Progress In Electromagnetics Research M, Vol. 52, 161–167, 2016

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)TiO_3 films in the millimeter wavelength range ($\sim 50\,\mathrm{GHz}$).

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 ($\tan\delta$) 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

Received 5 October 2016, Accepted 24 November 2016, Scheduled 8 December 2016
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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 ~ 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 ε (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 (Al_2O_3 substrate/(Ba,Sr)TiO_3film) 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 (SiO_2, $LaAlO_3$, Al_2O_3) and is used for definition of parameters of ferroelectric BSTO films (thickness $\sim 5\,\mu$m, $\varepsilon > 400$)at frequency ~ 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

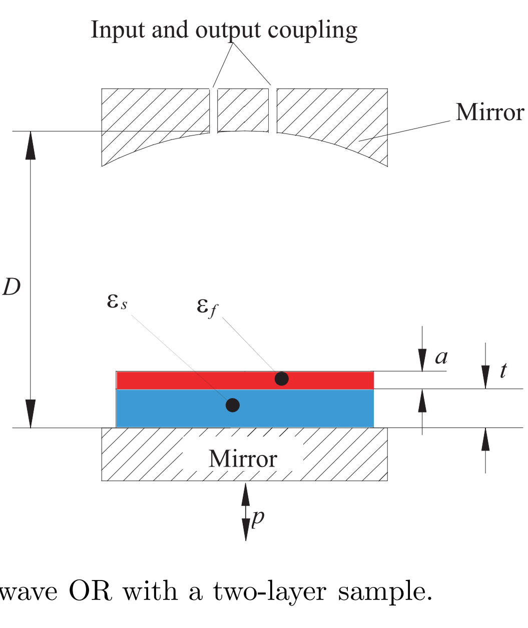

Figure 1. Schematic of microwave OR with a two-layer sample.

(f) and the permittivity of the film (ε_f) on the substrate (ε_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:

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

where: t and a — thicknesses of substrate and the film respectively, k — the propagation constant in the free space, ψ – 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 ε_f allows to define the so-called coefficient of the film inclusion (electric energy filling factor) (ξ_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:

$$\xi_f = \frac{W_f}{W_f + W_s + W_a}, \tag{2}$$

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:

$$\tan\delta_f = \frac{1}{\xi_f}\left(\frac{1}{Q_0} - \frac{1}{Q_{00}} - \xi_s tan\delta_s\right), \tag{3}$$

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, ξ_s — coefficient of the inclusion for the substrate, which is defined by equation analogical to (2), $\tan\delta_s$ — dielectric losses in the substrate.

It is clear from Eq. (3) that the correct definition of inclusion coefficients (ξ_s, ξ_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

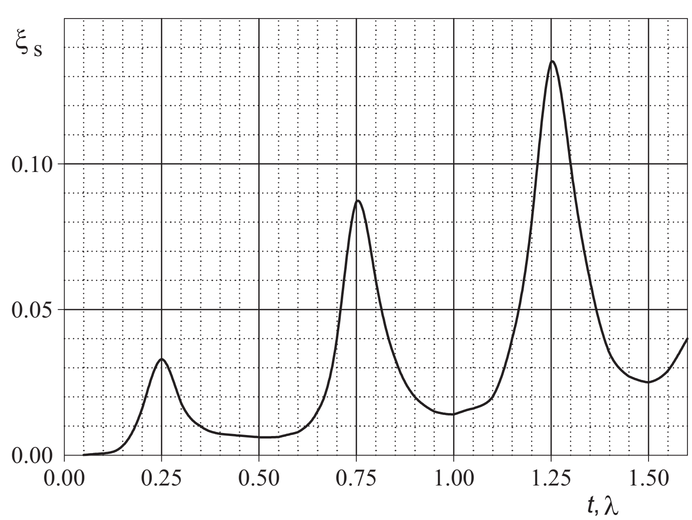

Figure 2. Inclusion coefficient of substrate ($\varepsilon_s = 10$) as a function of substrate thickness.

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 (ε_f, $\tan\delta_f$) after the preliminary substrate measurement without film (ε_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.

