ANISOTROPY IN THERMAL, ELECTRICAL AND MECHANICAL PROPERTIES OF SPIN-COATED POLYMER DIELECTRICS

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ABSTRACT

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 thermal, electrical and mechanical properties of the dielectric materials must be known for all orientations in order to correctly design and simulate devices. 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 property 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-IO 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 coefficient of thermal expansion (CTE) from these other techniques may not reflect the true thermal expansion expected in real devices.

Similarly, direct measurement of in-plane dielectric properties of in-situ, thin polymer films is difficult due to the small cross-sectional area in the thickness direction. To date, all reported dielectric constants for polymer films in the in-plane direction have been calculated from Maxwell's relation from the optical birefringence of the film. This calculation of the in-plane permittivity may not hold if there are any dielectric loss modes that are present at electrical frequencies that are not present at optical frequencies.

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).

This work also presents an in-situ measurement technique to directly measure the in-plane dielectric constant of thin polymer films. This technique utilizes a comb electrode for obtaining a measurement of the combined electrical response of the polymer film in both the in-plane and through-plane directions. A finite element model has been developed to extract the in-plane component of the dielectric constant if the through-plane dielectric constant is known.

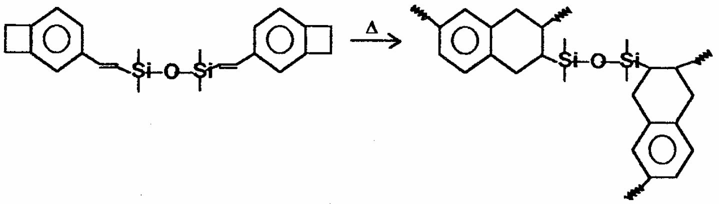
POLYMER DIELECTRIC MATERIALS

Divinyl siloxane benzocyclobutene (BCB, trade name: Cyclotene 3022, produced by Dow Chemical Co.) is a thermosetting, non-polyimide interlevel dielectric. The B-staged (partially reacted) BCB chains are suspended in mesitylene to provide a viscosity suitable for spin-coating. The benzocyclobutene reaction mechanism involves opening of the cyclobutene ring to form two sites that can then each react with other cyclobutene rings. II Since there is one benzocyclobutene group at each end of a chain segment, a total of four ~onds can be formed between each chain segment and other chain segments, thereby yielding a thermoset film, as shown in Figure 1 (a). Thermal cure of benzocyclobutene is performed at 2700C for one hour in a nitrogen purged furnace. BCB displays very low optical birefringence between the in-plane and through-plane directions and is generally considered to be completely isotropic due to its three-dimensional thermoset structure.

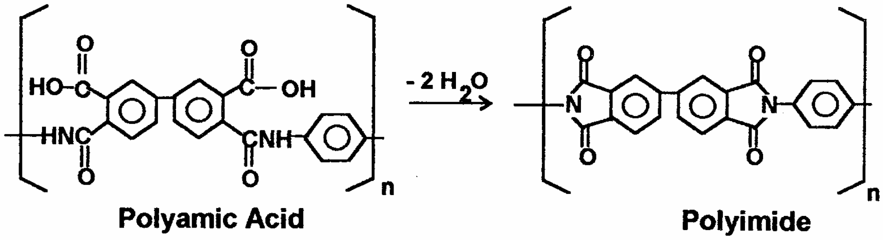
DuPont PI-2611 (biphenyl dianhydride-p-phenylenediamine or BPDA-PPD) is a polyamic acid-based polyimide that reacts via a condensation reaction mechanism upon thermal cure. The polymer is supplied as a B-staged polyamic acid solution in N-methyl pyrrolidone (NMP) solvent. The polyamic acid undergoes a ring closure imidization reaction during cure which produces water as a by-product as shown in Figure l(b).l2 BPDA-PPD polyimide is cured at 3500c for one hour in a nitrogen purged furnace. BPDA-PPD is a rigid rod polyimide system known for low residual stress. Also, BPDA-PPD has a high optical anisotropy as shown by the birefringence, between the in-plane and through-plane directions. [1]

Figure 1.

