

# COMPARATIVE MICROWAVE MEASUREMENTS OF COMPLEX DIELECTRIC CONSTANT OF HIGH PERMITTIVITY THIN FILMS

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In the proposed work, complex dielectric constants of high permittivity thin films of  $\text{BaTiO}_3$  and  $\text{PbNbO}_3$  are measured at microwave frequencies. Working equations are presented for  $\epsilon'$  and  $\tan\delta$  by means of a new method of measurement, earlier reported by the author, with corrections for supporting substrate reactance in the present work. Verification of experimental results are done by means of multilayered dielectric slab loaded waveguide method and modified Lrude's method. The working equations for latter two methods are also presented. Experimental results of  $\text{BaTiO}_3$  and  $\text{PbNbO}_3$  thin films as measured by these three methods are given.

## Introduction

Experimental results for complex dielectric constants of both high and low permittivity thin films are not available at microwave range of frequencies. This is because of the fact that most of the microwave methods of measurement which determine the complex dielectric constants of bulk samples are not effective in measuring the  $\epsilon^*$  of thin films. It has been reported that the modified cavity<sup>1</sup> perturbation technique is effective to measure complex dielectric constant of thin film samples. Ahluwalia et al.<sup>2</sup> also reports its effectiveness regarding measurement of  $\epsilon^*$  of biological thin film samples. Dubey et al.<sup>3</sup> have reported a method of measurement of complex dielectric constant of thin film samples. But in all these cases the samples are not so thin as to necessitate substrate for mechanical support.

Considerably lower values of  $\epsilon'$  have been reported in general for ferroelectric planar capacitors at microwave frequencies by Vendick.<sup>5</sup> The lowering of  $\epsilon'$  for a thin film sample compared to the bulk sample had been reported earlier by Bursian et al.<sup>6</sup> from R.F. measurements. This has been attributed due to presence of the ferroelectric surface.

It is necessary to have comparative measurement analysis of complex dielectric constants of high permittivity thin film samples having identical chemical composition. The polifactorial physical aspects of the thin film samples are also involved in determining their complex dielectric constants. Besides, the normal sources of inaccuracy inherent in solving the electrodynamical working equations of  $\epsilon'$  and  $\tan\delta$  of the sample affect the accuracy of the parameter estimated.

It is therefore all the more necessary to have the comparative experimental study of this relatively less studied field.

## Basic New Methods

### Method I

In the first proposed effective method by the author for measuring complex dielectric constant of high permittivity thin films at microwave frequency range, the experimental sample, in the form of a planar capacitor, supported by means of a  $\lambda_g/2$  (where  $\lambda_g$  is the guided wavelength) substrate, is placed inside a waveguide as shown in Fig. 1. In order to reduce the size of the sample and to increase its effect on the measurable parameter the height of the experimental waveguide is reduced. It is necessary to keep the width of the silvered portion smaller than that of the waveguide, not only to satisfy the condition of quasi-static solution, but also because substantial loading of the waveguide along its X-axis will make the guide wavelength higher than the cut off wavelength of the waveguide.

The upper and lower XZ surfaces of the substrate are silvered in order to ensure good electrical contact with the two surfaces of the waveguide. Extension of the silvered portion on the two corresponding XY planes along the sample form the two electrodes as shown in figure 1a.

The planar capacitor together with its silvered portion forms a series LC circuit across the waveguide. If the length of the substrate is  $\lambda_g/2$  at the resonant frequency,  $\epsilon'$  of the sample can be determined by noting the resonant frequency. If the resonant frequency is not equal to  $\lambda_g/2$ , corresponding reactance correction is to be done for substrate for determining  $\epsilon'$  of the sample.

### Method II

Modified Druae's Method. The modified Lrude's method<sup>9</sup> consists in finding out the change in measurable parameters of the parallel-wire transmission line system loaded with substrate and that loaded with substrate along with the thin film sample.

The method of measurement as shown schematically in figure 2, can be utilised as a non-destructive method of measuring complex dielectric constant of thin film samples of integrated circuits.

### Method III

Waveguide loaded with multilayered dielectric slab. This method is essentially the extension of any conventional partially

dielectric loaded waveguide method.  $\epsilon'$  and  $\epsilon''$  are determined by noting the phase and attenuation constants of the partially loaded waveguide with a thin film sample supported by means of substrates of known  $\epsilon^*$  and dimensions.

