Electrodeless Measurement Technique of Complex Dielectric Permittivity of High-K Dielectric Films in the Millimeter Wavelength Range

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Abstract—An electrodeless measurement technique of complex dielectric permittivity of high-K dielectric films is described. The technique is based on a quasi-optic Fabry-Perot resonator and modified for investigation of two-layer dielectric structures — substrate/K-film. This procedure is destined to be used for providing a simple intermediate control of parameters of high-K films before the following technological process. Regimes of measurements providing the most sensitive conditions for definition of film parameters are considered. The proposed method is tested on two-layer structures with well-known parameters and is used for characterization of ferroelectric (Ba,Sr)TiO₃ films in the millimeter wavelength range (∼50 GHz).

1. INTRODUCTION

The characterization of microwave parameters of dielectric films with elevated values of the permittivity is required for different types of microwave (MW) devices (MOSFET, MEM RF switches, MW-photonic elements-electro-optic modulators etc.). The development of technology required to create new materials and components for MW devices is associated with instruments adapted for fast and accurate MW measurement in order to control the parameters of the developed structures and to correct processing methods for their preparation.

One of the broadly used class of measurement procedures of high-K dielectric films is the measurement of metal/dielectric/metal (M/D/M) structures, which may be in the form of electrodynamically lumped (capacitors) or distributed (strip or coplanar lines and resonators) elements [1, 2]. The main disadvantages of this method are the uncertainty in the impact of the technological process of metal deposition on intrinsic (original) properties of dielectric film, and the problem of separation of microwave losses in dielectric, M/D interfaces and metal electrodes.

In order to avoid the above drawbacks, it is reasonable to use electrodeless measurements which allow characterization of the film itself, before the following technological process of subsequent layers deposition. To provide the electrodeless measurements of the dielectric permittivity (ε) and dielectric loss tangent (tan δ) of materials at microwave frequencies the metal cavity resonators are widely used [1–4]. However, for the millimeter and especially sub-millimeter wavelengths, the above method is faced with considerable difficulties: (i) the microwave surface resistance of cavity metal walls increases at higher frequencies that leads to the decrease of the quality (Q) factor of the resonator, i.e., the accuracy of measurements decreases, (ii) the installation of the tested sample into the cavity usually involves disassembling and reassembling which if done frequently inevitably results in a variation of resonator

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parameters, that in turn leads to the need for permanent calibration of the resonator, (iii) the frequency density of different resonance modes of the cavity is very high and the identification of the mode required is rather a complicated problem.

One of the most promising methods without the mentioned drawbacks is the use of the open quasi-optic Fabry-Perot resonator (hereinafter OR). OR are used on MW up to hundreds GHz [5] for definition of electrodynamical parameters of solid substances, gases and plasma. In accordance with [3] the Q-factor for empty OR operating at millimeter wave frequencies is typically in the range $(1-2) \times 10^5$ and can be increased by a factor of $\sim 5$ employing so-called Bragg reflectors [6]. There are a set of works devoted to measurements of the complex dielectric permittivity on the base of OR. In [7] the measurements of one-layer materials with low values of $\varepsilon$ (quartz and Teflon disks) are presented at 20–40 GHz. The aim of the work is to determine the measurement error as a function of disk diameter. In [8] the possibility of very precise measurements at Ka band is demonstrated for a set of one layer dielectric materials with $\varepsilon < 10$. Note that many of recent works are devoted to increasing the measurement’s accuracy of electrodynamic parameters of one layer solid materials with low dielectric constant [9–11].

In [12], the theory of OR for measurements of multilayer dielectric structure is well developed, and its correctness is confirmed by 35 GHz measurements of a two-layer structure ($\text{Al}_2\text{O}_3$ substrate/(Ba,Sr)TiO$_3$ film) with thickness of (Ba,Sr)TiO$_3$ (BSTO) film $\sim 50 \mu$m. Our present work which is based on the practically classical approach of [5,6,12,13] is focused on a more detailed consideration of the most sensitive regimes of measurements corresponding to different values of substrate thicknesses and dielectric constant of the high K-film.

The proposed method is tested on two-layer structures with known parameters ($\text{SiO}_2$, LaAlO$_3$, $\text{Al}_2\text{O}_3$) and is used for definition of parameters of ferroelectric BSTO films (thickness $\sim 5 \mu$m, $\varepsilon > 400$) at frequency $\sim 50$ GHz. This procedure is destined to be used for providing a simple intermediate control of parameters of high-K films before the following technological processes.

