Bulg. J. Phys. **45** (2018) 264–274

# Measurement of Uni- and Bi-Axial Dielectric Anisotropy of Crystalline Samples by Microwave Resonance Method

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Received: *March 21, 2018*

**Abstract.** Two variants of resonance measurement methods have been described in the paper for characterization of uni- and bi-axial dielectric anisotropy of crystalline samples in the microwave range using cylindrical resonators. The dielectric parameters of several known and some new, unknown crystalline samples have been determined by a pair of cylindrical resonators for modes with excited electric fields in two mutually perpendicular directions (based on the authors' two-resonator method) or by a fixed-mode resonator with different orientations of the sample. The proposed methods include applying of 3D electromagnetic simulators as an auxiliary tool. The obtained results are presented in tabular and graphical form and are compared and discussed. Some conclusions have been drawn related to the measurement accuracy of the applied methods and to the reliability of the results obtained for the anisotropy of different crystalline samples.

PACS codes: 77.22.d

## 1 Introduction

The present paper is related to determination of the dielectric anisotropy of crystalline samples – different values of the dielectric constant $\varepsilon_r'$ and the dielectric losses $\tan\delta_\varepsilon$ according to the orientation of the electric field relative to the sample axes. This property of the crystals is well known and it is a very natural characteristic of both monocrystalline and polycrystalline samples depending on the technology used and in relation to the non-spherical form of the building grains. There are two types of anisotropy: bi-axial and uni-axial ones; in both cases the complex dielectric permeability $\varepsilon_r = \varepsilon_r'(1 - j\tan\delta_\varepsilon)$ is represented by a diagonal tensor [1]. In the case of be-axial anisotropy, the diagonal components of the complex dielectric constant along the all three axes $O_x$, $O_y$ and $O_z$ are different, i.e. $\varepsilon_{xx} \neq \varepsilon_{yy} \neq \varepsilon_{zz}$, which is valid as for the real part of the dielectric constant $\varepsilon_{xx}' \neq \varepsilon_{yy}' \neq \varepsilon_{zz}'$, as well as for the dielectric loss tangent $\tan\delta_{\varepsilon_{xx}} \neq \tan\delta_{\varepsilon_{yy}} \neq \tan\delta_{\varepsilon_{zz}}$ (here $\tan\delta_\varepsilon = \sigma/\varepsilon_r' 2\pi f$, where $\sigma$ is the conductivity of the crystal sample in [S/m], and $f$ is the frequency). In the case of uni-axial anisotropy, two different diagonal components are formed – parallel (longitudinal) $\varepsilon'_{\|}$, where $\varepsilon'_{\|} = \varepsilon_{xx} \approx \varepsilon_{yy}$, and perpendicular (transverse) $\varepsilon'_{\perp}$, where $\varepsilon'_{\perp} = \varepsilon_{zz}$ (this also could be applied to the dielectric loss tangent, i.e. $\tan\delta_{\varepsilon_{\|}} = \tan\delta_{\varepsilon_{xx}} \approx \tan\delta_{\varepsilon_{yy}}$ and $\tan\delta_{\varepsilon_{\perp}} = \tan\delta_{\varepsilon_{zz}}$ respectively). For isotropic crystals, the equations for the complex dielectric permeability can be written as equalities $\varepsilon_r \approx \varepsilon_{xx} \approx \varepsilon_{yy} \approx \varepsilon_{zz}$. Bulky crystalline samples of different shapes, optical glasses, liquid crystals, most of the metamaterials, 3D printed materials, woven textile specimens for wearable antennas, plant tissues etc. have been usually characterized by bi-axial anisotropy. Contrariwise, reinforced microwave substrates, flat ceramic substrates, LTCC (low temperature co-fired ceramics), etc. have uni-axial anisotropy. Figure 1 illustrates different specimens with uni- and bi-axial anisotropy.

