MEASUREMENTS OF MATERIALS AT MICROWAVE FREQUENCIES

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Key words: microwave frequency, electronic material, resonance measurement techniques

Abstract: Recent advances in measurements of various electronic materials at microwave frequencies are presented. Special attention is devoted to resonance techniques that are more sensitive and accurate than the transmission-reflection methods. Several specific measurement methods are described. Simultaneous use of whispering gallery and quasi TE modes allows for multi-frequency measurements of low loss materials. Modification of the split post dielectric resonator technique can be used for measurements of both permittivity and permeability of laminar low and medium loss metamaterial. Resistivity of conductive materials such as semiconductors metals and polymers can be measured in the range of several decades employing single post dielectric resonator technique.

Meritve materialov pri mikrovalovnih frekvencah

Kjučne besede: mikrovalovne frekvence, elektronski material, resonančne merilne tehnike

Izvleček: V članku predstavljamo napredne tehnike meritev karakteristik različnih elektronskih materialov pri mikrovalovnih frekvencah. Posebna pozornost je namenjena resonančnim tehnikam, ki so bolj občutljive in natančne kot oddajno-povratne metode. Opisanih je nekaj specifičnih metod merjenja, ki dovoljujejo meritve materialov z nizko izgubo pri različnih frekvencah. Spremenjena tehnika meritve z dielektričnim resonatorjem omogoča meritve dielektrične konstante in izgub v materialih s srednjimi in nizkimi izgubami. S to tehniko lahko merimo tudi upornost prevodnih materialov, kot so polprevodne kovine in polimeri v širokem območju.

1 Introduction

Measurement techniques of the complex permittivity and in some cases the complex permeability are described for the following four groups of materials:

a) Bulk low loss dielectric materials including ceramics and uniaxially anisotropic single-crystals;
b) Laminar type dielectric materials such as LTCC ceramics, PWB substrates and thin ferroelectric films;
c) Semiconductors and conductors;
d) Metamaterials

At frequency domain the complex permittivity of any linear material is generally defined as a tensor quantity describing relationship between the electric displacement ($\vec{D}$) and the electric field ($\vec{E}$) vectors (1) /1/.

$$\vec{D} = \tilde{\varepsilon}\vec{E} \qquad (1)$$

For passive reciprocal materials such as ionic dielectric single crystals permittivity tensor is symmetric and can be diagonalized which means that at certain specific coordinate system it takes the diagonal form (2)

$$\tilde{\varepsilon} = \begin{bmatrix} \varepsilon_{11} & 0 & 0 \\ 0 & \varepsilon_{22} & 0 \\ 0 & 0 & \varepsilon_{33} \end{bmatrix} \qquad (2)$$

For polycrystalline materials, glasses, plastics and some crystals (e.g. having cubic crystallographic structure) all diagonal elements become identical and the complex permittivity becomes scalar quantity. The complex permittivity of an isotropic material in general can be written as

$$\begin{aligned} \varepsilon &= \varepsilon_0 \varepsilon_r = \varepsilon_0 (\varepsilon_r^{'} - j\varepsilon_{rd}^{''} - j\frac{\sigma}{\omega\varepsilon_0}) = \\ &= \varepsilon_0 \varepsilon_r^{'} (1 - j\tan\delta) \end{aligned} \qquad (3)$$

where - tan δ - total dielectric loss tangent

$$\tan\delta = \tan\delta_d + \frac{\sigma}{\omega\varepsilon_0 \varepsilon_r^{'}} \qquad (4)$$

ε_r - relative complex permittivity
ω - angular frequency
σ - conductivity
$\varepsilon_0 = 1/(c^2\mu_0) \approx 8.8542x10^{-12}$ (F/m) - permittivity of vacuum
$\tan\delta_d$ - dielectric loss tangent associated all other dielectric loss mechanisms except conductivity

When we measure the loss of a dielectric at a single frequency we cannot, in general, distinguish between them. Phenomenologically they all give rise to just one measurable quantity: namely the total measured loss tangent. Some materials commonly used at microwave frequencies such as ferrites, as well as metamaterials, exhibit magnetic properties that must be considered in measurements of their permittivity. Permeability tensor $\tilde{\mu}$ describes relationship between the magnetic induction $\vec{B}$ and magnetic field $\vec{H}$ vectors (5).