### Theory

#### Method 1

The exact equivalent circuit of the sample along with the substrate can be represented as shown in Figure 1c.  $X_L$  represents the inductive reactance of the silvered electrode of the planar capacitor.  $X_C$  - capacitive reactance of the planar capacitor.  $X_{L1}$  - interface reactance of the loaded and the unloaded waveguide.

Effect of this reactance was neglected in the earlier work.<sup>4</sup> Further approximation was also made by assuming that the length of the substrate is equal to  $\lambda_g/2$  at the resonance frequency, so that it serves only as a mechanical support for the sample. However, when this condition is not satisfied, the substrate will offer additional reactance.

Resonance frequency [and correspondingly the expression for  $\epsilon$ ] for the above equivalent circuit can be determined from the lumped circuit theory.

Inductive reactance of the silvered portion can be determined with the help of the equation:

$$X_L = \frac{a \rho_w}{2 \lambda_g} [-2 + 2.303 \log (8a/\pi L)] \quad (1)$$

where  $\rho_w$  - characteristic impedance of the waveguide,  $a$  - width of the waveguide,  $L$  - length of the planar capacitor, and  $\lambda_g$  - guide wavelength of the partially loaded waveguide.

$$X_C = \frac{1}{\omega C_p}$$

capacitance of the planar capacitor can be determined by means of conformal transformation:<sup>4</sup>

$$C_p = \frac{\epsilon' \epsilon_0 L}{[0.885 + d/t]} \quad (2)$$

$L$  - length of the planar capacitor in meter,  $\epsilon_0$  - absolute permittivity of free space,  $d$  - separation between planar capacitors,  $t$  - thickness of the sample.  $X_{L1}$  of the interface reactance can be determined from [7]; for sample thickness negligibly small compared to substrate thickness.

Additional reactance of the substrate when its length is not equal to  $\lambda_g/2$ ,<sup>8</sup>

$$X_s = j Z_0 \tan \beta l_s; \text{ for } l_s \neq \lambda_g/2$$

$$Z_0 = \eta \frac{k}{\beta}$$

$$\eta = 377 \text{ ohms} \quad (3)$$

$$\beta = \frac{2\pi}{\lambda_g}; \quad k = \frac{2\pi}{\lambda_0}$$

$$\lambda_g = \lambda_0 (1 - [\frac{2p(1+r)}{ka}])^{-1/2} \quad (4)$$

$$r = L/a/(1-L/a)$$

where  $p$  is the solution of the equations:

$$\frac{\tan p}{p} = r \frac{\cot q}{q} \quad (5)$$

$$\text{and } q^2 = r^2 p^2 + \frac{\epsilon_s - 1}{4} [\frac{rk}{1+r}]^2$$

Subject to the condition  $0 \leq q \leq \pi/2$

$$-\alpha \leq p^2 \leq (\pi/2)^2$$

#### Method 2

$\epsilon'$  and  $\epsilon''$  of the sample can be determined from the following equations.<sup>9</sup>

$$y_1 - y_0 = (\epsilon' - 1) d \quad (6)$$

$$y_2 - y_0 = \epsilon'' d \quad (7)$$

$y_0$  is the distance of the bridge from the boundary of the dielectric layer in the case of air dielectric, and  $y_1$  in the case of real dielectric sample.  $d$ , the sample width. In the same manner, we find the width of the resonance curve, if the distance of the plunger from the dielectric layer, for the condition, when the energy of oscillation drops to half the value, is denoted by  $y_2$ . If the corresponding displacements for the double-layered sample are  $y_1$ ,  $y_2$  and  $y_0$  and effective dielectric constants of the sample are  $\epsilon'_{TOT}$ , where

$$\epsilon'_{TOT} = \frac{(\epsilon'_{film} t_{film} + \epsilon'_{substrate} t_{substrate})}{t_{film} + t_{substrate}}$$

$$y_1' - y_0 = (\epsilon'_{TOT} - 1) d \quad (8)$$

$$y_2' - y_0 = \epsilon''_{TOT} d \quad (9)$$

Since  $t_{film}$ ,  $\epsilon_{substrate}$ ,  $t_{substrate}$  are known,  $\epsilon'_{film}$ ,  $\epsilon''_{film}$  can be determined from four measurements.

