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Indium Tin Oxide Film Characterization at 0.1–20 GHz Using Coaxial Probe Method

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ABSTRACT Indium tin oxide (ITO) is one of the most commonly used optically transparent conductors in applications, such as electro-optic antennas, displays, and optical coatings. However, their RF frequency-dependent electrical properties have not been reported in the literature. In this paper, we present measurements of the electrical properties (permittivity and conductivity) of ITO films in the 0.1–20-GHz frequency range. Measurements were carried out using an in-house open-ended coaxial probe technique employing a one-port reflection coefficient. As usual, calibration and numerical post-processing is needed to extract the electrical properties of the ITO film placed on a 0.5-mm-thick Eagle glass. The measured conductivity was on the order of 10^5 throughout the frequency range, and the real and imaginary parts of the permittivity were on the order of 10^6 at lower frequencies and 10^5 at higher frequencies.

INDEX TERMS Calibration, indium tin oxide (ITO), open ended coaxial probe, thin film characterization.

I. INTRODUCTION

Indium Tin Oxide (ITO) is one of the most commonly used optically transparent conductors (OTC) because a) it exhibits very good trade-off between conductivity and optical transparency, and b) can be easily deposited as a thin film on other glass surfaces using sputtering techniques reported in [1]–[3]. Indeed, thin ITO films are currently employed in a number of applications, including transparent conductive coatings for displays (e.g., touchscreens, liquid crystal displays, plasma displays, etc.), electro-optic antennas, antistatic and optical coatings, solar cells, strain gauges, etc. But to date, ITO films have only been widely characterized in terms of optical performance [4], electroluminescence [5], and structural properties [6]. Their frequency-dependent electrical properties have yet to be reported in the literature.

In this paper, we provide broadband measurements to extract the electrical properties of thin (100 nm) ITO films across a $0.1 - 20$ GHz frequency range. A challenge for our measurements is that commercially available material characterization probes are unsuitable. Specifically, commercial open-ended coaxial probes may provide broadband measurements, but require sample thicknesses of at least $20/\sqrt{\epsilon_r}$, where ϵ_r is the permittivity of the material under test [7]. Analysis using the parallel plate method may be employed for material sheets, but characterization is not broadband (typically goes up to 1 GHz), and requires sample thicknesses of at least 0.3 mm [7]. Alternatively, the standard 4-point probe method may be used for very thin conductive films, but this approach is suitable for DC characterization [8].

Given the small thickness (100 nm) of the ITO film under consideration and the need for broadband characterization $(0.1 - 20$ GHz), the aforementioned methods are of limited use. Our approach is to instead employ an open-ended coaxial probe (OECP) technique described in [9] and [10]. Specifically, we employed a 1-port reflection coefficient (S_{11}) measurement for the thin ITO film deposited on Eagle glass substrate (see Fig. 1). A post-processing method is then used

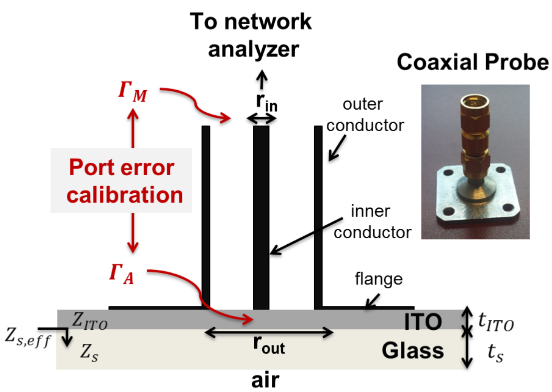

FIGURE 1. Illustration of the measurement setup showing the open ended coaxial probe and the ITO coated glass.

to de-embed the film's permittivity and conductivity from the measured S_{11}. As required, a simple one-port calibration process was employed with good accuracy across the $0.1 - 20$ GHz bandwidth.

This paper is organized as follows. Section II presents the employed measurement and calibration technique. Next, Section III provides measurements of the electrical properties for thin ITO films at $0.1 - 20$ GHz.

II. MEASUREMENT AND CALIBRATION TECHNIQUE

A. EXPERIMENTAL SETUP

The goal is to extract the frequency-dependent conductivity σ_{ITO} and permittivity $\epsilon_{r,ITO}(\omega)$ of the thin OTC (ITO), deposited on a glass substrate. The frequency of interest is from 0.1 to 20 GHz. To do so, we employed the open-ended coaxial probe method in [9] and [10]. The probe and sample geometry are depicted in Fig. 1.