(a) Polymer Chain Structure for DVS-BCB



(b) Imidization Reaction of BPDA-PPD



THROUGH-PLANE CTE MEASUREMENTS

Dual Capacitor Measurement Technique

The dual capacitor techniques utilizes a parallel plate capacitor and a comb electrode fabricated on the same substrate with a polymer dielectric. The parallel plate capacitors are used 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. The comb electrode structure is used to determine the permittivity changes in dielectric films with temperature. By combining these two electrodes on the same substrate, the through-plane change in film thickness as a function of temperature can be determined.

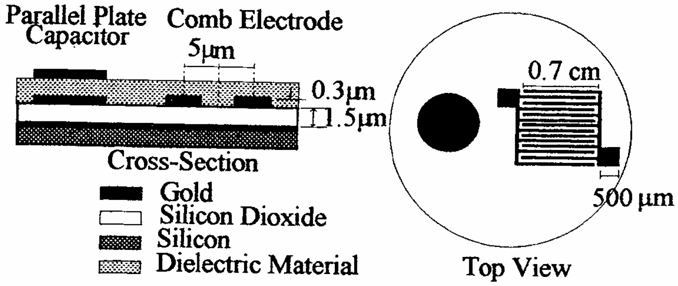
The lift-off fabrication sequence for the dual capacitor structures is listed in Table 1.

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^{o}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^{o}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^{o}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

To extract the permittivity from electrical measurements of the comb electrode structure, a proprietary electric field finite element simulation model was obtained from Micromet Instruments, Inc. (Cambridge, MA).[13] 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. Taking into account fabrication considerations, a suitable electrode geometry has been selected, as shown in Figure 2.

Figure 2. Actual combs have 50 fingers in each direction and a total meander length of 70 cm. The gold ground plane is 0.15 micrometers thick. Line width and spacing are each ten micrometers. The parallel plate capacitor diameter is 7 mm. Drawings are not to scale.



Actual electrodes vary somewhat from this desired geometry due to fabrication inaccuracies, so t the ctual physical parameters are quantified for each individual electrode. The input parameters to the modeling program are the electrode metal thickness, the distance from the center of an electrode finger t o to he center of the nearest space, the thickness and the dielectric properties of the insulator (generally I SiO_2 eneath 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 the dielectric properties of the polymer coating are isotropic, 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 indicates a lack of molecular orientation in the optical properties. 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. The second 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 CTE (less than I pprn/OC) and little change in permittivity with temperature. Another 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 25-2000C. 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 preliminary result for the capacitance as a function of temperature for a BCB dielectric is shown in Figure 3. Using the permittivity from the comb electrode simulation at room temperature, the film thickness, t, (in meters) can be determined from the equation:

$$t = (E' E_0 \mathbf{A})/\mathbf{C} \qquad (1)$$

where 8' is the pennittivity of the dielectric from the comb electrode simulation, 80 is the permittivity of free .space (8.85 x 10-12 F/m), A is the parallel plate capacitor area in square meters, and C is the measured parallel plate capacitance in farads, 14 Initially, the permittivity is assumed invariant with temperature and the value measured with the comb electrodes at *25OC* is used in equation (1). A measurement of thickness as a function of temperature for a BCB dielectric is shown in Figure 4, using the room temperature permittivity of the film.

Figure 3. Capacitance as a function of temperature as determined from parallel plate capacitor measurements.