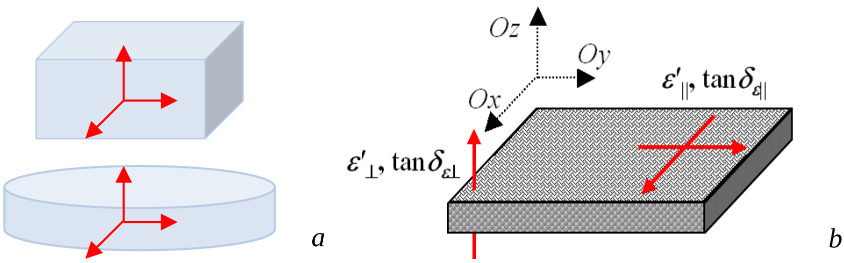


Figure 1. (Color on line) Anisotropic crystalline samples: $a$) bulky bi-anisotropic samples of different shapes: parallelepipeds (prisms) and disks; $b$) flat reinforced substrate with uni-axial anisotropy [3].

## 2 Measurement of Uni-Axial Anisotropy of Flat Crystalline Samples and Metamaterials by the Two-Resonator Method

Measurement of uni- or bi-axial dielectric anisotropy of crystalline samples is not an easy task; there is no universal method for this purpose. Very often the crystalline lattice specimens are of small size, complex shape, or have high dielectric permeability; therefore, the so-called perturbation waveguide or resonance method is commonly used [3,4]. The accuracy of this method, however, is not enough, especially for high values of the dielectric constant and strong anisotropy of the sample.

That is why we propose to use the more accurate resonance methods for measurement of dielectric crystalline samples' uni- or bi-axial anisotropy, especially in case of crystalline samples of the regular shapes (disks, prisms, cylinders, etc.). These methods are based on utilization of $TE_{011}$ and/or $TM_{010}$ cylindrical cavity measurement resonators combined with simulations on 3D electromagnetic simulator as an auxiliary tool for determination of the dielectric parameters in a particular direction. We have serious experience with similar resonance measurement methods applied to dielectric substrates, antenna radomes and other flat dielectric specimens [5-12].

The authorship two-resonator method for anisotropy characterization of single-layer samples has been discussed in detail in papers [5-7]. Two approaches of this method have been developed – both analytical and numerical, which can complement each other. The principle of the numerical approach of the two-resonator method is described in detail in [6,8], as well as the strategy for creation of stylized 3D models for reliable electromagnetic simulations, the measurement accuracy, etc. The method is based on the utilization of a pair of cylindrical resonators, each of them supporting either TE, or TM modes. The modes are with mutually perpendicular directions of the excited electric field, which is extremely comfortable for measurement the uni-axial anisotropy, for instance $TE_{011}$- and $TM_{010}$-modes in the cylindrical resonators.

As examples, Figure 2 presents the schematic views, stylized models and photos of two simplest cylindrical resonators, which have been designed to support high