To extract $\sigma_{ITO}(\omega)$ and $\epsilon_{r,ITO}(\omega)$ using the setup in Fig. 1, the coaxial probe is firmly placed on the ITO coated glass. The reflection coefficient Γ_M is then measured using a network analyzer. It is, of course, necessary to calibrate out errors prior to further processing. These errors are due to cable losses and probe imperfection. A description of the calibration process is given in Section II-B. Then, $\sigma_{ITO}(\omega)$ and $\epsilon_{r,ITO}(\omega)$ can be accurately extracted using the formula [10]

$$\sigma_{ITO}(\omega) = \frac{1}{t_{ITO} Z_0} Re\left\{\sqrt{\epsilon_r}\frac{1-\Gamma_A}{1+\Gamma_A}\right\} (S/m) \tag{1}$$

and

$$\epsilon_{r,ITO}(\omega) = \frac{\epsilon'_{ITO} - j\sigma_{ITO}/\omega}{\epsilon_0} \tag{2}$$

with

$$\epsilon'_{ITO}(\omega) = \frac{1}{\omega t_{ITO} Z_0} Im\left\{\sqrt{\epsilon_r}\frac{1-\Gamma_A}{1+\Gamma_A}\right\} (F/m) \tag{3}$$

Here, Γ_A is the corrected reflection coefficient after calibration. Also, t_{ITO} is the ITO coating thickness and ϵ_r is the coaxial probe's filling dielectric constant. Further, as usual, ϵ_0 and Z_0 are the free space permittivity and characteristic impedance ($\approx 377\Omega$), respectively. We note that (1) and (3) are valid for a thin ITO [11], that is,

$$|Z_{s,eff}| \gg |Z_{ITO}| \tag{4}$$

and

$$|k_{ITO} t_{ITO}| \ll 1 \tag{5}$$

where $Z_{s,eff}$ is the impedance looking toward the glass (at the glass surface, see Fig. 1). That is, $Z_{s,eff}$ is given by the general transmission formula

$$Z_{s,eff} = Z_s \frac{Z_0 + Z_s tanh(jk_s t_s)}{Z_s + Z_0 tanh(jk_s t_s)} \tag{6}$$

Also, Z_{ITO}, k_{ITO} refer to the characteristic impedance and wave propagation constant in the thin film, respectively.

In (6), Z_s and k_s are the characteristic impedance and wave propagation constant in the glass substrate of thickness t_s.

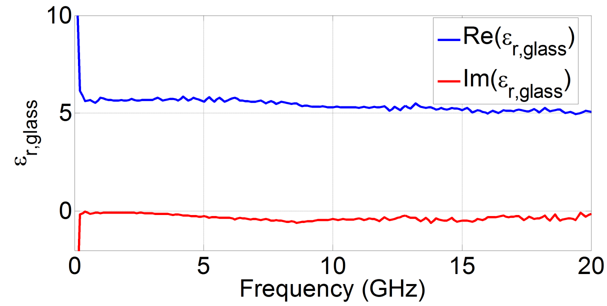

FIGURE 2. Measured relative permittivity for a sample of Eagle glass, of thickness 5 mm, using the Agilent® 85070D dielectric probe.

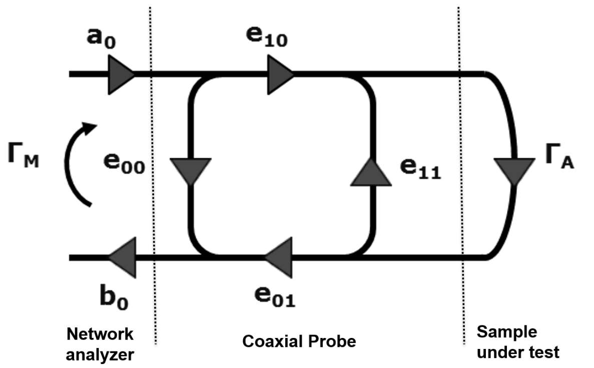

FIGURE 3. Signal flow diagram representing the coaxial probe. a_0 and b_0 are the incident and reflected waves at the input of the probe, respectively.

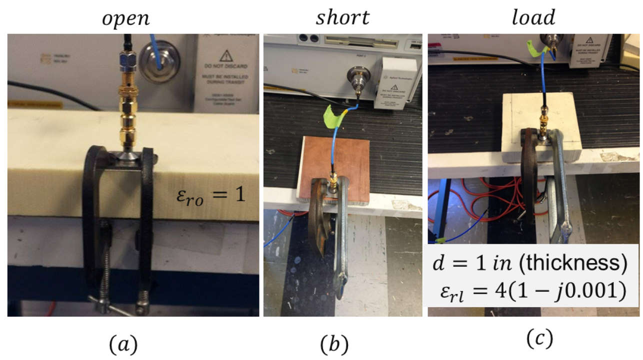

FIGURE 4. Three different calibration measurements: (a) open circuit ($\epsilon_{r,open} = 1$), (b) short circuit, and (c) matched load ($\epsilon_{r,load} = 4(1 - j0.001)$).