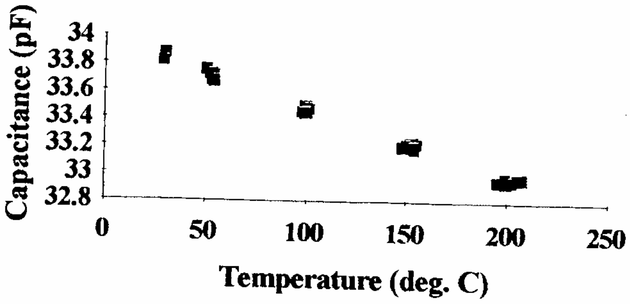
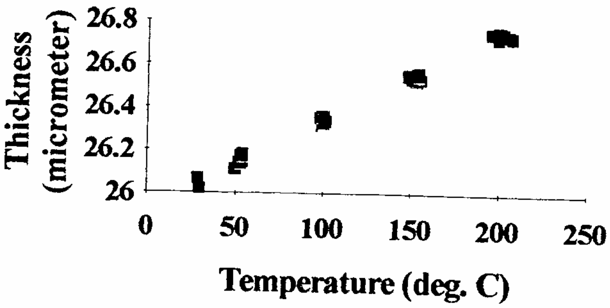


Figure 4. Film thickness as a function of temperature calculated from parallel plate capacitor data using the room temperature permittivity data.



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

$$\mathrm{CTE[ppm/^{o}C]} = (1/t_{25})(dt/dT) \times 10^{6} \qquad (2)$$

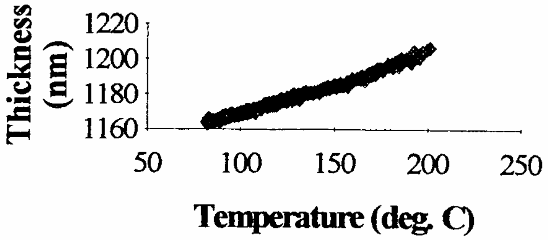
where t_{25} is the film thickness at 25°C and dt/dT is the slope of the plot in meter/°C. In general, the slope will be a constant over a given temperature range, provided that the measurement is not taking place near a physical transition of the film, such as a glass transition or a melting point. Performing this calculation for the BCB data gives a preliminary through-plane CTE of about 150 ppm/°C. Experiments are in progress to determine the permittivity as a function of temperature using the comb electrodes. An increasing or decreasing permittivity with temperature would lead to a corresponding increase or decrease in the through-plane CTE calculated using this technique.

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. 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. The refractive index of the air above the sample is assumed constant in the ellipsometry measurement. This assumption may not be valid due to density variations of the air caused by the heated surface. However, any changes would be expected to be small so this effect was not taken into account.

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 equation (2), the through-plane CTE is calculated to be around 200 to 250 ppm/°C.

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

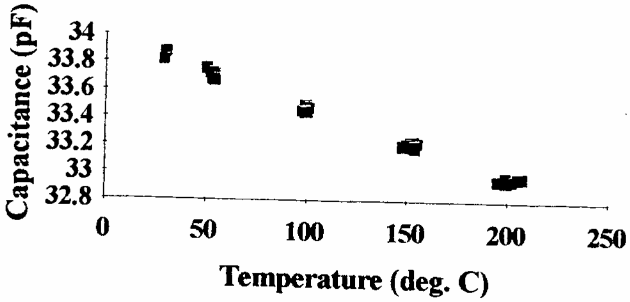


Benzocyclobutene Through-Plane CTE Results

BCB is a thermosetting benzocyclobutene polymer, known for having a very low birefringence.[11] 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. From the dual capacitor technique, the calculated through-plane coefficient of thermal expansion for BCB is about 150 ppm/°C. This result is significantly lower than the measured through-plane CTE of 200-250 ppm/°C obtained from the ellipsometry technique. However, while these two results are different, both measurements are larger than the literature value for in-plane CTE of 52 ppm/°C.[15]