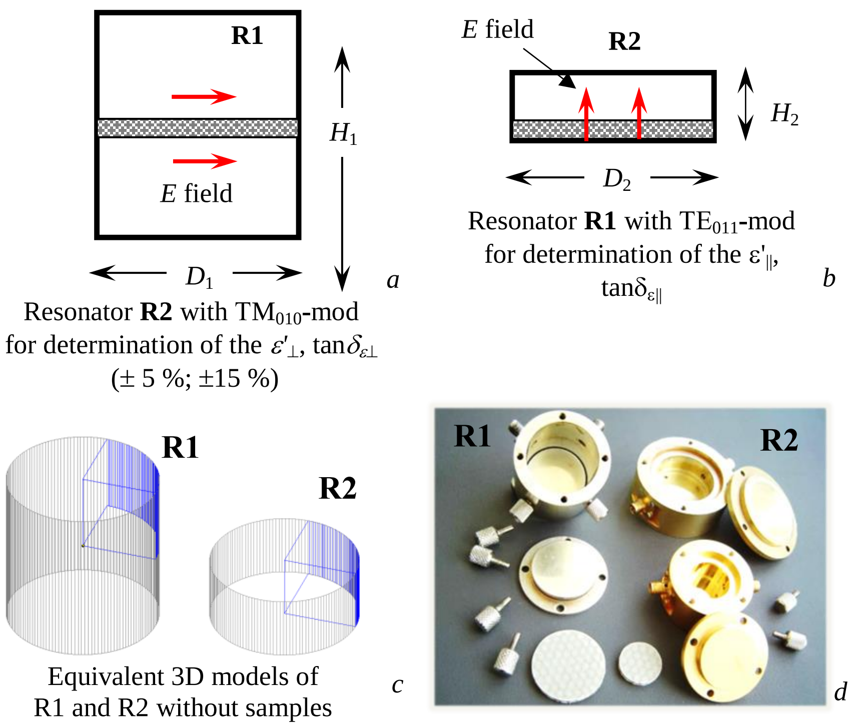


Figure 2. (Color on line) Resonators with $TE_{011}$- and $TM_{010}$-mode: $(a)$ R1; $(b)$ R2; $(c)$ equivalent 3D models; and $(d)$ photo of the measurement resonators R1 and R2 [8].

quality factor modes, necessary for accurate uni-axial anisotropy measurements: resonator R1 with $TE_{011}$ mode and resonator R2 with $TM_{010}$ mode [5,8].

The measurement error with each of the two resonators has been also shown in the figure captions of Figure 2, when the analytical model has been used. The accuracy is relatively high, because an approach with equivalent parameters has been introduced in the 3D models of the resonators, such as equivalent resonator diameter and equivalent conductivity of its walls, which can be changed at each measurement stages depending on different factors. Thus the inaccuracies in determining the geometric dimensions are taken into account as well as the inaccuracies due to the non-ideal inner surface of their walls and actual surface roughness.

The measurement procedure with the two-resonator method (as described in [8,9]) includes two main steps: first – determination of the equivalent parameters of the empty measurement resonators (without sample) and then – obtaining the complex dielectric permeability of the sample incorporated in the resonator volume. Measurements are performed at fixed frequencies of the excited modes. To avoid this disadvantage, it is possible to use adjustable resonators in a wide frequency range and measurement resonators with a more complex shape as described in [9].

In this paper we apply the described measurement procedure for several different types of resonators and for various known and unknown samples as examples. The first example is dedicated to an extreme characterization of the uni-axial anisotropy of very thin samples (measured thickness 12 $\mu$m) with fully unknown crystalline structure, obtained under vacuum deposition. For such thin samples that cannot be measured alone in the resonators, it is important to use a support-

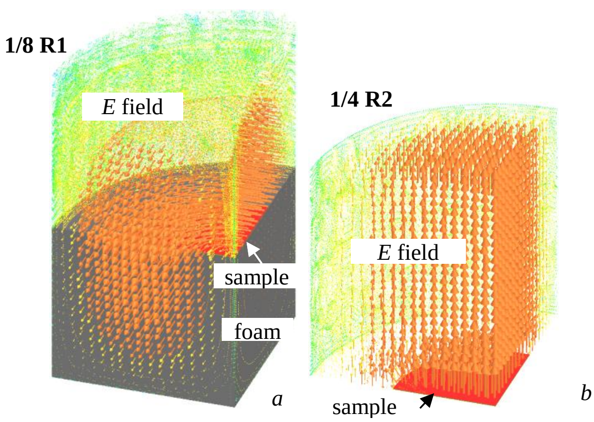


Figure 3. (Color on line) Electric field in simulated resonator models: $a$) R1 and $b$) R2 with a thin anisotropic sample and with Kapton isotropic supporting layer.

ing layer with good isotropy so as not to affect the anisotropy characterization of the basic sample. Here we have used the well-known Kapton tape. The simulated 3D models of the resonators R1 and R2 with disk samples are depicted in Figure 3 ($E$ fields are parallel and perpendicular respectively to the plane of the samples). The results for the measured parallel and perpendicular dielectric parameters of thin samples with uni-axial anisotropy have been presented in Table 1. Only the values of the dielectric constants $\varepsilon'_\perp$ and $\varepsilon'_\parallel$ have been shown graphically in Figure 4.