#### Method 3

With reference to the figure 3 of the multilayered dielectric slab loaded waveguide the  $\epsilon'$  and  $\epsilon''$  of the thin film material can be determined by measuring the phase constant  $\beta_r$  and the attenuation constant  $\beta_i$  of the transmission system, with the help of the following equation:<sup>7</sup>

$$\epsilon' = \frac{1}{k_0^2} \left[ \frac{2}{a t (\alpha^2 + n^2)} \left( \alpha (A+C) + \frac{2t}{a} (P P_1 - Q Q_1) \right) \right. \\ \left. + \eta (B+D + \frac{2t}{a} (P Q_1 + P_1 Q)) + \beta_r^2 - \beta_i^2 \right]$$

$$\epsilon'' = \frac{1}{k_0^2} \left[ \frac{2}{a t (\alpha^2 + n^2)} \left( \alpha (B+D + \frac{2t}{a} (P Q_1 + Q P_1)) \right) \right. \\ \left. - \eta (A+C + \frac{2t}{a} (P P_1 - Q Q_1)) - 2\beta_r \beta_i \right]$$

where  $k_0$  = free space wave number,  $P = \text{Re} [(k_0^2 - \beta^2)^{1/2} a/2]$ ,  $Q = \text{Im} [(k_0^2 - \beta^2)^{1/2} a/2]$ ,  $P_1 = \text{Re} [(k_0^2 \epsilon_s - \beta^2)^{1/2} a/2]$ ,  $Q_1 = \text{Im} [(k_0^2 \epsilon_s - \beta^2)^{1/2} a/2]$ ,  $\beta = \beta_r - j\beta_i$ .

$$A = \frac{Q \sinh 2Q_1 \cos 2P_1 - P \cosh 2Q_1 \sin 2P_1}{4 (\sinh^2 Q_1 \sin^2 P_1 + \cosh^2 Q_1 \cos^2 P_1)}$$

$$B = \frac{P \sinh 2Q_1 \cos 2P_1 + Q \cosh 2Q_1 \sin 2P_1}{4 (\sinh^2 Q_1 \sin^2 P_1 + \cosh^2 Q_1 \cos^2 P_1)}$$

Interchanging the positions in the above rela-

tions between P and P<sub>1</sub> and between Q and Q<sub>1</sub>, we obtain C and  $\nu$  respectively.

$$\alpha = \frac{(\cosh^2 a_4 \cos^2 a_2 - \cosh^2 a_3 \cos^2 a_1)}{(\cosh a_3 \cosh a_1 + \cosh a_4 \cosh a_2)^2 + (\sinh^2 a_3 \sin^2 a_1 - \sinh^2 a_4 \sin^2 a_2)} \dots$$

$$\dots \frac{(\sinh a_4 \sinh a_2 + \sinh a_3 \sinh a_1)^2}{(\sinh a_4 \sinh a_2 + \sinh a_3 \sinh a_1)^2}$$

$$\eta = \frac{1}{2} \frac{\sinh 2a_3 \sin 2a_1}{(\cosh a_3 \cosh a_1 + \cosh a_4 \cosh a_2)^2 - \sinh 2a_4 \sin 2a_2} \dots$$

$$\dots \frac{(\sinh a_4 \sinh a_2 + \sinh a_3 \sinh a_1)^2}{(\sinh a_4 \sinh a_2 + \sinh a_3 \sinh a_1)^2}$$

where  $a_1 = P+P_1$ ,  $a_2 = P-P_1$ ,  $a_3 = Q+Q_1$ ,  $a_4 = Q-Q_1$ .

### Experimental Results

Experimental results are obtained for BaTiO<sub>3</sub> thin film of thickness 20 $\mu$ , developed by means of lapping technique, which ensures that the chemical composition of BaTiO<sub>3</sub> and consequently polarizability of material is not likely to be changed.  $\epsilon'$  of this sample as measured by Method 1 is equal to 1008 when the correction for interface reactance and substrate reactance are taken into account. Considerable lowering of  $\epsilon'$  of thin film compared to that of the bulk sample has been attributed to the presence of surface layer, as already reported by Vendick et al.<sup>5</sup> and Bursian et al.<sup>6</sup>

Experimental samples of lead-niobate was formed on alumina and quartz substrate by means of evaporation technique, that may subsequently change the chemical constitution of the material.