$$\vec{B} = \tilde{\mu}\vec{H} \qquad (5)$$

The most important microwave applications of ferrites are related to their non-reciprocal properties. In a presence of static magnetic field magnetizing ferrite material along z-axis of Cartesian or cylindrical coordinate system permeability of ferrite material is represented by Polder's tensor (6) /6/. Off-diagonal components of Polder's tensor are purely imaginary but they don't describe any magnetic losses since they appear with opposite signs. If ferrite material is lossy than particular tensor components (μ, κ, μ_z) become complex.

$$\overleftrightarrow{\mu} = \mu_0 \begin{bmatrix} \mu & j\kappa & 0 \\ -j\kappa & \mu & 0 \\ 0 & 0 & \mu_z \end{bmatrix} \qquad (6)$$

This paper is principally devoted to the resonance techniques intended for the complex permittivity and the complex permeability measurements. These techniques are more sensitive and accurate than commonly used transmission-reflection methods.

Measured quantities in resonance techniques are the resonant frequency and the Q-factor of a specific mode excited in the resonant structure containing a sample under test. The complex permittivity of the sample can be evaluated from these two measured quantities providing that all other parameters of the structure are known. These parameters include dimension of the structure, surface resistance of metal parts, coupling coefficients, radiation losses, and the complex permittivities of dielectric supports. Usually advanced electromagnetic simulations are necessary to determine the complex permittivity even for relatively simple and regular shape of samples under test.

Resonant cavities having axial symmetry are the most often used in the dielectric metrology. Cavities of this kind can operate on different modes but in practice one of the few first modes of the frequency spectrum are used.

2 Measuremnts of bulk low loss dilectric materials

Bulk, low loss materials are usually measured employing dielectric resonator techniques when the sample under test concentrates most of the electromagnetic energy. These days the most frequently used method to measure bulk low loss dielectrics is the $TE_{01\delta}$ mode dielectric resonator technique /2/ with geometry schematically shown in Fig.1a. The same structure can be used for multi-frequency measurements employing higher order TE /3/ and whispering gallery modes /4/, although Hakki-Coleman dielectric resonator technique /5/ is still frequently employed to measure the real part of the complex permittivity of low loss high permittivity ceramic materials. If the losses in samples under test increase then the Q-factor of the resonance structure becomes to small to be measured. In order to measure medium or even high loss bulk dielectric samples it is necessary to reduce the amount of the electromagnetic energy in the sample under test. This can be done for bulk samples in the way that is shown in Fig.1b /6/,/7/.

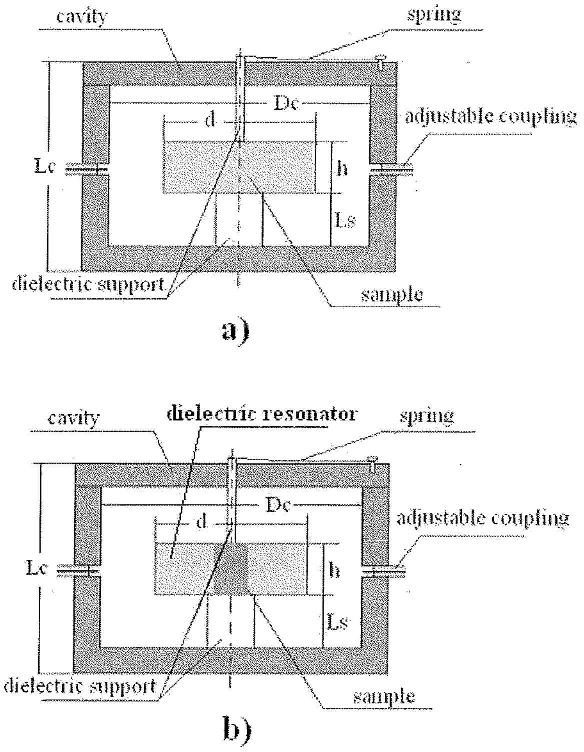


Fig. 1: Sketch of axially symmetric resonance structures used for measurements of bulk samples. a) TE or whispering gallery mode dielectric resonator intended for measurements of low loss samples, b) TE mode dielectric resonator intended for measurements of medium loss samples.

Measurement results of few bulk semi-insulating semiconductor and dielectric samples employing dielectric resonator techniques are shown in Figs.2-5.