There are several possible explanations for the difference in the measured through-plane coefficient of thermal expansion from the dual capacitor and ellipsometry techniques. The largest possible source of discrepancy is the assumption of constant dielectric properties for the underlying silicon dioxide layer and the dielectric film itself. The effect of temperature on dielectric constant should be larger for the polymer dielectric than for the inorganic silicon dioxide. Depending on the polarity of the polymer system the dielectric permittivity may either increase or decrease.[16] Work with other thermoset systems such as epoxy have shown an increasing permittivity as a function of temperature below the glass transition temperature.[17] If the permittivity does increase with temperature, the through-plane CTE calculated from the dual capacitor technique would indicate a higher value consistent with the ellipsometric measurements. Another source of possible error is the density fluctuations of the air above the surface of the film used in the ellipsometric technique and the associated differences in the refractive index of the air. Ideally the ellipsometric measurement should be run in a vacuum where the refractive index of the medium around the film is invariant. However, since air does change refractive index with temperature, the measurements may have a temperature dependent error in the thickness of the film. This result would lower the film thickness at the higher temperatures and lower the observed through-plane CTE of the film, bringing the result toward the measurement from the dual capacitor technique. The true through-plane coefficient of thermal expansion of BCB is bounded by these two measurements and should be within the range of 150-250 ppm/°C.

Figure 3. Capacitance as a function of temperature as determined from parallel plate capacitor measurements.



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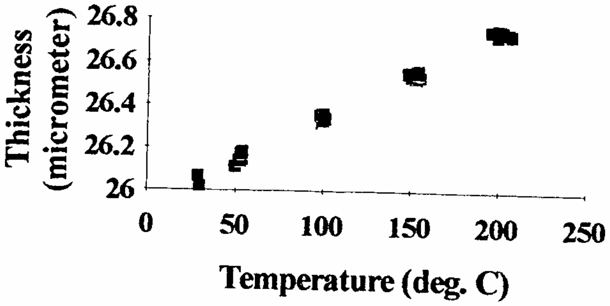

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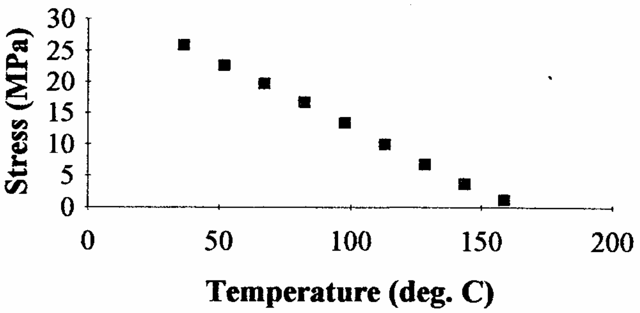
Independent of the actual value of the through-plane CTE, these measurements indicate a definite anisotropy in the mechanical properties of BCB. There are several possible explanations for this apparent anisotropy. One contribution to the higher through-plane CTE is Poisson's effect. In-situ films undergo changing stress states during temperature cycling. A typical in-plane 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 on a silicon wafer at room temperature would be thinner than an unstressed film released from a substrate surface.

Figure 6. Stress as a function of temperature for benzocyclobutene.



Finite element mechanical simulations have been performed to examine the magnitude of the Poisson's effect on the thickness of a BCB film undergoing heating. The system was modeled using ANSYS 4.4A from Swanson Analysis, Inc. The material parameters used for this modeling are shown in Table 2. The mechanical properties listed in Table 2 are for <100> silicon and free standing BCB films.[15,18] Due to limitations on the number of elements that can be modeled only a small portion of the four-inch diameter BCB coated silicon wafer can be modeled. The maximum number of available nodes allows a radius of 5000 μm for a 12.5° slice. Considering an isotropic BCB film with a CTE of 60 ppm/°C in both the in-plane and through-plane directions, the through-plane thickness change for an in-situ film is 115 ppm/°C. This demonstrates that the Poisson's effect is a substantial contribution to the apparent through-plane CTE of an in-situ film. However, compared to the measured value of 150-250 ppm/°C the modeled CTE is lower, thus, Poisson's effect can account for part, but not all, of the observed anisotropy.

Table 2. Parameters used in finite element mechanical analysis of BCB film on silicon substrate.