To compute Z_s and k_s, it is important to know *a priori* the frequency-dependent relative permittivity of the employed Eagle Corning 1737® glass substrate. This is found using the Agilent® 85070D dielectric probe placed on a a glass sample of thickness t_s =5 mm. The extracted ϵ_r is plotted in Fig. 2. As expected, Eagle glass has a relative permittivity of $\epsilon_r = 5.7$ over the entire frequency band of interest [12].

Having the glass substrate's ϵ_r, we proceed to measure the ITO coated glass properties. We used an RG-12A/U coaxial probe having inner and outer diameter $r_{in} = 1.13$ mm and $r_{out} = 3.63$ mm, respectively. The dielectric filling

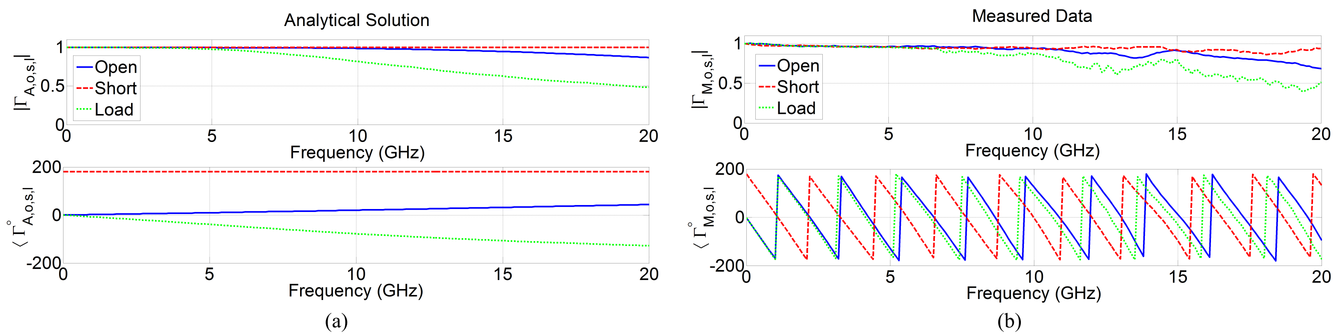

FIGURE 5. Magnitude and phase of the (a) computed and (b) measured reflection coefficients for the three different setups: open circuit, short circuit, and matched load.

of the probe was Polystyrene and of a relative permittivity $\epsilon_r = 2.25(1 - j0.001)$. Also, as already noted, the sample under test (SUT) is a 100 nm of ITO coating on a 25×25 mm^2 Eagle Glass of 0.5 mm thickness.

B. BROADBAND PROBE CALIBRATION

The probe's calibration is necessary to compensate for losses and equipment non-idealities. The signal flow diagram for obtaining the overall reflection coefficient is given in Fig. 3. Here, e_{00} is the directivity error, e_{10} and e_{01} are the reflection tracking errors, and e_{11} is the source matching error [10]. Solving the flow diagram, we obtain

$$\Gamma_M = \frac{b_0}{a_0} = e_{00} + \frac{e_{01}e_{10}\Gamma_A}{1 - e_{11}\Gamma_A} \tag{7}$$

From (7), we can solve for Γ_A to get

$$\Gamma_A = \frac{\Gamma_M - b}{-c\Gamma_M + a} \tag{8}$$

Here, $a = e_{01}e_{10} - e_{00}e_{11}$; $b = e_{00}$; $c = -e_{11}$ and can be found by measuring the reflection coefficient (Γ_M) of the film for a given Γ_A.

To find a, b, and c, we need three independent measurements. As an example, we can use the open circuit, short circuit, and known load terminations to generate three equations of the form:

$$a\Gamma_{A,i} + b - c\Gamma_{A,i}\Gamma_{M,i} = \Gamma_{M,i} \tag{9}$$

where the subscript i denotes either *open*, *short*, or *load* terminations.

