Electromagnetic characterization of transparent conducting thin films using two-port flange coaxial probe and thin-film transfer matrix method

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Abstract
An efficient method to determine the electromagnetic characteristics of transparent conducting oxide (TCO) thin films is presented herein. When a two-port flange coaxial probe is used for electromagnetic characterization, it is important to note that since the thickness of the film is very thin, it should be taken into account that the measured S parameters not only correspond to the thin film, but also to the substrate. The main idea of this study is to exploit the S parameters corresponding solely to the thin film by employing transfer matrix method. Then, by using the obtained S parameters, the effective conductivity (σ) and relative permittivity (ε_r) of the TCO thin film are determined. By applying this method to a sample under test, the conductivity and relative permittivity are obtained in the order of 10^5 (S/m) and 10^6 in the 2–20 GHz frequency range, respectively. These results are compared with those obtained by the Nicolson–Ross–Wier and open-ended coaxial probe methods. Simulation results show that when the TCO thin film thickness reaches 0.5 mm, its conductivity increases up to two times. Also, when the gap between the coaxial probe and the thin-film surface is 0.1 mm, the conductivity approaches zero. Using this method, the material and thickness of the substrate have slight effect on determining the electromagnetic characteristics.

1 | INTRODUCTION

Transparent conducting oxide (TCO) thin films, which exhibit transparency and conductivity simultaneously, have found many scientific and industrial applications. Solar cells, OLEDs, electromagnetic shielding and transparent antennas are prime examples of these materials applications [1–5]. In applications such as transparent antennas, the most important issue to consider is determination of their exact electromagnetic characteristics, i.e. their conductivity and relative permittivity, especially in the microwave frequency band. Therefore, knowing their exact electromagnetic characteristics is necessary to correctly select them in this frequency band.

The important issues in determining the measurement method are frequency range, the material size and thickness, the substrate material, being destructive or non-destructive, with or without contacting, measurement accuracy, material properties (homogeneous and isotropic), material shape, temperature, cost and simplicity of procedure.

There are various methods to determine electromagnetic characteristics of thin films. Planar circuits such as microstrip lines and coplanar waveguides have been used for thin-film electromagnetic characterization [6,7]. In these methods, thin film is considered as a part of the transmission line. Then, the electromagnetic characteristics of the thin film are extracted based on the characteristics of the other parts of the transmission line [8–12]. Determining accurate electromagnetic characteristics using this method is relatively difficult. One of the most commonly used methods is the open-ended coaxial probe (OECP) [13–16]. This method is based on scattering parameter (S_{11}) measurement and relation between S_{11} and (σ,ε_r). It works well in determining the electromagnetic characteristics of bulk materials, but its accuracy is questionable for thin films of very small thickness (e.g. about 500 nm). In this method, errors that may occur are calibration error, error due to the gap between the probe and thin film, and error due to small thickness of the thin film. To overcome the first two error cases, several methods have been proposed [14–16].
However, in case of the third error, when the thickness of the thin film is much less than the wavelength, the results may show error and uncertainty. Since this method is based on the scattering parameter \(S_{11}\) measurement, it should be made clear whether the measured \(S_{11}\) parameter is exclusively related to the thin film or it is also affected by the substrate. In addition, in employing this method, some assumption should be taken into account as mentioned in [15].

Another similar developed method is two-port flange coaxial probe (TPFCP) [17]. In this method, a network analyser and a pair of coaxial probes are employed. The sample under test (SUT) is inserted between the probes as schematically shown in Figure 1. Then, by measuring the scattering parameters and their relations with \((\sigma, \varepsilon)\), the electromagnetic characteristics of the material are obtained.

This configuration was applied for electromagnetic characterization of thin films in [18]. Theoretical reflection and transmission coefficients by TPFCP method were obtained using a full wave analysis based on mode-matching technique and Hankel transform [19]. These methods require solving complex equations. In addition, a one-layer structure is considered. Since the structure of TCO thin film and the substrate is considered as a two-layer structure, these equations will be more complex and difficult, which in turn their utilization will increase the computational error.

Another important issue is the very small thickness of the thin film. The thickness of the thin film is less than one micrometer and the wavelength of the electromagnetic wave used to measure the \(S\) parameters is about 3 cm. Therefore, there is a concern that the incident wave may not properly observe the thin film and the measured \(S\) parameters (and hence the derived electromagnetic characteristics) depend not only on the thin film but also on the substrate. This issue has not been addressed so far in the reported methods.