Table 1. Dielectric parameters of four thin metamaterial samples (12 $\mu$m tick) with different structures, placed between two Kapton supporting layer (2×50 $\mu$m), measured by the two-resonator method in Ku and K frequency bands

| Sample | Ku band | | K band | |
|---|---|---|---|---|
| | $\varepsilon'_\parallel$ / $\tan\delta_{\varepsilon_\parallel}$ | $\varepsilon'_\perp$ / $\tan\delta_{\varepsilon_\perp}$ | $\varepsilon'_\parallel$ / $\tan\delta_{\varepsilon_\parallel}$ | $\varepsilon'_\perp$ / $\tan\delta_{\varepsilon_\perp}$ |
| 1D | 3.780 / 0.0085 | 1.295 / 0.0033 | 4.030 / 0.0094 | 1.180 / 0.0011 |
| 2D | 5.450 / 0.0390 | 2.100 / 0.0100 | 6.880 / 0.0382 | 1.580 / 0.0190 |
| 3D | 3.535 / 0.0047 | 1.147 / 0.0015 | 3.445 / 0.0053 | 1.040 / 0.0025 |
| 4D | 3.950 /0.0099 | 2.100 / 0.0100 | 4.940 / 0.0121 | 1.405 / 0.0045 |
| Kapton | 3.150 / 0.0225 | 3.100 / 0.0217 | 3.184 / 0.0230 | 3.280 / 0.0180 |

The results show a pronounced uni-axial anisotropy of the considered artificial crystalline samples with a character of metamaterials ($\varepsilon'_\parallel$ is in the interval 3.4–6.9, while $\varepsilon'_\perp$ is in the interval 1.04–2.1 due to the porosity). Also, the excellent measured isotropy of the Kapton supporting layer can be seen. This example shows the good sensitivity and selectivity of the two-resonator method even for very thin crystalline samples with air inclusions with well-expressed uni-axial anisotropy.

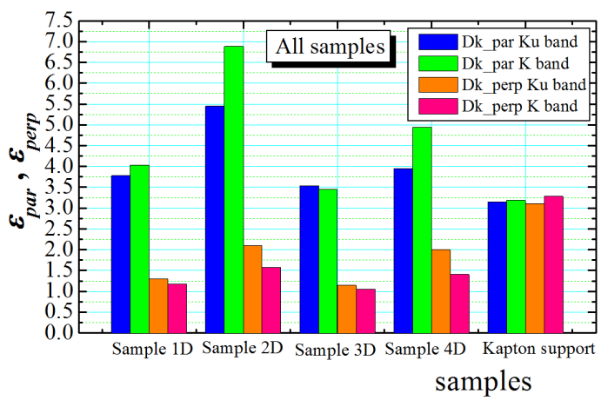


Figure 4. (Color on line) Graphical representation of the parallel and the perpendicular dielectric constants of 4 thin samples with Kapton supporting layer.

## 3 Measurement of Anisotropy of Bulky Crystals with a Pair of Split Resonators

The second example is referred to the measurement of relatively large in size poly- or monocrystalline samples, uniformly cut as prisms, cylinders, rings, etc. In this case, a pair of split measurement resonators (Split-Post Dielectric Resonators, SPDR's) is more comfortable to be applied for characterization of the sample uni-axial anisotropy. The measurement procedure with such a pair of resonators is described in the papers [9-11]. As in the previous case of conventional cylindrical measurement resonators, one of the resonators from the pair supports $TE_{011}$ mode, while the other one – $TM_{010}$ mode (see Figure 5). The difference here is that these are resonances in the dielectric resonator (the crystal sample itself), whose resonance characteristics are directly related to the sample dielectric parameters. Analytical solutions in this case are difficult to be developed due to the complexity of the structure. Instead, applying reliable simulations of well-constructed 3D model of the resonance structure with electromagnetic simulator, we can extract the sample parameters in the corresponding direction. The basic principles of the model creation and the measurement procedure are described in [10]. Again, a match between the measured and simulated resonance parameters is sought as with ordinary cylindrical measurement resonators. Here usually the quality factor is higher, which allows measurement of samples with lower dielectric losses.

