle 0001 \rangle$  oriented  $\text{Al}_2\text{O}_3$ . The agreement between the experimental data and the recommended expansion values from the literature was within the experimental uncertainty of both data sets. A new design was conceived to enable the measurement of thin polymer films on silicon substrates.

## EXPERIMENTS AND RESULTS

### Cell Design

The new design involved several simple modifications of the original design. The lower electrode area was cut in half while the upper electrode had a guard ring added. Therefore, the effective area was still governed by the lower electrode and electrode alignment errors did not enter into the calculation. Furthermore, because the sample could now be held at ground potential, both semiconductors and conductors could now be measured. A detailed schematic of the capacitance cell is shown in Fig. 2. (Detailed information on the construction of this new version of the capacitance cell can be found elsewhere [16].)



**FIGURE 2.** Diagram of the capacitance cell design for measurement on conducting and semiconducting samples..

### Data Reduction

At this point, a brief presentation will be made of the data reduction procedure used for performing measurements with this capacitance cell. Measurements are typically performed in an environmental chamber so that the temperature ( $T_{\text{chamber}}$ ) and relative humidity ( $H$ ) can be determined. Additionally, a pressure sensor is used to determine the atmospheric (barometric) pressure ( $p_{\text{atm}}$ ). All of the data is passed through a digital Bessel filter described previously to eliminate time-lagged correlations between  $C$  and the intensive variables  $T$  and  $P$  [15].

The dielectric constant of air can be described through the molar polarization ( $P$ ) and the molar volume ( $v$ ) as

$$\varepsilon = \frac{2(P/\nu) + 1}{1 - (P/\nu)} \quad (2)$$

To obtain  $P$  in the above equation, we assume molar additivity of the molar polarizabilities, *i.e.*

$$\frac{P_T}{\nu} = \frac{P_w}{\nu_w} + \frac{P_{air}}{\nu_{air}} \quad (3)$$

where  $\nu_w$  and  $\nu_{air}$  are the molar volumes of water and air, respectively, and  $P_w$  and  $P_{air}$  are the molar polarizabilities of air and water vapor, given by [17]

$$P_{air} = 4.31601 \times 10^{-3} \text{ L/mol} \quad (4)$$

and

$$P_w = \frac{20.847 \text{ K L mol}^{-1}}{T} + 3.9 \times 10^{-3} \text{ L/mol} \quad (5)$$

It is clear that we have all the quantities necessary for the calculation of  $\varepsilon$ , except for  $\nu_w$  and  $\nu_{air}$ . To calculate the molar volume of dry air, we use the ideal gas law

$$\nu_{air} = \frac{RT}{P_{air}} \quad (6)$$

where  $R$  is the ideal gas constant,  $T$  is the temperature of the cell (different from  $T_{\text{chamber}}$ ) and  $p_{air}$  is the partial pressure of air. To convert  $H$  and  $T$  to the molar volume of water vapor ( $\nu_w$ ) several calculations need to be performed. The vapor pressure equation is used to obtain the saturation pressure of water ( $p_{\text{sat}}$ ) from  $T_{\text{chamber}}$  [18]. From this and the relative humidity  $H$ , the partial pressure of water ( $p_w$ ) can be obtained by

$$p_w = p_{\text{sat}} \times H \quad (7)$$

The partial pressure of air is then given by

$$p_{air} = p_{\text{atm}} - p_w \quad (8)$$

Because of the errors that would be introduced by using the ideal gas law for water vapor, more extensive calculations need to be performed to determine the molar volume of water. The compressibility factor ( $z = p\nu/RT$ ) can be obtained by a cubic fit to steam table data

$$z = \frac{p\nu}{RT} = AT^3 + BT^2 + CT + D, \quad (9)$$

where  $A = -1.026 \times 10^{-8} \text{ }^\circ\text{C}^{-3}$ ,  $B = -1.289 \times 10^{-7} \text{ }^\circ\text{C}^{-2}$ ,  $C = -3.382 \times 10^{-5} \text{ }^\circ\text{C}^{-1}$ , and  $D = 0.9998$  [15]. However, as  $z$  will increase towards unity as  $H$  decreases, the relative humidity corrected value for the compressibility factor needs to be used, which is given by Glasstone to be [19]