The main purpose of this study is to propose a new method based on TPFCP configuration for determining the exact electromagnetic characteristics of the TCO thin films \((\sigma, \varepsilon)\) in the microwave band in presence of the two mentioned issues specially the small thickness of the thin film. We use the transfer matrix of the thin film for determining \(S\) parameters corresponding solely to the TCO thin film. Then, by using these \(S\) parameters, \((\sigma, \varepsilon)\) of the TCO thin film are obtained.

In Section 2, the electromagnetic behaviour of the wave passing through a thin film is analysed. In Section 3, the proposed method is described. A brief explanation of how to make a TCO thin film is described in Section 4. The experimental and simulation results are provided and compared with each other. For comparison, \((\sigma, \varepsilon)\) are also determined by Nicolson–Ross–Wier (NRW) method without substrate effect elimination and OECP method. In Section 5, the effects of thin film thickness and gap between flange and SUT are examined. Finally, the effects of material, thickness and length (size) of the substrate are investigated in Section 6.

### 2 | ELECTROMAGNETIC BEHAVIOUR OF TCO THIN FILM UNDER TPFCP TEST

For a conductive material, the complex permittivity coefficient is:

\[
\tilde{\varepsilon} = \varepsilon - j\sigma/\omega
\]  

(1)

Dispersion relation for a conductor can be expressed as:

\[
k = \omega\sqrt{\mu_0\varepsilon} = k_+ + jk_-
\]  

(2)

where

\[
k_+^2 = \frac{\omega^2\mu_0\varepsilon}{2} \left[ 1 + \left( \frac{\sigma}{\varepsilon\omega} \right)^2 \right]^{\pm 1}
\]  

(3)

and \(k\) is the propagation constant in the conductor [20]. Let the interface between thin film and substrate be located at \(z = 0\) as shown in Figure 1. Thus, a plane wave propagating in the \(+z\) direction can be expressed as:

\[
E(z, t) = E_0 e^{j(\omega t - kz)} = E_0 e^{-k_+ z} e^{j(\omega t - k_- z)}
\]  

(4)

The plane wave from coaxial cable (TEM mode) is divided into propagation and exponential damping components along the \(z\) axis. The skin depth of the TCO thin film \(\delta\) for the damped wave by \(e^{-z/\delta}\) is obtained by:

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**Figure 1** The schematic diagram of the TPFCP method.
\[ \delta = \frac{1}{k_c} = \frac{1}{\sqrt{\frac{\omega^2 \mu_0 \varepsilon}{2} \left[ \sqrt{1 + \left( \frac{\sigma}{\omega \varepsilon} \right)^2} - 1 \right]} } \] (5)

TCO thin film is a good conductor in microwave region. In this region, \( \delta \) is about micrometers while the thin film thickness is about a few hundred nanometers. Therefore, the electromagnetic wave penetrates into the thin film and then to the substrate. The effective impedance of each layer \( (Z_l) \) is determined by the transmission line theory and impedance transformation [21] as follows:

\[ Z'_l = Z_c Z'_{l+1} + jZ_c \tan (k_c t_l) \]
\[ Z_l + jZ'_{l+1} \tan (k_c t_l) \] (6)

where \( Z_l \) is the impedance of the \( l \)-th layer, \( k_c = \frac{\mu_0 \omega}{Z_c} \) is the propagation constant and \( t_l \) is the thickness of the \( l \)-th layer. \( Z_c = \sqrt{\frac{\mu_0}{\varepsilon}} = \sqrt{\frac{\mu_0}{\varepsilon - j\omega \sigma}} \) and \( Z_s = \sqrt{\frac{\mu_0}{\varepsilon \varepsilon_0 (1 - j\omega \delta)}} \) are the thin film and substrate impedances, respectively. \( \sigma \) and \( \varepsilon \) are the conductivity and permittivity of the TCO thin film; \( \varepsilon_0 \) and \( \tan \delta \) are the relative permittivity and loss tangent of the substrate, respectively. Also, \( t_c \) and \( t_s \) are thicknesses of the thin film and substrate, respectively. The primes (') indicate the effective quantities. As Equation (6) shows, the effective impedance of TCO thin film and the scattering parameters are dependent on the substrate characteristics. Therefore, in general, the determination of the electromagnetic characteristics of each layer in a multilayer system depends on the subsequent layers and we have to find a way to reduce or eliminate this effect. Usually the substrate impedance is much higher than the impedance of the next layer (flange), and hence the effective impedance of the substrate is

\[ Z'_s = jZ_s \tan (k_c t_s) \] (7)