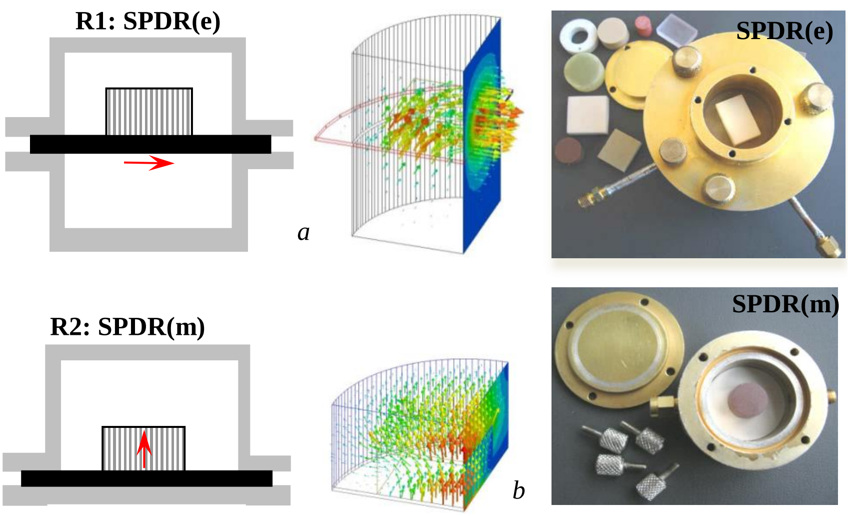


Figure 5. (Color on line) Pair of two split resonators: $(a)$ R1 SPDR(e) with $TE_{011}$-mode; equivalent 3D model; experimental resonator; $(b)$ R2 SPDR(m) with $TM_{010}$-mode; equivalent 3D model; experimental resonator [9,11].

Table 2. Dielectric parameters of crystalline samples with prismatic form, measured with a pair of SPDR resonators (see [11])

| Dimensions of the crystalline sample, mm | SDPR (e) resonator $TE_{011}$ mode $\varepsilon'_{\|} / \tan\delta_{\varepsilon_{\|}}$ ($f$, GHz / $Q_0$) | SDPR (m) resonator $TM_{010}$ mode $\varepsilon'_{\perp} / \tan\delta_{\varepsilon_{\perp}}$ ($f$, GHz / $Q_0$) |
|---|---|---|
| Empty resonator | 1 / 0<br>13.1574 / 8171 | 1 / 0<br>7.6382 / 3820 |
| Alumina $19.8 \times 19.6 \times 6.0$ | 9.883 / 0.0000523<br>(5.8039 / 8638) | 9.0895 / 0.000028<br>(5.2302 / 3482) |
| Alumina $19.3 \times 11.9 \times 6.0$ | 9.885 / 0.0000331<br>(6.8144/ 14498) | 8.660 / 0.0000285<br>(5.5567 / 3670) |
| Alumina $19.0 \times 6.8 \times 6.0$ | 9.724 / 0.0000134<br>(8.9997 / 7342) | 8.322 / 0.0000323<br>(5.9774 / 3805) |
| Alumina $12.5 \times 11.1 \times 6.0$ | 9.704 / 0.0000318<br>(7.7052 / 18514) | 8.289 / 0.0000331<br>(5.8379 / 3831) |
| Alumina $12.6 \times 7.0 \times 6.0$ | 9.708 / 0.0000695<br>(9.3160 / 12137) | 8.103 / 0.0000342<br>(6.1552 / 3899) |
| Sapphire $17.7 \times 12.5 \times 6.6$ | 10.1118 / 0.0000293<br>(6.5510 / 16655) | 8.425 / 0.000001<br>(5.3658 / 3800) |
| Sapphire $10.3 \times 8.0 \times 5.3$ | 10.2915 / 0.0000302<br>(9.1840 / 20750) | 7.820 / 0.000005<br>(6.4057 / 3938) |
| Quartz $12.8 \times 12.3 \times 9.3$ | 4.462 / 0.0000685<br>(9.4450 / 9700) | 4.177 / 0.000021<br>(5.3520 / 3766) |