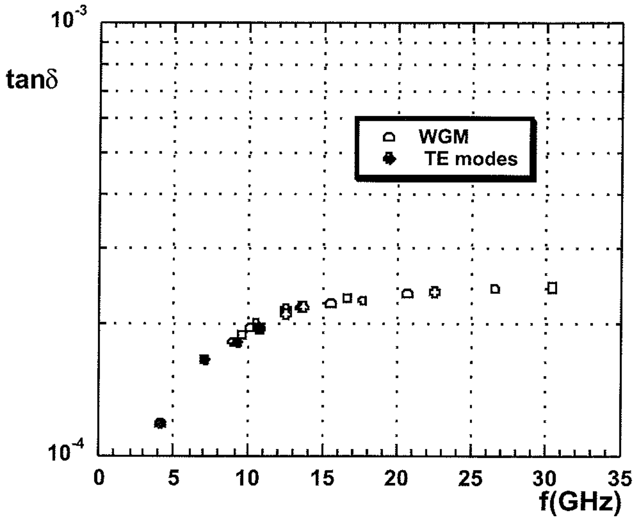
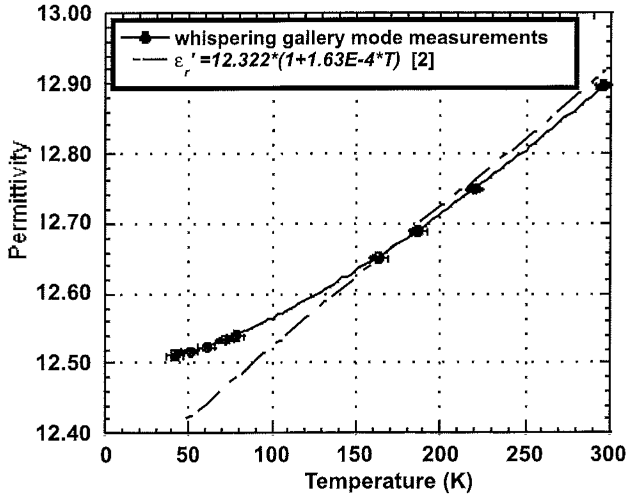


Fig. 2: Dielectric loss tangent for the GaAs sample versus frequency measured at room temperature employing several quasi TE and whispering gallery modes /8/.

Typical resolution of loss tangent resolution employing TE mode dielectric resonators technique with optimised enclosure is about 10^{-6} for high permittivity samples (ε_r>20) and uncertainty of the real part of permittivity measurements of the order of 0.3% (similar for low and high permittivities). For whispering gallery modes loss tangent resolution can be even better then 10^{-10} (dielectric loss tangent of high purity sapphire at 4 K) /4/.



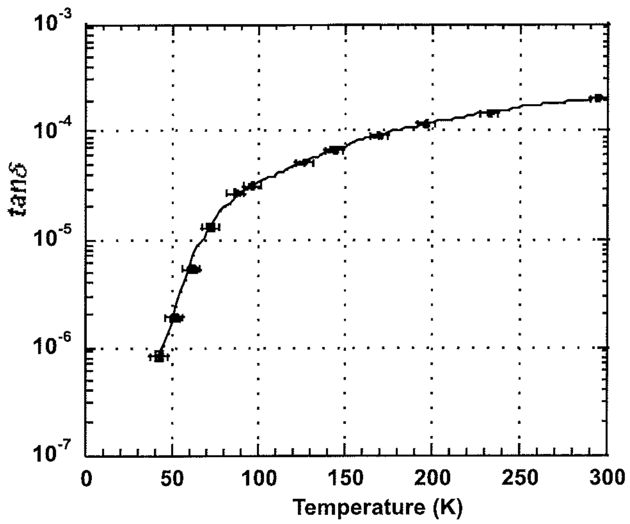


Fig. 3: Permittivity versus temperature for the GaAs sample. The experimental data points were extracted from measurements of the whispering gallery mode with frequencies near 18.9 GHz /8/.

Fig. 4: Measured and predicted theoretical loss at 3 GHz and zero bias for $KTaO_3$ /9/.

It should be pointed out that employing whispering gallery modes it is possible to measure two components of the complex permittivity tensor for oriented uniaxialy anisotropic crystals /10/.