This relation shows that resonances can occur due to frequency dependence of \( k_c \). To determine the effective impedance of TCO thin film, according to \( |k_c t_c| \ll 1 \), we can use the first-order expansion of \( \tan(k_c t_c) \) in Equation (6). The result is:

\[ \frac{1}{Z_c} = \frac{1}{Z'_c} + \frac{k_c t_c}{Z_c} - j \frac{\mu_0 \omega t_c}{Z'_c} \] (8)

This relation provides the equivalent admittance of the structure shown in Figure 2. It indicates that the resonances occur at specific frequencies. In fact, at the frequency where \( Z'_c = \infty \), the imaginary part of Equation (8) is zero, and as a result we will have resonance at this frequency. This structure (thin film–substrate flange) can be viewed as a resonator.

\[ \begin{align*}
S_{11} & = \frac{Z'_c}{Z_c} & T_{11} & = \frac{1}{\varepsilon_c} \\
S_{12} & = \frac{Z'_c T_{11} + Z_c T_{12}}{Z_c} & T_{12} & = \frac{Z_c}{\varepsilon_c} \\
S_{21} & = \frac{Z_{s2}}{T_{21}} & T_{21} & = \frac{\varepsilon_c}{Z_s} \\
S_{22} & = \frac{Z_{s2}}{T_{21}} & T_{22} & = \frac{S_{22}}{S_{22}}
\end{align*} \]

**TABLE 1** Conversion relations between \( S \) and \( T \) parameters

\[ T_M = T_i \times T_s \]

(9)

Therefore:

\[ T_i = T_M \times T_s^{-1} \] (10)

where \( T_i \) and \( T_s \) are the transfer matrices of thin film and substrate, respectively.

Therefore, the proposed procedure is as follows: First, the \( S_M \) parameters (\( S_{M\ 11}, S_{M\ 12}, S_{M\ 21} \) and \( S_{M\ 22} \)) of the two-layer structure, including thin film and the substrate, are measured.
by a network analyser. Then, the same procedure is performed for the \( S \) parameters of the substrate without the thin film \( (S_{t,11}, S_{t,12}, S_{t,21} \) and \( S_{t,22} \)). By converting \( S \) to \( T \) according to Table 1 and using Equations (9) and (10), \( T \) parameters \( (T_{t,11}, T_{t,12}, T_{t,21} \) and \( T_{t,22} \)) are determined. Then, the \( S_{t,11} = \frac{T_{t,11}}{\rho_{11}} \) and \( S_{t,21} = \frac{T_{t,21}}{\rho_{11}} \) of the thin film are obtained using the relations of Table 1 [22].

The next step is determining \((\sigma, \varepsilon)\) from scattering parameters. According to the structure of TPFCP shown in Figure 1, we can use the relations mentioned in [7,23,24] which are also known as the NRW method relations.

\[
K = \frac{(S_{t,11}^2 - S_{t,21}^2) + 1}{2S_{t,11}} \quad (11)
\]

\[
\Gamma = K + \sqrt{K^2 - 1} \quad (12)
\]

\[
T = \frac{(S_{t,11} + S_{t,21}) - \Gamma}{1 - (S_{t,11} + S_{t,21})\Gamma} \quad (13)
\]

\[
\frac{1}{\Lambda^2} = \left[ 1 + \frac{1}{2\pi d} \ln \left( \frac{1}{T} \right) \right]^2 \quad (14)
\]

\[
\mu = \frac{1 + \Gamma}{(1 - \Gamma) \lambda_c \sqrt{\left( \frac{1}{\lambda_c^2} \right) - \left( \frac{1}{\Lambda^2} \right)}} \quad (15)
\]

\[
\varepsilon = \frac{\lambda_c^2}{\mu} \left[ \left( \frac{1}{\Lambda^2} \right) + \left( \frac{1}{\lambda_c^2} \right) \right] = \varepsilon' - j\sigma/\omega\varepsilon_0 \quad (16)
\]

where \( \lambda_0 \) is the free space wavelength and \( \lambda_c \) is the cut-off wavelength of the transmission line section \( \lambda_c = \infty \) for a TEM line.