Ideally, $\Gamma_{A,open} = 1$, $\Gamma_{A,short} = -1$, and $\Gamma_{A,load} = 0$. However, in practice, these values are frequency-dependent and need to be carefully considered. Therefore, it is necessary to use more accurate formula. To do so, we follow the method in [13]. We also represent Γ_{Ai} as

$$\Gamma_{Ai} = \frac{1 - y_i}{1 + y_i} \tag{10}$$

where i refers to *open*, *short*, or *matched load* terminations, respectively. In (10), y_i is the coaxial probe aperture admittance, given by [13]

$$y_i = \frac{\epsilon_{ri}}{\sqrt{\epsilon_r} ln(\frac{r_{out}}{r_{in}})} \int_0^\infty \frac{[J_0(k_0\zeta r_{out}) - J_0(k_0\zeta r_{in})]^2}{\zeta} F_i(\zeta) d\zeta \tag{11}$$

where J_0 is the Bessel function of the first kind of zeroth order. The integrand function $F_i(\zeta)$ is associated with the boundary conditions enforced at each layer, and ϵ_{ri} is the relative permittivity of the load under test.

To realize an *open*, the probe's reflection coefficient is measured without the coated substrate. The actual setup is given in Fig. 4(a). We note that for the open case,

$$F_i(\zeta) = \frac{1}{\sqrt{\epsilon_{r,open} - \zeta^2}} \tag{12}$$

Using (12) into (11) and then (10), $\Gamma_{A,open}$ is computed and plotted in Fig. 5(a).

To realize a *short*, the probe's end is simply shorted, as depicted in Fig. 4(b). In this case,

$$y_s = \infty \Rightarrow \Gamma_{A,short} = -1 \tag{13}$$

Finally for the load termination, we employ a metal-backed dielectric, as illustrated in Fig. 4(c), of thickness $d = 1''$ and relative permittivity $\epsilon_{r,load} = 4(1 - j0.001)$. For this metal-backed slab, we find that [13]

$$F_i(\zeta) = \frac{1}{\sqrt{\epsilon_{r,load} - \zeta^2}} \frac{1 + e^{-j2k_0d\sqrt{\epsilon_{r,load} - \zeta^2}}}{1 - e^{-j2k_0d\sqrt{\epsilon_{r,load} - \zeta^2}}} \tag{14}$$

Upon substitution of (14) into (11), and then (10), $\Gamma_{A,load}$ is computed. The magnitude and phase of these computed $\Gamma_{A,i}$'s are given in Fig. 5(a). Alternatively, the measured $\Gamma_{M,i}$'s are plotted in Fig. 5(b). Next, $\Gamma_{A,i}$'s and $\Gamma_{M,i}$'s for the three different setups are used in (9) to solve for the calibration complex coefficients a, b, and c. Results are shown in Fig. 6 and used to calibrate subsequent measurements.

III. ITO CHARACTERIZATION

As mentioned earlier, the SUT is a $25 \times 25 \times 0.5$ mm^3 Eagle Glass with 100 nm of ITO coating. Fig. 7 shows the measurement setup. Two clamps were

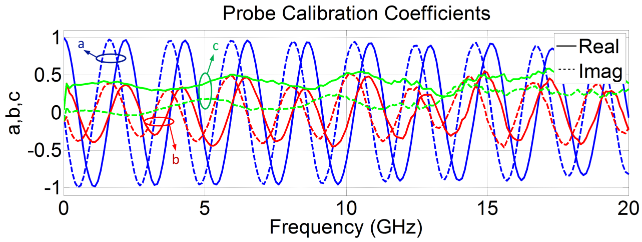

FIGURE 6. OECP calibration complex coefficients.

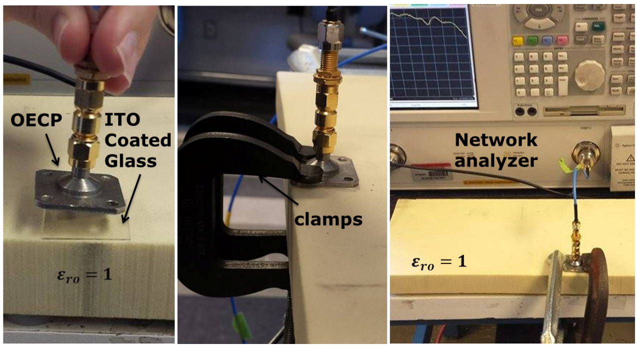

FIGURE 7. Photo of the setup for ITO on Eagle glass characterization.