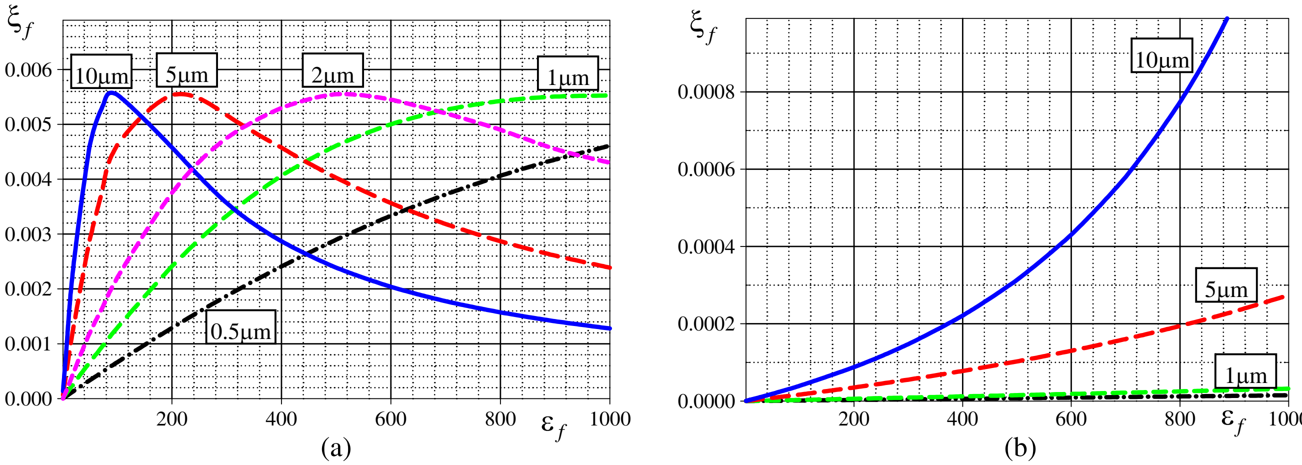

Figure 3. Inclusion coefficient of the film as a function of it's permittivity at different film thicknesses. Substrate ($\varepsilon = 10$) with thickness (a) $\lambda/4$ and (b) $\lambda/2$.

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 (∅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_2O_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 ε and $\tan\delta$ in comparison with handbooks data were not more than 1% and 5% respectively.

Results of the measurements at frequency ~ 50 GHz of three types of $(Ba_{0.5}Sr_{0.5})TiO_3$ films with different concentration of Mg_2TiO_4 dopants [16] and with thickness of 5 μm 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 ~ 50 GHz the data of definition of ε_f and $\tan\delta$ on the base of lower frequency measurements of capacitance structures Metal/BSTO/Metal are also presented in Fig. 4.

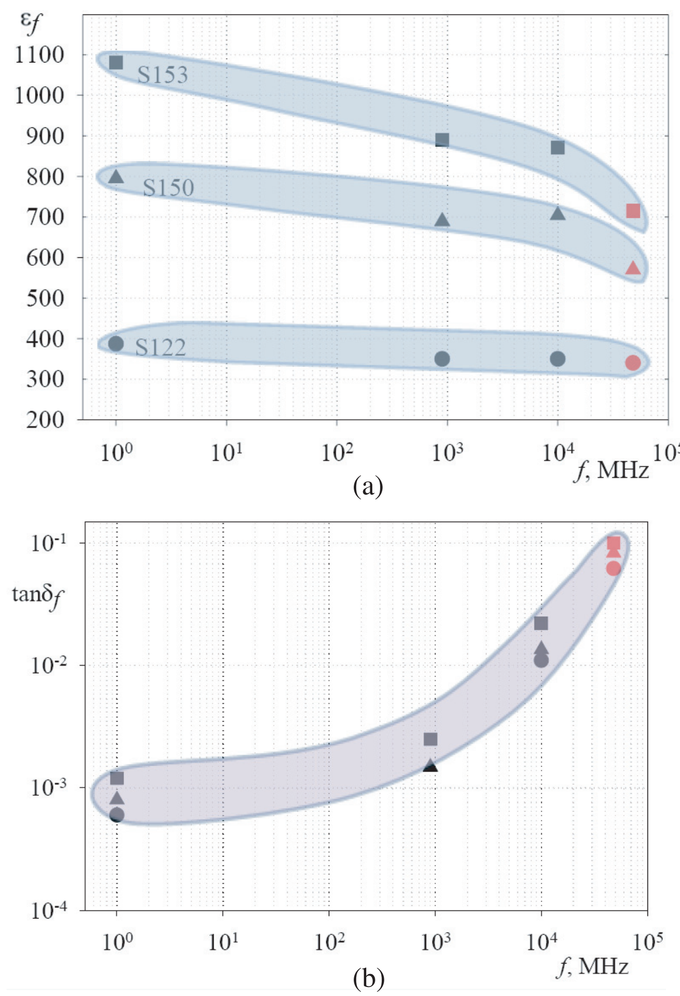

Figure 4. Results of electrodeless measurements ((a) ε_f and (b) $\tan\delta_f$) of three types of $(Ba_{0.5}Sr_{0.5})TiO_3$ films with different dopant concentration at frequency $\sim$ 50 GHz (red icons) and generalized frequency dependences of dielectric parameters of films (violet filling — guide for eyes).