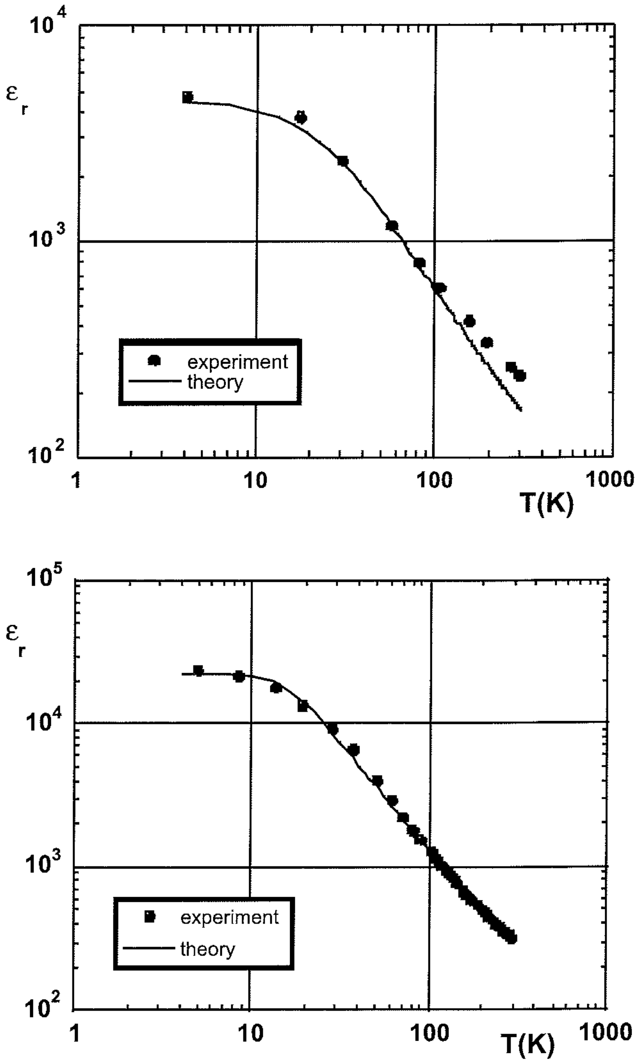


Fig. 5: Theoretical and measured real permittivities for high-purity single-crystals of $KTaO_3$ and $SrTiO_3$ /9/.

3 Measuremnts of laminar dielectric materials

The most convenient methods to measure the complex permittivity of isotropic laminar dielectric materials are the split cylindrical cavity method /11/ and the split post dielectric resonator method /12/. Schematic diagram of the split post dielectric resonator is shown in Fig. 6. In both methods the TE modes are employed that have only azimuthal component of the electric filed. This makes the methods no sensitive to the presence of air gaps between sample and the other parts of the resonance structures because due to their axial symmetry the electric field is tangential to the surface of test samples. Employing these methods one can measure permittivity to within 0.3%. The dielectric loss tangent resolution is typically better than 10^{-4}. Split post dielectric resonator can be also employed for the complex permittivity measurements of thin ferroelectric films /13/, as well as for the surface resistance measurements of thin conducting films having surface resistance larger than 3 kΩ /14/.

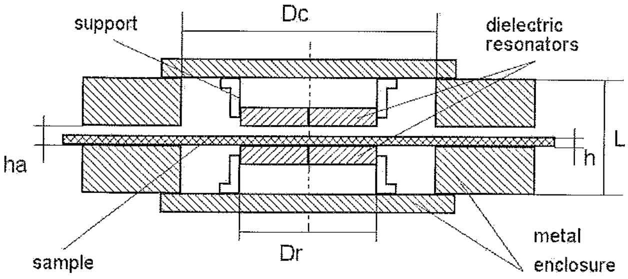


Fig. 6. Schematic diagram of split post dielectric resonator

4 Measurements of semiconductors and conductors

Contact-less conductivity measurements of semiconductors are very attractive especially for such materials for which it is difficult to create linear current contacts with four-point probe technique (e.g. SiC). This can be done with a single post dielectric resonator technique /15/. Schematic of the 10 GHz single post dielectric resonator intended for conductivity measurements of typical semiconductor wafers is shown in Fig.7.

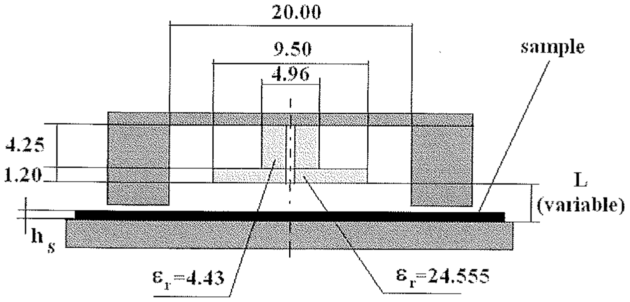


Fig. 7: Schematic of 10 GHz single post dielectric resonator intended for measurements of semiconductor wafers and thin conducting films deposited on dielectric substrates