2. THEORETICAL ASPECTS

The schematic of OR with the sample located on the flat mirror is presented in Fig. 1. The sample is a two-layer structure — substrate/high K-film. The microwave properties of substrate are known from handbooks or on the base of preliminary measurement of the substrate without film in open resonator. Installation of the sample changes the resonance frequency and to restore the mode of measurements the frequency shift is compensated by the movement of the plane mirror at a distance $p$. Theoretical approach of measurements is based on results of theory developed by [13] for E- and H-field distribution in open resonator with one layer dielectric. Relationship between the resonance frequency

![Figure 1. Schematic of microwave OR with a two-layer sample.](image-url)
(f) and the permittivity of the film ($\varepsilon_f$) on the substrate ($\varepsilon_s$) can be obtained on the base of boundary condition equations for three areas of material (substrate/film/air) between metal mirrors of OR and impedance transformation approach (see for example [14]). Solution of the system of equations results in a transcendental equation, which provides the relation between resonance frequency and permittivity of the film:

$$\frac{1}{\sqrt{\varepsilon_f}} \cdot \frac{1}{\sqrt{\varepsilon_s}} \tan (\sqrt{\varepsilon_s} kt) + \frac{1}{\sqrt{\varepsilon_f}} \tan (\sqrt{\varepsilon_f} ka) = - \tan (kd + \Psi),$$  

where: $t$ and $a$ — thicknesses of substrate and the film respectively, $k$ — the propagation constant in the free space, $\psi$ — the value correcting the phase shift at the plane mirror taking into account the spherical shape of the wave front (see in [15]), $d = D - p - t - a$ is the distance between mirrors with sample after the resonator adjusting on resonance frequency of empty OR.

The definition of $\varepsilon_f$ allows to define the so-called coefficient of the film inclusion (electric energy filling factor) ($\xi_f$) in the resonator. The coefficient is defined as the ratio of electric field energy stored in the film to the total energy of the electric field stored in the resonator:

$$\xi_f = \frac{W_f}{W_f + W_s + W_a},$$  

where $W_f$, $W_s$ and $W_a$ — energies of the electric field stored in the film, the substrate and the air regions respectively. These values are calculated by the volume integration of the E-fields strength distribution in OR with sample.

On the base of the simplest equivalent circuit presentation of the resonator, the dielectric losses of the film located on the substrate can be defined as:

$$\tan \delta_f = \frac{1}{\xi_f} \left( \frac{1}{Q_0} - \frac{1}{Q_{00}} - \xi_s \tan \delta_s \right),$$  

where: $Q_0$ — the unloaded quality factor of the open resonator with substrate/film sample, $Q_{00}$ — the unloaded Q-factor of the empty resonator without sample, $\xi_s$ — coefficient of the inclusion for the substrate, which is defined by equation analagical to (2), $\tan \delta_s$ — dielectric losses in the substrate.

It is clear from Eq. (3) that the correct definition of inclusion coefficients ($\xi_s$, $\xi_f$) plays a dominant role in the procedure of measurements. At first glance, it would seem that the increase of the thickness of the substrate without film must result in an increase of the inclusion coefficient of substrate. However, experimental and modeling data demonstrate the periodic $\xi_s(d)$ dependence (Fig. 2), which is

![Figure 2. Inclusion coefficient of substrate ($\varepsilon_s = 10$) as a function of substrate thickness.](image)
explained by the periodically changing of E-field coupling between substrate and air arias. Calculation demonstrates that maximums of $\xi_s(d)$ dependence corresponds to the maximums E-fields strength at the substrate surface, that is the most sensitive regime for measurements of film located on the substrate. This fact is illustrated by $\xi_f(\varepsilon_f)$ dependences (Figs. 3(a), (b)) for films of different thicknesses located on the substrate with electrical thicknesses $\lambda/4$ and $\lambda/2$. The thickness of $(2n - 1)\lambda/2$ (where $n$ is an integer) provides a minimum of the electric field at the substrate-film boundary, and accordingly, the lowest value of inclusion coefficient in comparison with $(2n - 1)\lambda/4$ conditions. One can conclude that to provide the most accurate measurement of film parameters ($\varepsilon_f$, $\tan\delta_f$) after the preliminary substrate measurement without film ($\varepsilon_s$, $\tan\delta_s$), the final film measurements must be done at frequencies corresponding to $(2n - 1)\lambda/4$ conditions for the substrates. If the thickness of the substrate does not correspond to the condition of maximum sensitivity of measurements of the film, the additional layer of substrate material of required thickness must be located on the metal mirror under the substrate/film structure.

![Figure 3](image)

**Figure 3.** Inclusion coefficient of the film as a function of it’s permittivity at different film thicknesses. Substrate ($\varepsilon = 10$) with thickness (a) $\lambda/4$ and (b) $\lambda/2$.