The results from measurements of the complex dielectric permeability of uniform crystalline samples with prismatic form of Alumina, Sapphire and Quartz are presented in Table 2 for the X and C frequency bands. It can be noticed, that these crystal samples have extremely low dielectric losses. In fact, this is the great advantage of the presented variant of measurement method based on split dielectric resonators. As can be seen from the results the measured quality factors for some samples at much lower frequencies are even higher than those in the empty resonators (without sample). The disadvantage of the method is the fact that the measurements have been performed only at frequencies, which are defined mainly by sizes and the dielectric parameters of the samples (i.e. at the frequencies, where the samples have their own resonances, which are slightly affected by the presence of the metal walls of the split cylindrical resonators).

## 4 Measurement of Bi-Axial Anisotropy of Bulky Crystalline Samples by the One-Resonator Method

In the last third example, we offer a method that uses only one resonator (we use resonator R2 because the direction of the excited electric field of $TM_{010}$ mode is strictly along one $(Oz)$ axis [12]). This method and the resonator used, are the best suited to measure the bi-axial anisotropy, typical for the most crystalline samples. In order to obtain information about the dielectric constant and the dielectric loss tangent in the all three directions of the structure, the sample is placed in the resonator successively in three different positions

relative to the $0z$ axis, one for each direction of the coordinate system, as shown in Fig. $6a$. Three different measurements have to be performed for each sample. The obtained information is processed by simulating the 3D model of the structure with the aid of 3D electromagnetic simulator and three values for the relative dielectric permeability $\varepsilon'_{xx} \neq \varepsilon'_{yy} \neq \varepsilon'_{zz}$, as well as the dielectric loss tangent $\tan\delta_{\varepsilon_{xx}} \neq \tan\delta_{\varepsilon_{yy}} \neq \tan\delta_{\varepsilon_{zz}}$ of the measured sample are obtained. The basic principles of creating the 3D model as well as the measurement procedure are similar to those described so far. The 3D models of the measurement

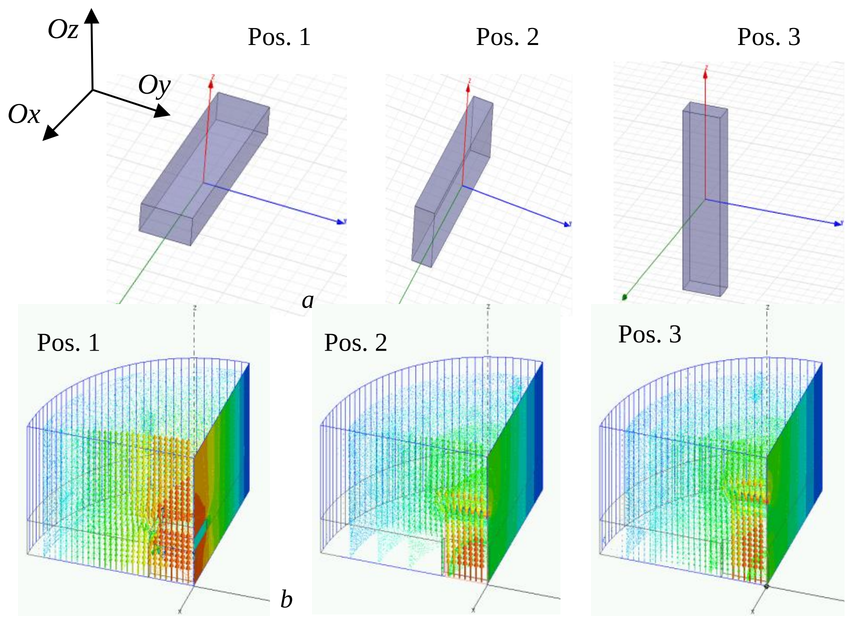


Figure 6. (Color on line) One-resonator method (example for resonator R2 with excited $TM_{010}$ mode): a) positions of the sample in the resonator; b) distribution of the electric field for the three positions of the crystalline sample.

resonator along with the sample and the distribution of the electric field for the three positions of the sample are shown in Figure 6*b*.