Value of  $\epsilon'$  of sample of thickness 1  $\mu$  at room temperature in the form of planar capacitor<sup>4</sup> was equal to 92. This measurement at radio frequency and at room temperature was done by means of usual Q-meter.  $\epsilon'$  of this bulk sample at radio frequency is around 200 at room temperature. Measurement of  $\epsilon'$  of film of lead niobate by these methods are at around 80. [A detailed table of experimental results of better accuracy is to be included during oral presentation of the paper and to be published (if accepted) in IEEE Transaction MTT. December 1979 Symposium issue.]

### Conclusion

Any microwave method of measurement that enables to determine complex dielectric constant of thin films has immense scientific importance.

Since this subject has so far not been investigated at all and sources of uncertainty due to polifactorial physical aspects of the sample fabrication is involved, it is important to systematize the subject through comparative experimental measurement of identical samples that may exclude the sources uncertainty due to chemical decomposition of molecular structure (that effectively

changes the polarisability) of the sample.

Thus the systematic study of the dielectric properties of these samples will help to apply them in many solid state devices and circuits.

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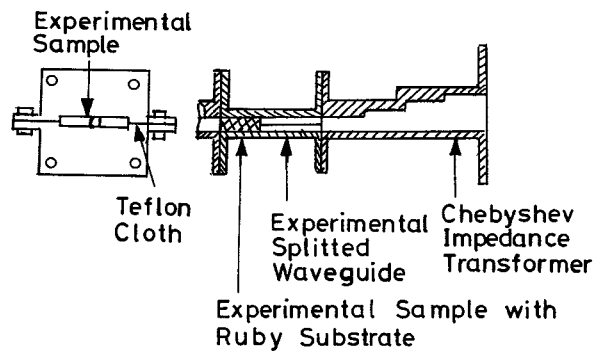


Fig.1 Experimental Set-Up

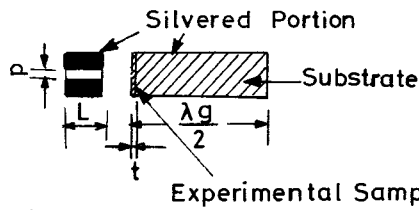


Fig.1a Schematic Representation of the Experimental Sample with the Substrate

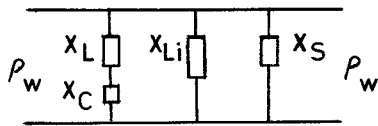


Fig.1c Equivalent Circuit of Method I

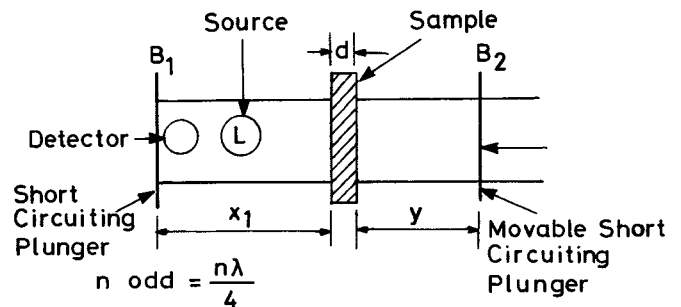


Fig.2 Schematic Representation of Experimental Set-Up

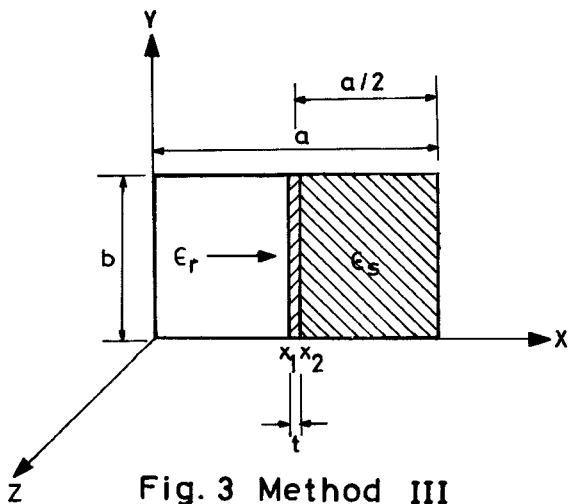


Fig.3 Method III