### 3. EXPERIMENTS

OR used in experiments is characterized by the following parameters: plane mirror diameter 120 mm, diameter aperture of concave mirror 200 mm and its curvature is of 139 mm. OR is destined to be used in the frequency range of $f = (40 \div 75)$ GHz. Unloaded quality factor of the empty OR is of $Q_{00}$ (60 GHz) $= (5–6) \times 10^4$. The flat mirror movement is provided by the micrometer scale mechanical screw. Mode TEM$_{00q}$ was used for investigation. The mode identification is done by $|S_{21}|$ measurements of OR at different positions of small disturbing absorber ball ($\geq 1.0$ mm) above the flat mirror. Correctness of Eqs. (1) and (3) to define the parameters of the film layer on the substrate was confirmed by the measurements of structures consisting of two layers with well-known MW parameters of each layer. For samples containing the different combination of layers (SiO$_2$, LaAlO$_3$, Al$_2$O$_3$) with scale of thicknesses ($0.2 \div 1.5$ mm) the simulation of parameters of layer which was considered as an “unknown layer” have been done on the base of our measurements and Eqs. (1), (3) in the frequency range (45 $\div$ 70) GHz. For these “unknown layers” the errors of $\varepsilon$ and $\tan\delta$ in comparison with handbooks data were not more than 1% and 5% respectively.

Results of the measurements at frequency $\sim 50$ GHz of three types of (Ba$_{0.5}$Sr$_{0.5}$)TiO$_3$ films with different concentration of Mg$_2$TiO$_4$ dopants [16] and with thickness of 5 $\mu$m on MgO substrate (0.5 mm) destined to be used in electro-optical devices are demonstrated in Fig. 4. Along with the results of electrodeless measurements at $\sim 50$ GHz the data of definition of $\varepsilon_f$ and $\tan\delta$ on the base of lower frequency measurements of capacitance structures Metal/BSTO/Metal are also presented in Fig. 4.
Figure 4. Results of electrodeless measurements ((a) $\varepsilon_f$ and (b) $\tan\delta_f$) of three types of (Ba$_{0.5}$Sr$_{0.5}$)TiO$_3$ films with different dopant concentration at frequency $\sim$ 50 GHz (red icons) and generalized frequency dependences of dielectric parameters of films (violet filling — guide for eyes).

4. DISCUSSION

Theoretical dependences of inclusion coefficients ($\xi_s$, $\xi_f$) (Figs. 2, 3) were used to provide the most sensitive regimes of measurements. Operation frequency and substrate thickness correspond to conditions close to the first maximum of $\xi_s(t)$ (Fig. 2) that determines the value of $\xi_f = 0.003 \div 0.005$ for 5 µm K-films with $\varepsilon = (400 \div 800)$ (Fig. 3(a)). The unloaded Q factor of OR with different samples corresponds to $Q_0 = 3500$–5000. Note that for high-K films with significantly higher $\tan\delta$ the use of the measurement regimes close to maximums of dependencies $\xi_s(t)$ are problematic due to degradation of OR resonance. In this case for reliable visualization of resonance curve it is necessary to use the regime corresponding to lower values of $\xi_s$ and $\xi_f$, that is the range between maximum and minimum of $\xi_s(t)$ dependence. Obviously that results in decrease of accuracy of measurements.

Due to technological and constructive differences of structures tested at different frequencies, it is not correct to establish quantities frequency dependencies (law of frequency dispersion) for each BSTO composition. However, it is clear the results of electrodeless measurements qualitatively correspond to data of lower frequency measurements and in any case do not contradict them. In fact the frequency dispersion of dielectric constant is practically absent for composition of $\varepsilon_f \sim 400$, but clearly clarified for compositions with higher values of dielectric constant, that corresponds to the physical nature of
ferroelectrics [17, 18]. The results of electrodeless measurements of dielectric losses also correspond to nature of ferroelectrics: the higher dielectric constant — the higher the dielectric losses.

The main source of basic errors is connected with correct definition of the film inclusion coefficient and in accordance with our simulation for the most critical situation corresponding to thin film (0.5 µm) with low permittivity ($\varepsilon_f = 100$) the relative errors are not more than $\Delta\varepsilon_f/\varepsilon_f = 5\%$ and $\Delta\tan\delta_f/\tan\delta_f = 10\%$. For thick films ($\geq 10 \mu m$) with the same permittivity the errors are less than 2% for permittivity and 5% for losses.

5. CONCLUSION

The developed technique based on the quasi-optical Fabry-Perot resonator provides the dielectric constant and dielectric loss tangent measurements of high-K films deposited on the substrate in the mm-wave frequency range with a rather high accuracy. To provide the most accurate measurement of film parameters ($\varepsilon_f, \tan\delta_f$), the measurements must be done at frequencies corresponding to $(2n - 1)\lambda/4$ conditions for the thickness of substrates. The developed technique can be used for both the laboratory study of high-K films and due to its simplicity for fast (express) control measurements in serial production. Results of measurements of BSTO film parameters ($\varepsilon, \tan\delta$) at frequency $\sim 50 \text{GHz}$ can be interesting for designers of ferroelectric MW devices.

ACKNOWLEDGMENT

This work was supported by the Ministry of Education and Science of the Russian Federation in the framework of “Research and development in priority areas of advancement of the Russian scientific and technological complex for 2014–2020”, agreement No. 14.608.21.0002 of 27.10.2015 (unique number of agreement RFMEFI60815X0002).

REFERENCES


