

AN IN-SITU MEASUREMENT TECHNIQUE FOR THROUGH-PLANE THERMAL PROPERTIES OF THIN DIELECTRIC FILMS

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In MCM-D applications, interlayer dielectrics separate and insulate metal conductors to form a three-dimensional interconnection structure. Due to the three-dimensional nature of these structures, the electrical and mechanical properties of the dielectric materials must be known in all directions for proper device design. The most commonly used polymer in microelectronics, polyimide, exists in formulations which have been shown to have a high degree of orientation and exhibit anisotropic properties.¹⁻¹⁰ Measurement of through-plane mechanical properties of thin films is difficult due to the high resolution required to measure the small thickness changes. Existing techniques require either stacked thin films or a single cast thick film of 100 micrometers or more to achieve dimensional changes large enough to be measurable.⁷⁻¹⁰ In addition, most existing techniques require removing a large area of film, if not the whole film, from a supporting substrate to perform the measurement. These techniques neglect the effects of the dielectric-substrate interaction, such as Poisson's effect and adhesive effects. Since most MCM-D structures utilize thin films adhered to a substrate, the measured through-plane CTEs from these other techniques may not reflect the true thermal expansion expected in real devices.

This work proposes two in-situ measurement techniques capable of measuring through-plane CTE of insulating thin films. The first technique is based on in-situ dielectric measurements utilizing two electrode geometries, a comb electrode structure and a parallel-plate capacitor structure. The second technique is based on ellipsometric measurements of very thin polymer films (one micrometer or less).

Dual Capacitor Measurement Technique

In the dual capacitor technique, a parallel plate capacitor and a comb electrode structure are fabricated on the same substrate. The lift-off fabrication sequence is listed in Table 1. The dielectric to be measured may be either a spin coated polymer, such as polyimide or benzocyclobutene (BCB), or an inorganic film, such as plasma enhanced chemical vapor deposited silicon dioxide.

Parallel plate capacitors are particularly useful for measurement of through-plane permittivity of thin dielectric films, given the capacitor area and material thickness. Conversely, the dielectric thickness can be determined if the permittivity and area are known.

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Comb electrode structures have been widely used as moisture and cure sensors for polymer films.¹¹⁻¹³ Comb electrodes can monitor moisture uptake and reaction products in the polymer film by measuring the dielectric properties of the film. This application of comb electrodes is marketed by Micromet Instruments, Inc. (Cambridge, MA) in their microdielectrometer. In this work, the dielectric monitoring capability of the comb electrode structure will be used to determine the permittivity changes in dielectric films with temperature. To extract the permittivity from electrical measurements of the comb electrode structure, a proprietary electric field finite element simulation model was obtained from Micromet.¹⁴ This electrical field simulation provides accurate analysis of the fringing fields in the comb electrode structure for situations when the dielectric thickness is at least twice the distance between the combs, i.e., for five micrometer comb spacing, at least ten micrometers of dielectric is required to prevent significant fringing into the air above the dielectric. The electrical fields produced by a comb electrode are highly dependent on the electrode geometry. To determine the ideal electrode geometry for monitoring small changes in permittivity of thin films, a number of simulation were run with the proprietary Micromet software. Taking into account fabrication considerations, a suitable electrode geometry has been selected, as shown in Figure 1.

Actual electrodes vary somewhat from this desired geometry due to fabrication inaccuracies, so the actual physical parameters are quantified for each individual electrode. The electrode geometrical parameters required as input parameters are the electrode metal thickness, the distance from the center of an electrode finger to the center of the nearest space, the thickness and the dielectric properties of the insulator (generally SiO₂) beneath the electrode, and the relative ratio of line width to space in the comb. The ratio of line width to spacing is limited by the use of a relatively coarse finite element grid above the comb electrode. To compensate for the coarse grid, measurement of the comb electrode in air can be used to calibrate the sensor by interpolating simulations for a dielectric with a permittivity of one with varying numbers of nodes. The hypothetical, fractional number of nodes can be determined from the measured electrical properties in air and can be used in later simulations to correctly determine the response of the comb electrode.