$$z' = 1 - H \cdot (1 - z) \quad (10)$$

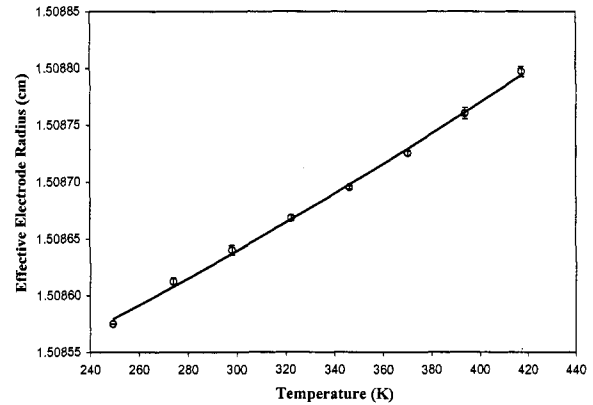
The molar volume of water is then given by

$$\nu_w = \frac{z' RT}{P_w} \quad (11)$$

From the above equations, the value for  $\varepsilon$  can be obtained for Eqn. (1). (The uncertainties in these calculations have been discussed elsewhere [15].) To determine  $d$  as a function of temperature (to calculate the CTE), the only parameter needed is  $A$  as a function of temperature.

## Calibration

The effective area of the electrode  $A$  must be calibrated as a function of temperature. To accomplish this, 3 pieces of Zerodur (a Schott material having a CTE of less than  $0.05 \text{ } \mu\text{m/m K}$ ) were measured over a temperature range of  $0 \text{ }^\circ\text{C}$  to  $150 \text{ }^\circ\text{C}$  [20]. The results of these measurements are shown in Fig. 3.

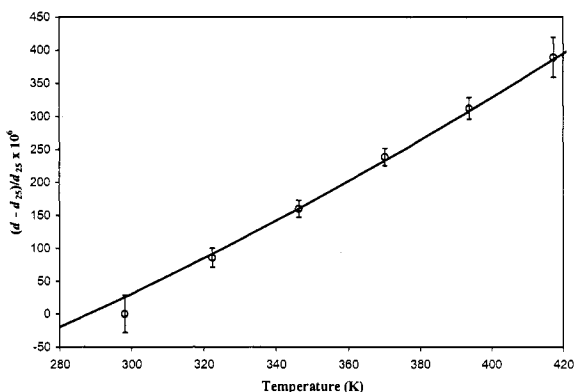


**FIGURE 3.** Effective electrode radius  $A$  as a function of temperature obtained by measurements performed on Zerodur samples. (For information on the error bars see Ref. 11.)

## Results on Single Crystal <100> Silicon

To verify the ability of the capacitance cell to (1) produce accurate CTE values and (2) to measure

silicon, measurements were performed on a 0.6 mm thick wafer of single-side polished, back side stress relieved, *p*-type, <100> oriented single crystal silicon with a resistivity of 15 Ω cm that was broken (by scribing) into three pieces. The results of these measurements are shown in Fig. 4, along with the recommended expansion data of Swenson for <100> single crystal silicon [21].



**FIGURE 4.** Expansion of single crystal <100> oriented silicon relative to its value at 25 °C ( $d_{25}$ ). The line corresponds to the recommended expansion data from the literature [21]. (For information on the error bars see Ref. 11.)

It is readily apparent that the two sets of data agree within the experimental uncertainty. To verify that measurements could be performed on conducting samples as well as silicon, a single measurement was taken on the silicon by connecting the braids from the high and low terminals together, shorting the two guard rings as if it were done by a metallic sample. The measured capacitance was unchanged, demonstrating that conducting materials could be measured.

The reproducibility for measurements performed under dry conditions was identical to those obtained using the previous version of the capacitance cell, *i.e.* on temperature cycling the relative reproducibility was on the order of 2 μm/m and for isothermal measurements the uncertainty was less than 0.1 μm/m. Because of this, it was assumed that the reproducibility under humid conditions would be identical to those calculated previously [15]. (*N.B.* A -90 °C dew point purge was utilized to guarantee dry conditions in the current set of experiments. This corresponds to a water vapor pressure of  $9.3 \times 10^{-3}$  Pa.)

## CONCLUSIONS

The design and implementation of the new capacitance cell for the measurement of conducting and semiconducting materials (as well as dielectrics) has been presented. Thermal expansion data, obtained with the new version of our capacitance cell, on <100> *p*-type doped single crystal silicon have demonstrated both the ability of the cell to measure silicon and conducting samples and the ability of the cell to provide accurate CTE data on these types of materials. It is apparent that this metrology can now be applied to thin polymer films deposited on silicon substrates. Furthermore, this cell can also be used to study the hygrothermal expansion (swelling due to the presence of moisture) by utilizing the data reduction techniques described earlier in Ref. 15. Accordingly, this technique should be especially useful to the microelectronics packaging industry for the characterization of inner layer (and inter-level) dielectrics as well as composite structures.

## ACKNOWLEDGMENTS

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