Principles of conductivity measurements employing single-post dielectric resonators can be explained using graphs showing $TE_{01\sigma}$ mode resonant frequencies and Q-factors due to conductor losses (Q_S) in the sample versus conductivity for different thicknesses of samples. Such graphs are shown in Fig.8. One can observe that for σ<1 S/m Q_S-factors and resonant frequency shifts behave as for "proper dielectrics". In this conductivity range resonant frequency shifts depend on the real part of permittivity and thickness of samples, while Q_S-factors decrease with increase of conductivity. When σ>1000 S/m Q_S-factors and resonant frequency shifts behave as for metallic samples. In this case electromagnetic fields decay exponentially in the direction perpendicular to the surface of the sample (skin effect).

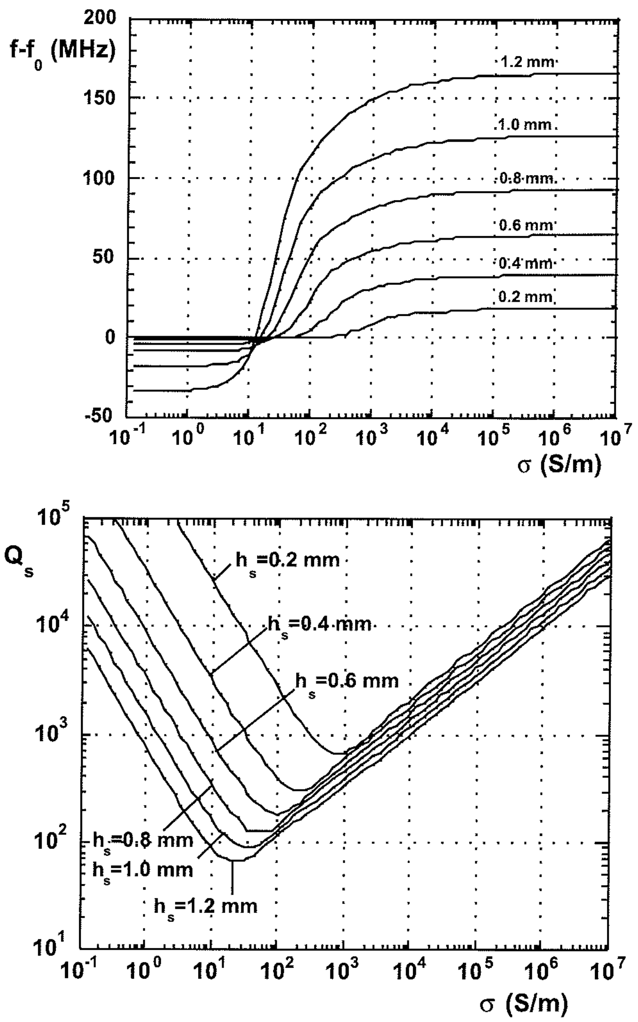


Fig. 8. Computed resonant frequency shifts and Q-factors due to conductor losses in the sample, (Q_S), for the single-post dielectric resonator shown in Fig. 7 with L=4.3 mm.

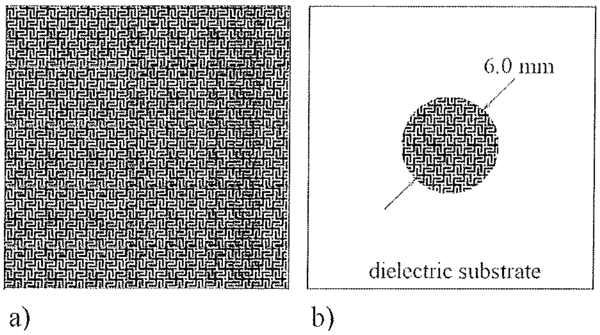
As one can notice unique determination of conductivity (resistivity) is possible if both the resonance frequency shift due to the presence of the sample and the Q-factor due to conductor losses in the sample are known.

The single post dielectric resonator technique can be also used for the measurements of the surface resistance of thin conducting films /14/.