In summary, the proposed method consists of two steps. The first step is to determine the scattering parameters of thin film \( S_{t,11} \) and \( S_{t,21} \) at the coaxial probe apertures. The second step is to use the relations of \( S_{t,11} \) and \( S_{t,21} \) to determine \( \sigma \) and \( \varepsilon \). Using this method, the effect of the substrate on \( \sigma \) and \( \varepsilon \) of TCO thin film is eliminated. To the best of our knowledge, this method does not have the complexity of the usual methods and also eliminates the effects of the substrate effectively. Figure 3 shows the flowchart of the method.

### 4 | EXPERIMENTAL AND SIMULATION RESULTS

#### 4.1 | TCO thin-film fabrication and experimental procedure

Spray pyrolysis method has been used for TCO thin-film deposition. The deposition was applied on the Pyrex substrate of 100 mm \( \times \) 100 mm dimensions and 2 mm thickness. In order to apply this method, a precursor solution has been prepared as referred to in [25]. The solution used for fluorine tin oxide (FTO) spray pyrolysis deposition is composed of pentahydrate tin tetrachloride \((SnCl_4\cdot5H_2O)\), ethanol
(C₆H₃OH) and ammonium fluoride (NH₄F), which were gradually dissolved in distilled water at 40–50°C. A few drops of hydrochloric acid was added to the solution to increase the clarity. Sample solution volume was 20 cc. Then, multiwall carbon nanotube (MWCNT) is added to the solution. The substrate was cleaned and placed on the hot plate and its temperature was maintained at 500 °C. Spray nozzle to the substrate distance and spray flow rate were 35 cm and 5 cc/min, respectively. Among several depositions, the best result for transparent antenna applications corresponded to the FTO sample doped with 5 mg of MWCNT. Figure 4 shows the scanning electron microscope (SEM) micrograph of the sample’s cross section and an image of the deposited MWCNT-doped FTO thin film.

Figure 5 shows optical transmittance of the sample thin films obtained by the Perkin Elmer Lambda 9 UV-Vis-NIR spectrophotometer. For comparison, the Pyrex substrate transparency and transmittance of the undoped FTO are also shown. Table 2 summarizes the surface resistance, optical transmittance and thickness measurements of the sample. The surface resistance is measured by four-point probe and the thickness of the thin film layer is determined by the method proposed by Swanepoel [26].

The image of the measurement setup is shown in Figure 6. We used the E8363B PNA Network Analyser for applying the method mentioned in Section 3. The coaxial cable probe is Agilent 85133–60017. The dielectric filling of the probe is polystyrene with ε_{coax} = 2.25(1 – j0.001) in the frequency range tested (2–20 GHz) [27]. The device was first calibrated by the procedure mentioned in [14] using short-open-load matching calibration and gated reflect line to shift the phase reference planes to the front and back of the TCO thin film.

| Material          | R_{s}(Ω/□) | Average transparency (%) | Thickness |
|-------------------|------------|--------------------------|-----------|
| Pyrex (Substrate) | ----       | 90                       | 2 mm      |
| FTO+MWCNT         | 5          | 77                       | 500±25 nm |

4.2 Measurement and simulation results

In accordance with the method described in Section 2, first, two-port S parameters of TCO thin film with substrate are measured. The same procedure is then repeated for the substrate alone. Using Equation (10) and transferring T to S, the S_{11} and S_{21} of the TCO thin film are extracted. After that, Equations (11)–(16) are used to determine \( (σ,ε) \) of the TCO thin film. In order to further evaluate the experimental results, the proposed method was also simulated using finite element method by COMSOL Multiphysics software to obtain the electromagnetic characteristics of the thin film. The obtained \( (σ,ε) \) of thin film derived from NRW method relations are employed in simulations. That is, first we obtained \( (σ,ε) \) of thin film without removing the substrate effect by Equations (11)–(16), then we obtained the S parameters by simulation of structure in COMSOL. After eliminating the substrate effect, we determined \( (σ,ε) \) of thin film. Figure 7 shows the measurement and simulation results of \( (σ,ε) \) of TCO thin film.

The conductivity of TCO thin film is in the order of \( 10^5 \) S/m and the real and imaginary parts of relative permittivity \( (ε) \) are in the order of \( 10^5 \) in both measurement and
simulation results. A fairly good agreement can be seen between measurement and simulation results. Some irregular small fluctuations are observed at high frequencies in measurement results that are probably due to the measurement errors, which are negligible. The observed resonances in simulation results are due to the flange reflection [28] that we have explained in Section 2. In order to have a reference, we also extract the electromagnetic characteristics of TCO thin film by NRW method without considering thin-film transfer matrix. To compare further, the results of the OECP method mentioned in [25] are shown as well.