There are several inherent assumptions in the modeling of the comb electrodes. The first assumption, that of isotropic dielectric properties of the dielectric coating, is inherent to the simulation software. For this reason, the initial dielectric examined by this technique was chosen to be BCB, due to its low optical isotropy (birefringence > 0.002) which may indicate a lack of molecular orientation. Measurements using BCB in both parallel-plate capacitors (through-plane dielectric properties) and comb electrodes (a combination of in-plane and through-plane dielectric properties) have produced the same permittivity at room temperature. This result indicates that the dielectric properties of the film are isotropic, and the assumption of isotropic electrical properties is satisfied. The second main assumption is that the insulating film does not change significantly during heating, i.e., the thickness and dielectric properties of the underlying SiO₂ do not change with temperature. This assumption is valid for high quality oxides that exhibit very low CTEs (less than 1 ppm/°C) and little change in permittivity with temperature. The last assumption is that the electrode geometry does not change during the temperature cycling, except for the expansion of the dielectric film. The thin electrodes, good adhesion between the electrode and the underlying SiO₂, and low CTE of gold support this assumption over the temperature range from 25-200°C.

By placing the dual capacitor structure on a hot plate and varying the hot plate surface temperature, the effect of temperature on the dielectric film thickness in the through-plane direction is determined. By comparison of the simulation to the electrical measurements of the comb electrode structure, the permittivity as a function of temperature can be determined. A typical result for a BCB dielectric is shown in Figure 2, with data points from

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different temperature cycles represented by different shapes. Using the permittivity from the comb electrode simulation, the film thickness, t , (in meters) can be determined from the equation:

$$t = (\epsilon' \epsilon_0 A)/C \quad (1)$$

where ϵ' is the permittivity of the dielectric from the comb electrode simulation, ϵ_0 is the permittivity of free space (8.85×10^{-12} F/m), A is the parallel plate capacitor area in square meters, and C is the measured parallel plate capacitance in farads.¹⁵ A measurement of thickness as a function of temperature for a BCB dielectric is shown in Figure 3.

From the plot of thickness versus temperature, the through-plane coefficient of thermal expansion can be determined by the relation:

$$\text{CTE}[\text{ppm}/^\circ\text{C}] = (1/t_{25})(dt/dT) \times 10^6 \quad (2)$$

where t_{25} is the film thickness at 25°C and dt/dT is the slope of the plot in meter/°C. In general, the slope will be a constant over a given temperature range, provided that the measurement is not taking place near a physical transition of the film, such as a glass transition or a melting point. Performing this calculation for the BCB data gives a through-plane CTE of about 230 ppm/°C.

Ellipsometry Measurement Technique

Ellipsometry utilizes plane polarized laser light shown onto a thin film that is refracted through the film and reflected back to a detector as shown in Figure 4. The detector analyzes the phase shift and rotation of the polarization to determine the refractive index and thickness of the film. In this CTE measurement technique, a standard ellipsometer has been adapted to measure film thickness as a function of temperature by placing a heated metal stage beneath a dielectric coated wafer. The ellipsometer monitors the change in film thickness as the stage and wafer cool. An infrared temperature sensor is used to monitor the wafer temperature during the testing. The films measured by this technique must be about one micrometer in thickness to prevent optical harmonics from confusing the data interpretation.

Measurements using this technique have been performed using one micrometer thick BCB films. The thickness as a function of temperature is shown in Figure 5. Using Eq. 2, the through-plane CTE is calculated to be about 260 ppm/°C. This result is very similar to the value found with the dual capacitor technique.

Benzocyclobutene Through-Plane CTE Results

BCB is a thermosetting benzocyclobutene polymer, known for having a very low birefringence. From dielectric measurements using comb electrode and parallel plate capacitor structures, little dielectric anisotropy is observed. Therefore, since BCB exhibited isotropic optical and dielectric properties, isotropic mechanical properties were also assumed. However, the measured through-plane CTE indicates anisotropic mechanical properties. From the dual capacitor technique, the calculated through-plane coefficient of thermal expansion for BCB is about 230 ppm/°C. This result is in close agreement with the measured through-plane CTE of 260 ppm/°C obtained from the ellipsometry technique. However, while these two results are similar, they are very different from the literature value for in-plane CTE of 52 ppm/°C.¹⁶

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There are several possible explanations for this apparent anisotropy. One contribution to the higher through-plane CTE is Poisson's effect. In-situ films undergo changing stress states during temperature cycling. A typical stress-temperature plot for BCB is given in Figure 6. Because of this changing stress state, the film is placed under increasing tensile stress at lower temperature, stretching the polymer in the in-plane direction. By Poisson's effect, when the polymer is stretched in the in-plane direction, it is simultaneously thinned in the through-plane direction. Thus, a stressed polymer film at room temperature would be thinner than an unstressed film. However, from finite element mechanical simulations, this effect would increase the apparent through-plane CTE by only 30%. Therefore, Poisson's effect can account for part, but not all, of the observed anisotropy.