	Young's Modulus [GPa]	In-plane CTE, free film	Through-plane CTE, free film	Poisson's Ratio	Thickness [μm]
Silicon	180.5	5 ppm/°C	5 ppm/°C	0.3	525
BCB	2	60 ppm/°C	60 ppm/°C	0.33	30

A second possible explanation is differences in the BCB films measured in the literature and in this work. The films characterized in in-plane CTE measurements by Dow 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. Additionally, the spin-coated polymer may have a Poisson's ratio different from the 0.33 value obtained on the thicker films. Modeling the effect of BCB Poisson's ratio on

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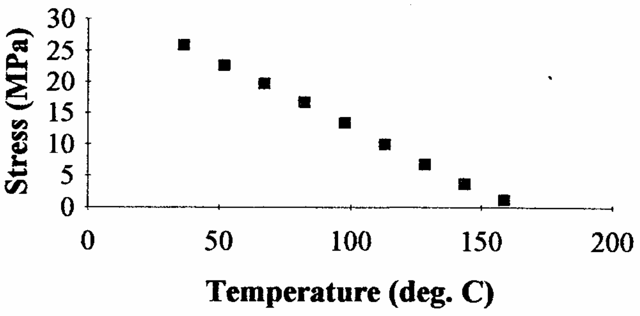

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IN-PLANE DIELECTRIC CONSTANT DETERMINATION

The dielectric properties of solid polymer films are typically determined from parallel plate capacitors which effectively give a measure of the through-plane dielectric response of the film. Due to geometrical limitations, an experimental technique to measure the in-plane dielectric properties has been lacking. An estimate of the in-plane dielectric constant may be obtained by applying the Maxwell relation relating the optical refractive index and the relative permittivity,

$$\varepsilon' = n^2 \tag{3}$$

where ε' is the relative permittivity and n is the refractive index.[19] The refractive index in the in-plane and through-plane direction can be determined by use of a prism coupler system.[20] Results of this measurement are shown in Table 3 for several spin-coated polyimide films. The through-plane dielectric constant is measured using a parallel plate capacitor geometry with the polymer film as the dielectric. These polyimides, all spin-coated from their respective polyamic acids, possess varying degrees of backbone rigidity and optical anisotropy. To estimate the dielectric anisotropy, the difference between the measured through-plane dielectric constant and that calculated from equation (3) is assumed to apply to the frequency dependence of the in-plane dielectric constant. The effect of backbone rigidity on the optical and electrical properties is evident in Table 3, as the predicted dielectric anisotropy ranges from 0.03 for the most flexible 6FDA/ODA polyimide to 0.83 for the most rigid BPDA/PPD polyimide. In using the Maxwell relation to estimate the electrical anisotropy, the frequency dependence of the in-plane dielectric constant is assumed equal to that of the through-plane dielectric constant. The validity of this assumption is questionable; therefore, an in-situ technique to obtain the in-plane dielectric constant of thin films is desired to determine the actual dielectric anisotropy.

Table 3. Effect of polyimide chemistry on the optical and electrical anisotropy of 5 μm thick film.

Polyimide (Dianhydride/Diamine)	Measured Refractive Index (Through-Plane)	Measured Refractive Index (In-Plane)	Measured Dielectric Constant (Through-Plane)	Predicted Dielectric Constant (In-Plane)
6FDA/ODA	1.58	1.59	2.93	2.96
BTDA/ODA-MPD	1.67	1.69	3.13	3.20
PMDA/ODA	1.64	1.73	3.07	3.37
BPDA/PPD	1.61	1.85	3.04	3.87