In order to have a reference, we also extract the electromagnetic characteristics of TCO thin film by NRW method without considering thin-film transfer matrix. To compare further, the results of the OECP method mentioned in [25] are shown as well. The comparison shows that the conductivity curve obtained using the TPFCP method has better agreement with the simulation than the one obtained by the OECP method over the desired frequency band. This is due to the fact that we have extracted the exact value of $S_{11}$ that corresponds to the thin film. In the NRW method without substrate effect elimination, the conductivity changes about $1.7 \times 10^5$ (S/m) over the frequency band. In the OECP method, this change is $\sim 0.85 \times 10^5$ (S/m). Such change is not observed in the TPFCP method, since the substrate effect has been removed. Figures 8 and 9 show the measurements of scattering parameters of thin-film structures. The results show the magnitude and phase of the measured $S_{11}$ and $S_{12}$.

5 | EFFECTS OF THIN FILM THICKNESS AND THE GAP BETWEEN THE FLANGE AND THE THIN FILM

Two important parameters that affect the obtained values of $(\sigma, \varepsilon_r)$ of the thin film are thin film thickness and the gap between flange and the thin film when applying the proposed method. We simulated the structure shown in Figure 1 by finite element method to investigate these effects. Figure 10a shows the effect of thin film thickness on conductivity. It is important to note that as the thickness of the thin film increases, the conductivity decreases initially, and then increases rapidly. This is probably because the energy is coupled to substrate and then the thin film gradually behaves like a good conductor (for thicknesses up to 3 $\mu$m). For thicknesses greater than 500 $\mu$m, there is no noticeable change, and the conductivity is almost constant.
Figure 10b shows the effect of the gap between the flange and the thin-film surface on conductivity. In measuring by a network analyser, it is very important that the surface of the coaxial probe and the thin film stick together tightly, but in practice a gap might exist between the two surfaces. As shown in Figure 10b, the conductivity decreases with increasing the gap. For gaps greater than 100 μm, the conductivity diminishes.

6 | EFFECTS OF THICKNESS, MATERIAL AND LENGTH (SIZE) OF THE SUBSTRATE

In this section, the effect of the substrate on $\sigma$ of TCO thin film is investigated. Therefore, the thickness and material of the substrate were changed and simulated. The thickness of the substrate is considered to be 2, 4 and 8 mm (Figure 11a). The results show that by doubling the thickness of the substrate, the conductivity is reduced by about 1000 $(S/m)$, which is a small amount compared with the conductivity of the TCO thin film. In addition, three substrates with materials Pyrex, PET and Quartz were considered and simulated. Figure 11b shows that the conductivity did not change significantly with the change in the substrate material, but the resonances caused by edge reflection changed.

7 | CONCLUSION

An effective method for measuring the electromagnetic characteristics of a TCO thin film has been proposed. The advantage of this method is that despite the very thin thickness of TCO thin film compared with the substrate and wavelength, $S$ parameters are calculated fairly accurate and the effects of the substrate in determining ($\sigma,\varepsilon_r$) are reduced as much as possible. Another advantage of this method is its relative simplicity compared with solving the full electromagnetic equations, which leads to complex mathematical computations and
increases the computational error. In addition, there is no need for the assumptions made in the OECM method. The measurement and simulation results show similar behaviour. The resonances that appear on the simulation results are due to the reflection effect from the flange edges. Increasing the thickness of the thin film results in the increasing of the conductivity up to a maximum of twice. The conductivity calculation of the thin film is lost when the gap reaches 100µm. The results show that the thickness and material of the substrate used do not have significant effect on determining the electromagnetic characteristics of the TCO thin film. The proposed method can be used to measure the electromagnetic characteristics of any thin conductive/semi-conductive materials, especially TCO thin films.

ACKNOWLEDGEMENT
The authors hereby express their gratitude and thanks to Dr. Bagheri-Mohagheghi, Dr. Joodaki, Dr. Forouzanfar and Mr. Azimi-Juybari for their support in fabrication and measurement.

CONFLICT OF INTEREST
The authors declare that there is no conflict of interest.

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