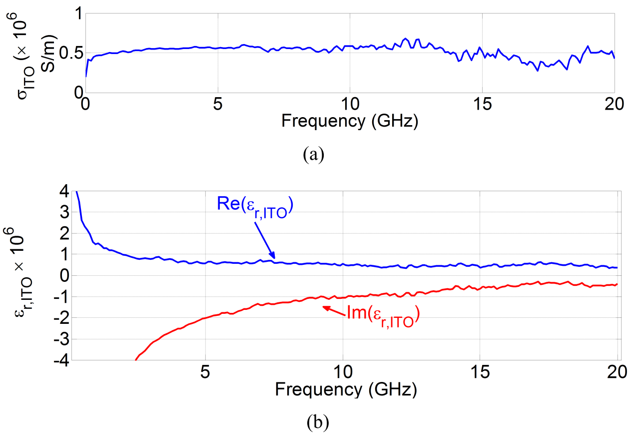

FIGURE 8. (a) Conductivity $\sigma_{ITO}(\omega)$ and (b) relative permittivity $\epsilon_{r,ITO}(\omega)$ of ITO film measured from 0.1-20 GHz.

employed to ensure firm contact of the probe and the sample. We then used the network analyzer to measure the reflection coefficients, Γ_M, across 0.1-20 GHz. Next, the actual reflection coefficient, Γ_A, is extracted after calibration using (8) and substituted into (1)-(3) to extract $\sigma_{ITO}(\omega)$ and $\epsilon_{r,ITO}(\omega)$. The extracted $\sigma_{ITO}(\omega)$ and $\epsilon_{r,ITO}(\omega)$ are plotted in Fig. 8(a) and 8(b). As expected, the ITO's conductivity is on the order of 10^5 [10]. Also, the real and imaginary parts of its permittivity are on the order of 10^6 at lower frequencies and 10^5 at higher frequencies.

After extracting the ITO's conductivity and relative permittivity, we proceeded to check the validity of the approximations by checking (4) and (5). We found that the minimum value of the ratio $|Z_{s,eff}|/|Z_{ITO}|$ in Fig. 9(a) is 670 $\gg$ 1 validating (4). Also, the maximum value of $|k_{ITO}t_{ITO}|$ in Fig. 9(b) is 0.03 $\ll$ 1, validating (5).

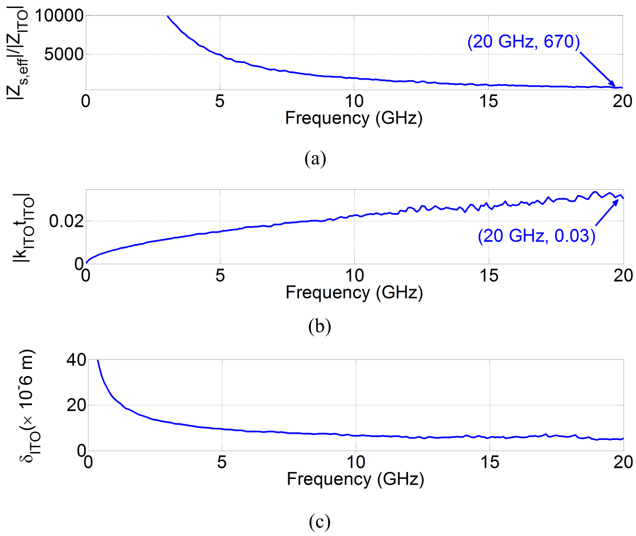

FIGURE 9. Thin film approximation: (a) $|Z_{s,eff}| \gg |Z_{ITO}|$. (b) $|k_{ITO}t_{ITO}| \ll 1$. (c) Skin depth δ_{ITO} from the measured data.

The skin depth ($\delta_{ITO} = 1/\sqrt{\pi f \sigma_{ITO} \mu_0}$, where μ_0 is the free space permeability) was also computed and plotted in Fig. 9(c). As expected, δ_{ITO} at low frequencies is much higher than the ITO thickness ($\delta_{ITO} = 40$ μm versus $t_{ITO} = 100$ nm), again validating the condition (5).

IV. CONCLUSION

We presented measurements of the electrical properties (permittivity and conductivity) of ITO in the 0.1-20 GHz frequency range. To our knowledge, these are the first extracted electrical properties of the ITO across this wideband range. The samples under test were 100 nm thick ITO films deposited on 0.5 mm thick Eager glass. To overcome limitations of commercial material characterization techniques, viz. limited frequency range and requirement for thick material samples, we used an in-house open-ended coaxial probe technique. Our one-port calibration approach removed the effect of the employed probe to accurately compute the frequency-dependent material properties of the ITO coated glass. Post-processed results showed that ITO conductivity was on the order of 10^5. The real and imaginary parts of permittivity were also found on the order of 10^6 at lower frequencies, and on the order of 10^5 at higher frequencies.

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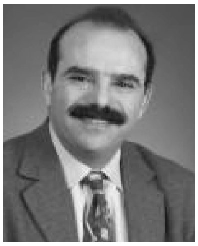

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