The results from the measurements of crystalline samples with prismatic form of unknown material using $TM_{010}$-mode measurement resonator are presented in Table 3. The data show that the differences in the dielectric constant values in the three directions for each crystal sample can be accurately measured, in order to make comparisons (pairs of samples 1/2 and 3/4 have different compositions). From the presented in this example data, it can be concluded that not in all cases the walls of the manufactured prismatic samples are orientated on the relevant crystallographic axes of the material with a cubic crystalline structure and this fact can be detected by the measurements.

Table 3. Dielectric parameters of crystalline samples from unknown (new) material, measured with a $TM_{010}$-mode resonator (one-resonator method) in frequency range C (about 6–7 GHz)

| Sample No. | Dimensions of the sample, mm | $\varepsilon'_\perp / \tan\delta_{\varepsilon_\perp}$ Pos. 1 | $\varepsilon'_\perp / \tan\delta_{\varepsilon_\perp}$ Pos. 2 | $\varepsilon'_\perp / \tan\delta_{\varepsilon_\perp}$ Pos. 3 |
|---|---|---|---|---|
| 1 | $8.13 \times 5.66 \times 0.81$ | 3.70 / 0.00018 | 4.14 / 0.00016 | 5.00 / 0.00022 |
| 2 | $7.60 \times 6.16 \times 1.10$ | 3.90 / 0.0006 | 4.75 / 0.00064 | 4.20 / 0.00058 |
| 3 | $7.60 \times 5.49 \times 0.99$ | 3.50 / 0.00047 | 4.50 / 0.00047 | 3.96 / 0.00038 |
| 4 | $7.93 \times 6.98 \times 1.22$ | 3.96 / 0.0001 | 4.75 / 0.00022 | 4.20 / 0.00021 |

An advantage of the presented method is the ability to measure both uni-axial and bi-axial anisotropy of crystalline samples of not very large heights/diameters (i.e. when the resonances of the crystalline samples are at much higher frequencies than those of the measurement resonator). The method also allows flat samples with uni-axial anisotropy to be measured, but if the largest sample size is no more than 30-50% of the resonator height, in order not to be strongly influenced by the shielding effect of the walls.

## 5 Conclusion

In the present work are given three different examples of measurement of uni- and bi-axial dielectric anisotropy of crystalline samples with different values of dielectric permeability, different shape and different composition. First of all, it is shown that the author's two-resonator method gives sufficiently accurate results for thin crystalline specimens with uni-axial anisotropy. The disadvantage of the method is that the measurements are made at different frequencies for the various excited modes and in the presence of frequency dependence of dielectric parameters, inaccurate conclusions about anisotropy can be made. A similar problem can also occur with the method of two split resonators. However, this method allows the parameters of crystals with regular shape, with high values of the dielectric constant and with very low losses, to be determined with great accuracy. The results obtained for known materials such as quartz ($\varepsilon_r \sim 4.3$), Alumina ($\varepsilon_r \sim 9.8$), sapphire ($\varepsilon_r \sim 10.2$) coincide with the known values in the literature.

To measure bi-axial anisotropy, when the parameters of the crystalline sample are different in different directions, we have proposed one-resonator method, but for measuring samples with dimensions up to 30-50% of the dimensions of the measurement resonators (diameter and height). In this case, the measured anisotropy is important in investigating of the unknown samples. If the samples are cut out in regular shape in the direction of the crystallographic axes, valuable information about the dielectric anisotropy of the samples is obtained.

In conclusion, the proposed methods provide a very good opportunity to measure dielectric anisotropy of crystalline samples, regardless of the measuring equipment used and the software product, in the microwave range (in our case from C to Ka range, i.e. from 4 to 38 GHz).

## Acknowledgements

The authors are grateful to the National Fund for Scientific Research for the financial support under contract DN-07/15.

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