Finite Element Modeling

Finite element analysis is utilized to solve for the electrostatic potential distribution in a dielectric[21], in order to determine the effect of electrode geometry and anisotropic material properties on the capacitance of the dielectric under an applied potential difference. Using direct heat conduction analogies, ANSYS is used to calculate the capacitance of the dielectric utilizing Laplace's equation. The capability of ANSYS to accept direction-dependent material properties is useful for modeling spin-coated polyimide films with anisotropic dielectric properties. The boundary conditions and model layout used for this simulation are depicted in Figure 7. The input geometric parameters for this model are the film thickness, the electrode spacing and the electrode width. The required material properties for this model are the dielectric constant of air and silicon dioxide (both assumed isotropic) and the in-plane and through-plane dielectric constant of the polymer film. Using this model, charge on electrode 1, Q_1, is determined for a set potential on electrode 2, Φ_2. Using these values, the capacitance, C, of the system can be determined by the relation,

$$C = \frac{Q_1}{\Phi_2} \qquad (4)$$

By varying the in-plane dielectric constant input into the model, the correct value is determined when the calculated capacitance is equal to the experimentally measured capacitance.

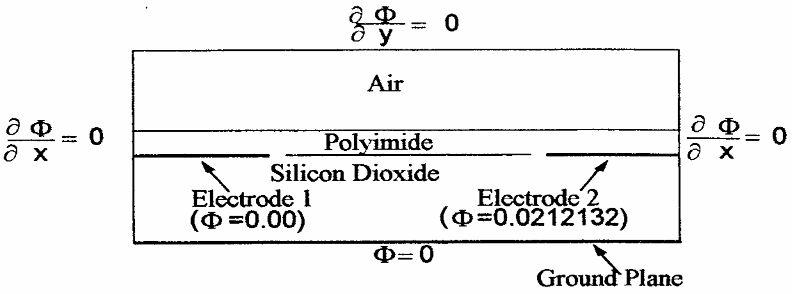

Figure 7. Regions included in the ANSYS model of IDE with ground plane.

Results

Two interdigitated electrode (IDE) designs similar to that described in Figure 1 were used in the testing: One with 10μm line/10μm spacing and the other with 10μm line/6μm spacing, designated as IDE A and B, respectively. The capacitance for IDE A and B with no dielectric film have been measured and are listed in Table 4. The magnitude of the capacitance is greater for IDE B than IDE A due to the narrower spaces between the electrodes. Using the model and assuming the in-plane and through-plane dielectric constant of 1 for the air, the response of the electrode model is also shown in Table 4. The predicted capacitance of the electrodes closely matches the experimentally measured capacitance within the scatter of the experimental data.

Table 4 Capacitance of IDE with air dielectric.

	C of IDE A (pF)	C of IDE B (pF)
Calculated	0.64	1.29
Measured	0.63±0.03	1.20±0.06

The capacitances of IDE A and B have been determined with a 4.9μm film of BPDA/PPD polyimide have been measured and are listed in Table 5. The through-plane permittivity used in the model, 3.05, was experimentally determined from a series of parallel plate capacitors. Using this data, from IDE A the in-plane dielectric constant is 4.07+/-0.26 and from IDE B data, the in-plane dielectric constant is 3.84+/-0.19. These results indicated 33% and 26% dielectric anisotropy, respectively, which compares favorably with the 27% anisotropy previously predicted from the Maxwell relation (Table 3). These results confirm that there is a significant anisotropy in electric properties for DuPont PI-2611 polyimide.

Table 5 Capacitance experimentally measured at 100kHz for IDE with 4.9mm of BPDA/PPD polyimide. Calculated in-plane dielectric constant from finite element modeling.

	IDE A	IDE B
Measured C (pF)	1.96 +/- 0.14	3.95 +/- 0.14
In-Plane Dielectric Constant	4.07 +/- 0.26	3.84 +/- 0.19
% Anisotropy	33%	26%

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. BCB appears to have mechanical anisotropy in spite of optical and electrical isotropy. Possible causes of this effect are Poisson's effect and structural orientation during processing.

Comb electrodes have been shown to be capable of direct measurement of in-plane dielectric properties of thin polymer films. Finite element modeling is used to extract the in-plane portion of the dielectric response of the comb electrode capacitance. Measurements of comb electrodes with air as the dielectric closely agree with the model. In-plane dielectric constant measurements of BPDA/PPD polyimide using this technique indicate that Maxwell's relation is a valid relation for this polyimide system.

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