Fabrication of an interdigitated sample holder for dielectric spectroscopy of thin films

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Abstract: A planar interdigitated sample substrate has been designed to support a thin-film sample of a polymer while the frequency-dependent dielectric properties of the thin film are measured. Trenches for the electrodes were etched into a SiO₂/Si wafer surface. Chromium was used as an adhesion layer prior to thermal evaporation of copper for the body of the electrode. The device was placed in a standard probe station and the dielectric character was recorded as a function of frequency with an impedance analyser. Devices with 20 to 70 fingers were measured and the results compared to analytical and finite element simulations. At 1 kHz, the total capacitance of a typical 20-finger device was 8 pF. The capacitive contribution of the thin film due to the fringing field in the polymer was about 2% of the total capacitance of the fabricated structures.

1. Introduction

Dielectric spectroscopy may be used to characterize dielectric material character. For bulk samples, the material is generally clamped between planar electrodes and the dielectric character is extracted from measurements of the capacitance [1]. Despite considerable interest in thin film samples for a range of technology applications [2], these samples are not well-suited to the mechanical loading associated with this approach. The dielectric properties of thin films have been shown to differ from those measured for bulk materials [3]. However, the challenge associated with performing accurate dielectric measurements for thin films is centered on designing an appropriate sample holder [4].

Open ended coaxial probes [5] and short-terminated [6] probes demand additional fixtures as well as specific geometric dimensions of the sample to obtain accurate results. The free space method, in which a free-standing sample is placed between transmitting and receiving antennas and a loss measurement determined, has limited applicability as the sample width is generally less than the incident wavelength, thus edge diffraction has a considerable impact on the measurements [7]. The inability to produce free-standing samples makes approaches based on resonant cavities infeasible, as precisely-shaped samples are required [8]. Similarly, Fabry-Perot [9], coaxial [10] and split-post [11] techniques all rely on specific sample sizes and/or free-standing samples. A scanning near-field probing technique, sometimes termed scanning microwave microscopy [12], is limited to a sequence of pre-determined frequency measurements rather than the accessible frequency sweep of a network or impedance analyzer. The goal is to develop non-contact electrostatic force microscopy (EFM) to map dielectric variations with submicron resolution. The independence of the swept-frequency response to the (narrow) bandwidth of the EFM probe has already been demonstrated [13,14].
In this design, interdigitated electrodes were embedded in a substrate to provide a deposition plane for the thin film sample (figure 1). Well-separated contact pads provided contact locations for measurement probes.

Figure 1. a) Plan view of the interdigitated sample holder. b) Cross-section of the embedded electrodes. The thickness of the p-type silicon wafer was 400 µm. The chromium adhesion layer is indicated at the electrode interface with SiO₂.

Analytical descriptions for the capacitances due to interdigitated electrodes fabricated in the SiO₂ layer of a SiO₂/Si wafer have been reported in the literature [15]. The complex capacitance, $C_{\text{substrate}}^*$, associated with fringing capacitance contribution due to the SiO₂ substrate is given by

$$C_{\text{substrate}}^* = \left( \frac{N-1}{2} \right) \left[ \frac{1}{\varepsilon_{\text{SiO}_2} \varepsilon_0} + \frac{1}{\varepsilon_{\text{SiO}_2}^\ast} \right]^{-1}$$

(1)

where $N$ is the number of interdigitated electrodes, $C(h_{\text{SiO}_2})$ is the capacitance contribution due to the silicon dioxide, $C(\infty)$ arises from the infinite layer of air above the substrate when no polymer is present, $\varepsilon_{\text{SiO}_2}^\ast$ is the complex permittivity of silicon and $\varepsilon_{\text{SiO}_2} = 4.5$ is the real part of the relative permittivity of SiO₂ [15]. The capacitance contribution from the polymer layer may be described [15]

$$C_{\text{polymer}} = (N-1)\varepsilon_0 \frac{k_{\text{polymer}}}{k_{\text{polymer}}'} L$$