A second possible explanation is differences in the BCB films measured in the literature and in this work. The films characterized in the Dow product literature were thick films cast from monomer, while the films studied in this work were spin-cast from B-staged, partially polymerized BCB material. The spin-coat processing of the partially polymerized material may impart an orientation to the BCB crosslinked structure that is not observable in the dielectric and optical properties.

Conclusions

A dual capacitor technique and an ellipsometric technique have been developed for in-situ measurement of through-plane thermal expansion of thin dielectric films. The techniques are self-consistent for BCB films. BCB has been shown to have mechanical anisotropy in spite of optical and electrical isotropy. Possible causes of this effect are Poisson's effect and orientation due to spin-coat processing.

References

1. L. Lin and S.A. Bidstrup (1993). "Processing Effects on Optical Anisotropy in Spin-Coated Polyimide Films", *Journal of Applied Polymer Science*, 49, 1277-1289.
2. S. Herminghaus, et al. (1991). "Large anisotropy in optical properties of thin polyimide films of poly(p-phenylene biphenyltetracarboximide)", *Applied Physics Letters*, 59(9), 1043-1045.
3. D. Boese, et al. (1991). "Stiff Polyimides: Chain Orientation and Anisotropy of the Optical and Dielectric Properties of Thin Films", *MRS Symposium Proceedings Vol. 227*, 379-386.
4. S.T. Chen and H.H. Wagner (1993). "Out-of-Plane Thermal Expansion Coefficient of Biphenyldianhydride-Phenylenediamine Polyimide Film", *Journal of Electronic Materials*, 22(7), 797-799.
5. T.P. Russell, et al. (1983). "In-Plane Orientation of Polyimide", *Journal of Polymer Science: Polymer Physics Edition*, 21, 1745-1756.
6. N. Takahashi, et al. (1984). "Molecular Order in Condensed States of Semiflexible Poly(amic acid) and Polyimide", *Macromolecules*, 17, 2583-2588.
7. G. Elsner, et al. (1990). "Anisotropy of Thermal Expansion of Thin Polyimide Films", *Thin Solid Films*, 185(1), 189-197.
8. H.M. Tong, et al. (1991). "Thickness-direction coefficient of thermal expansion measurement of thin polymer films", *Rev. Sci. Instrum.*, 62(2), 422-430.
9. H.M. Tong, et al. (1991). "Thickness Direction Measurements of Polymer Thin Film Thermal Expansion Coefficients", *ANTEC Proceedings 1991*, 1727-1730.
10. M.J. Pottiger and J.C. Coburn (1992). "Out-of-Plane Expansion Measurements in Polyimide Films", *Polymers for Microelectronics; ACS Symposium Series*.
11. N.F. Sheppard, et al. (1982). "Microdielectrometry", *Sensors and Actuators*, 2, 263-274.
12. S.D. Senturia, et al. (1982). "In-Situ Measurement of the Properties of Curing Systems with Microdielectrometry", *Journal of Adhesion*, 15, 69-90.

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13. D.R. Day (1989). "Moisture Monitoring at the PI-SiO₂ Interface Using Microdielectric Sensors", Polyimides: Materials, Chemistry and Characterization, ed. by C. Feger, M.M. Khojasteh and J.E. McGrath, 537-548.
14. H.L. Lee, Optimization of a Resin Cure Sensor, M.S. Thesis, Massachusetts Institute of Technology, Cambridge, MA, 1982.
15. ASTM D150-92, "Standard Test Methods for A-C Loss Characteristics and Permittivity (Dielectric Constant) of Solid Electrical Insulating Materials".
16. P.E. Garrou, et al. (1993). "Rapid Thermal Curing of BCB Dielectric", IEEE Transactions on Components, Hybrids and Manufacturing Technology, 16(1), 46-52.

Table 1. Fabrication sequence for comb electrode structures.