5 Measurements of metamaterials

Metal films deposited on a dielectric substrate may exhibit dielectric properties that significantly deviate from those of the bulk metals /16/-/17/. Very rapid change of dielectric properties takes place near percolation threshold when films become very thin and have island structure. If one assumes that the island structure is periodic, such a patterned metal film can be considered as a 2D metamaterial /18/-/20/. Electromagnetic properties of periodic 2D heterogeneous structures consisting of unit cells of various shapes such as metal dots, rings, split rings, double split rings and many other ones are of interest for many applications including manufacturing of a bulk artificial dielectric /21/. If metal patterns posses high degree of symmetry than the in-plane electromagnetic properties of such created 2D metamaterial are expected to be isotropic. By stacking several layers of such 2D metamaterials one can obtain 3D metamaterials having uniaxialy anisotropy. It is known that the electromagnetic properties of such metamaterial can be characterized by the effective permittivity and the effective permeability. For more complicated structures that do not posses in plane symmetry it is necessary to introduce additional material parameters (material properties) that take into account the coupling between the electric displacement vector and the magnetic field as well as the coupling between magnetic induction and the electric field. Such metatamaterials are called bianisotropic. It is also possible to create isotropic 3D metamaterials by random mixing of dielectric and metal particles. Such isotropic metamaterials also exhibit both electric end magnetic properties but in this case permittivity and permeability are scalars. Measurements of both permittivity and permeability of metamaterials are difficult especially for relatively low loss materials.

I the recently published paper /23/ a method have been described that allows to measure the effective complex permittivity and the effective complex permeability of isotropic and uniaxialy anisotropic metamaterials. Separation of the complex permittivity from the complex permeability for a specific metamaterial was achieved performing measurements of the resonance frequencies and the Q-factors of a split post dielectric resonator with two samples having different diameters but identical film patterns as it is shown in Fig.9. Measurements of the resonance frequencies and Q-factors of empty substrates served as reference materials to determine the resonance frequency shifts and Q-factor changes due to the presence of the metamaterial only. electromagnetic Determinations of the effective complex permittivity and permeability were performed on the basis of rigorous modeling of the resonance structures containing the samples. To check the validity and accuracy of presented technique two reference "materials" were measured.



a) b)

Fig. 9: Two samples of metamaterial having the same pattern that are used to determine both permittivity and permeability of this metamaterial.

The first reference material was made from a single piece of transparency foil, 122 mm thick placed on the substrate. A small sample of reference "material" was just cut from the transparency foil in a 6 mm diameter circle. Measurements of the dielectric reference "material" should yield known results (permittivity of 3.18 and unity permeability). Due to unavoidable measurement uncertainties real measurements would differ slightly. The second reference magneto-dielectric material was yttrium-iron garnet YIG. Again two samples of this material, 508 mm thick, have been used in our measurements, one was 30 mm x 30 mm sample and the other 6 mm diameter sample.

In the addition two metamaterial samples were measured. These samples were made as a random mixtures of very fine Aluminium powder (with a mean particle size of the order of 1 mm) and a polymer (PMMA) deposited by painting onto transparency polyester foils. The results of measurements on reference materials and on metamaterials at frequency 4.9 GHz are summarized in Table 1. Measurements on the reference materials (PET and YIG) are consistent with the literature values. It should be noted that permittivities of metamaterials are very large (comparable to the permittivity of ferroelectrics). The effective permeabilities of both metamaterials are smaller than unity which is related to the eddy currents induced in metal particles.

Table 1. Results of permittivity and permeability determination for "normal" reference materials and planar, isotropic 3D metamaterials

Material	Re(ε_r)	Im(ε_r)	Re(μ_{ZY})	Im(μ_{ZY})	h_f (μ_m)
PET	3.145	-0.0154	1.00448	< 0.0001	122
YIG	15.45	-0.0011	0.64900	-0.00945	508
Metamaterial 1	341	-17.5	0.28900	-0.02870	7
Metamaterial 2	3146	-88	0.15100	-0.13400	4

6 Summary

This paper overviewed only small fraction of available techniques for measurement of material properties at microwave frequencies. A lot of techniques exist that were not mentioned in this paper that utilize microstrip, stripline and coplanar waveguide cells (both transmission line and resonant ones). Typically transmission/reflection technique are useful for characterization of high and medium loss materials and resonant techniques can be in principle used for measurements of materials having arbitrary losses. On the other hand resonant techniques are in most cases limited to one fixed frequency (although as it has been shown multi-frequency measurements in one measurements cell are possible) while transmission/reflection techniques can typically operate at broad frequency bands. One of the most important issues for all techniques is their sensitivity to the presence of air gaps between the sample and other parts of the measurement cell. Resolution of loss tangent measurements for arbitrary technique is associated with the presence of parasitic losses in measuring cells. They must be calculable and relatively small with respect to the losses in the sample to in order to measure precisely losses in the material under test.

Acknowledgments

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