(2)

where $L$ is the finger length, $K$ is an elliptical integral of the 1st kind and the parameters $k_{\text{polymer}}$, $k'_{\text{polymer}}$ are derived from the electrode geometry [15]. The direct capacitances between electrodes as well as between the nearest contact pad and the interdigitated electrodes are, respectively, expressed:

$$C_{\text{elec}} = 2(N-1)\varepsilon_0 \frac{Ls}{s}$$

(3)

$$C_{\text{elec-pad}} \approx 2\varepsilon_0 \frac{Ls}{H}$$

(4)

where $t$ is the depth of the electrodes inside the SiO₂ substrate, $s$ represents the spacing between the fingers and $H$ is the spacing between the interdigitated electrodes and the neighbouring pad. The summation of these contributions yields the total capacitance between the contact pads:

$$C_{\text{total}} = \text{Re}(C_{\text{substrate}}^*) + C_{\text{elec}} + C_{\text{elec-pad}} + C_{\text{polymer}}$$

(5)

and predicts a linear relationship between the total capacitance, $C_{\text{total}}$, and the number of fingers, $N$.

2. Experimental

The capacitance of the system was estimated analytically using (1) – (5) and verified using a standard finite-element solver (COMSOL). The devices were fabricated on a 4” SiO₂/Si wafer, exploiting the
The insulating properties of silicon dioxide ($10^{16} \Omega \cdot \text{cm}$). The electrodes were patterned in a positive photoresist (HPR 504) using a shadow mask. Plasma etching (~ 20 nm/min) was used to create trenches in the SiO$_2$ layer. A 20 nm chromium adhesion layer was deposited and the remainder of the trench was filled with thermally-evapourated copper. The photoresist was dissolved in an ultrasonic bath containing acetone. For the capacitance measurements, the polymer film was deposited from a 2% wt/wt solution of polystyrene (PS) dissolved in toluene and was spin-coated at 1000 rpm for 30 seconds. The pads were exposed with a cotton swab dipped in toluene followed by surface cleaning using isopropyl alcohol (IPA).

A KLA Tencor AS-500 Alpha-Step profilometer was used for surface relief measurements to confirm the electrode trench etch depth and spin-coated polymer thickness. The difference between the deposited copper and SiO$_2$ surface was confirmed using contact atomic force microscopy (Veeco DI3100 and a Nanoscope IV controller). An Agilent 4294A impedance analyzer was used to obtain capacitance measurements in conjunction with an Alessi REL 6100 probe station.

3. Results

The target polymer film thickness was 100 nm, 30 independent replications (stripping and redeposition) confirmed the film thickness was always in the range 100 ± 30 nm. The polymer contribution was estimated experimentally from the difference between a measurement of the interdigitated device without a before the polymer was spin-coated and a second measurement after the spin-coating (figure 2).

![Figure 2: a) Total capacitance ($C_{\text{total}}$) of a 20-finger device with/without the thin polymer film; b) The difference between the 2 cases, i.e. $C_{\text{polymer}}$.](image)

The experimental data were also compared to the results obtained analytically and from finite element simulation. The linearity of $C_{\text{total}}$ in $N$ (number of fingers) anticipated from (1) – (5) is clearly shown over the range $N = 20$ to $N = 70$ in figure 3.

4. Discussion

The data points in figure 3 represent an average of 5 independent measurements. Following each measurement, the polymer film was removed and a fresh film deposited. The maximum and minimum $C_{\text{total}}$ values from each set of replicate data points were within 3% of the average. Thus, the uncertainty may be reasonably considered to lie within the scale of the data points shown in figure 3.

The resonant peak in figure 2a was associated with the combination of cables and probes used in conjunction with the probe station and the impedance analyzer. These parasitic contributions are also evident in the higher-frequency $C_{\text{total}}$ measurements (figure 3b). The shallower gradient of the analytical calculation at 1 MHz was attributed to the quasi-static coplanar line model for interdigitated systems that formed the basis of these calculations [15].
Figure 3: Comparison between the total capacitance obtained from analytical calculations, finite element (FEM) simulations and experimental measurements. The capacitance is expressed in pF: a) Data at 9.414 KHz; b) data at 1 MHz.

5. Conclusions
An interdigitated capacitor to measure the capacitance contribution and, in turn, the dielectric character of a thin polymer film has been designed and demonstrated. The capacitance contribution of a 100 nm polystyrene film was shown to be measureable using this technique.

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