4. DISCUSSION

Theoretical dependences of inclusion coefficients (ξ_s, ξ_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 ξ_s and ξ_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 ($\geq 10$ μm) 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 (ε_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 (ε, $\tan\delta$) at frequency ~ 50 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

1. Chen, L. F., C. K. Ong, C. P. Neo, V. V. Varadan, and V. K. Varadan, *Microwave Electronics. Measurement and Materials Characterization*, John Wiley & Sons, Ltd., 2004.
2. Spartak, G., *Ferroelectrics in Microwave Devices, Circuits and Systems: Physics, Modeling, Fabrication and Measurements*, Springer Science & Business Media, 2009.
3. Jerzy, K., "Frequency domain complex permittivity measurements at microwave frequencies," *Measurement Science and Technolpgy, Institute of Physics, Meas. Sci. Technol.*, 17, 2006.
4. Lee, C.-S. and C.-L. Yang, "Thickness and permittivity measurement in multi-layered dielectric structures using complementary split-ring resonators," *IEEE Sensors Journal*, Vol. 14, No. 3, March 2014.
5. Clarke, R. N. and C. B. Rosenberg, "Fabry-Perot and open resonators at microwave and millimeter wave frequencies, 2–300 GHz," *Journal of Physics E: Scientific Instruments*, Vol. 15, 1982.
6. Krupka, J., A. Cwikla, M. Mrozowski, R. N. Clarke, and M. E. Tobar, "High Q-factor microwave Fabry-Perot resonator with distributed Bragg reflectors," *IEEE Trans. Ultrason. Ferroelectr. Freq. Control*, Vol. 52. 2005.
7. Yao, Y., H. Cui, J. Wang, E. Li, and B. Tao, "Broadband measurement of complex permittivity by an open resonator at 20–40 GHz," *International Conference on IEEE Communication Problem-Solving (ICCP)*, 2014.
8. Gui, Y. F., W. B. Dou, P. G. Su, and K. Yin, "Improvement of open resonator technique for dielectric measurement at millimeter wavelengths," *IET Microw. Antennas Propag.*, Vol. 3, No. 7, 2009.
9. Libich, J., P. Dvorak, P. Piksa, and S. Zvanovec, "Correction of thermal deviations in Fabry-Perot resonator based measurements of specific gases in millimeter wave bands," *Radioengineering*, Vol. 21, No. 1, 459, April 2012.

10. Hong, Y.-P., H. Koo, and M. J. Salter, "Measurement of high-permittivity dielectric characteristics at microwave and mm-wave frequencies," *Conference on Precision Electromagnetic Measurements (CPEM 2016)*, July 2016.
11. Quan, W., D. J. Downing, and M. NurulAfsar, "A signal processing approach for recovery of precision high-Q dielectric resonance profile measurements," *2016 Conference on Precision Electromagnetic Measurements (CPEM 2016)*, July 2016.
12. Anatoli, D. and G. Spartak, "Open resonator technique for measuring multilayered dielectric plates," *IEEE Transactions on Microwave Theory and Techniques*, Vol. 53, No. 9, 2005.
13. Yu, P. K. and A. L. Cullen, "Measurement of permittivity by means of an open resonator," *Proc. R. Soc. London: Ser. A.*, Vol. 380, 1982.
14. David, P., *Microwave Engineering*, 4th Edition, John Wiley & Sons Inc., 2012.
15. Cullen, A. L., "Millimeter-wave open-resonator techniques," *International Journal of Infrared and Millimeter Waves*, Vol. 10, Springer, 1983.
16. Andrey, K., K. Alexei, N. Elizaveta, O. Vitaly, and K. Dmitry, "Observation of an anomalous correlation between permittivity and tunability of a doped $(Ba,Sr)TiO_3$ ferroelectric ceramic developed for microwave applications," *Applied Physics Letters*, Vol. 95, 012908, 2009.
17. Vendik, O. G. and S. P. Zubko, "Ferroelectric phase transition and maximum dielectric permittivity of displacement type ferroelectrics $(Ba_xSr_{1-x}TiO_3)$," *Journal of Applied Physics*, Vol. 88, 5343, 2000.
18. Tagantsev, A. K., V. O. Sherman, K. F. Astafiev, J. Venkatesh, and N. Setter, "Ferroelectric materials for microwave tunable applications," *Journal of Electroceramics*, Vol. 11, 2003.