Step	Purpose	Action
1	Ground Plane Metallization	Sputter Ti/Au/Ti
2	Isolate Ground Plane	PECVD Silicon Dioxide
3	Clean Surface	RCA; Bake @120°C for 10 min
4	Spin-Coat Photoresist	Microposit 1400-27 resist 500 rpm/6 sec; 5000 rpm/25 sec
5	Soft Bake Photoresist	95°C for 30 min
6	Expose Pattern	6.2 sec @ 435 nm, with 6 mW/cm ²
7	Resist Surface Hardening	Soak in chlorobenzene, 80 sec
8	Bake Hardening	95°C for 2 min
9	Develop Resist	14 sec in Shipley 354 developer
10	Metal Deposition	Evaporate 300A Ti/3000A Au
11	Lift Off Resist and Excess Metal	Soak in Acetone

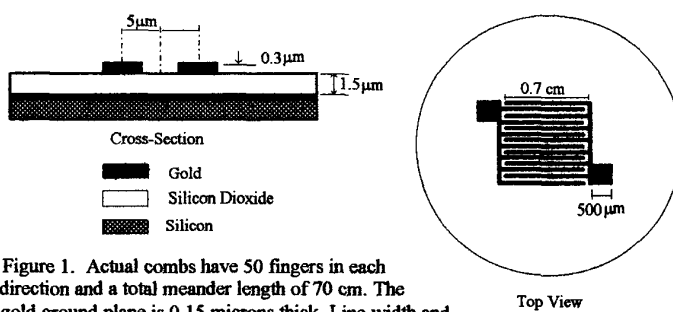


Figure 1. Actual combs have 50 fingers in each direction and a total meander length of 70 cm. The gold ground plane is 0.15 microns thick. Line width and spacing are each 5 microns. Drawings are not to scale.

AN IN-SITU MEASUREMENT TECHNIQUE FOR THROUGH-PLANE THERMAL PROPERTIES OF THIN DIELECTRIC FILMS
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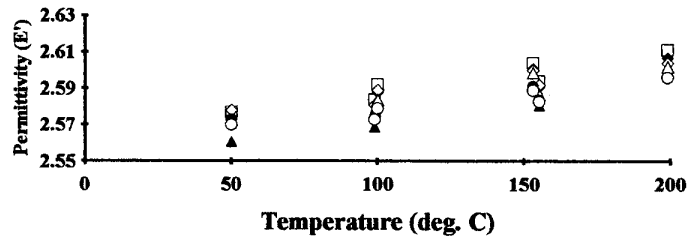


Figure 2. Permittivity as a function of temperature as determined from comb electrode measurements.

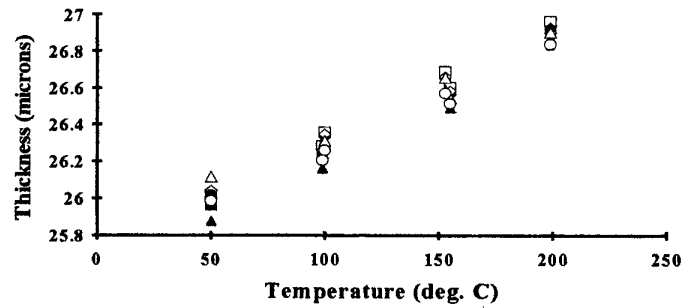


Figure 3. Film thickness as a function of temperature calculated from parallel plate capacitor data using the permittivity data given in Figure 2.

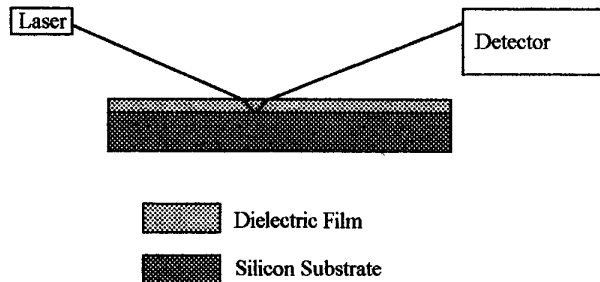


Figure 4. Set-up of ellipsometer measurement for thickness of thin films.

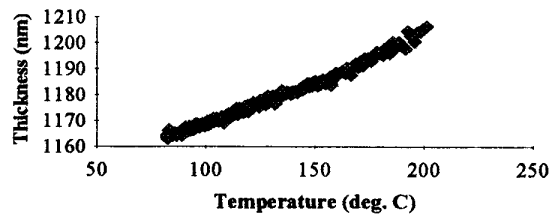


Figure 5. Typical data produced by ellipsometric through-plane CTE technique.