The Measurement of Permittivity Tensor of Uniaxial Crystals with Tetragonal and Hexagonal Symmetry at Microwaves

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Abstract- A nondestructive method for measuring permittivity tensor of uniaxial crystals with tetragonal and hexagonal symmetry in microwave range was proposed. The developed measuring complex was used for testing several dielectrics, inaccuracy of the dielectric permittivity measurements for isotropic dielectrics was less than \( \pm 0.5\% \), for components of dielectric permittivity tensor error didn’t exceed \( \pm 1\% \) and for dielectric loss tangent – \( \pm 10\% \).

Keywords- Nondestructive Method; Permittivity Tensor; Uniaxial Crystal

I. INTRODUCTION

The overwhelming majority of existing devices for measuring the material parameters in a microwave range is the destructive one. Non–destructive measuring devices (that we are aware of) don’t allow measuring components of permittivity tensor, dielectric losses tangent \( (\tg_1 \text{ and } \tg_\perp) \) along the main axes in anisotropic crystals. A lot of known dielectrics (including artificial) belong to some extent anisotropy, and the question is whether it can be neglected at a certain investigation phase. The purpose of this work is to develop some techniques and a corresponding device for a non–destructive measurement of permittivity tensor components for uniaxial crystals.

II. THE EQUIPMENT

We have developed a measuring complex and the software for it that allows conducting the automatic measurements of permittivity tensor components, factors of dielectric losses in uniaxial crystals without their destruction in a microwave range. The complex is built of some off the shelf equipment: a scalar network analyzer (P2–61, P2–67) with some replaceable generating blocks, a digital ondometer (CH3–54), a personal computer (PC), and of some non–standard equipment specially designed for the complex: a set of waveguide junctions and flanges of various configuration; a data acquisition and control device, software for the PC (Fig.1). The waveguide junctions are made of silver covered brass and are designed for the measurements in a frequency range 7–37 GHz. Using half-opened waveguide junctions of various configuration allows us to study local (not averaged on all volume of the sample) characteristics of dielectric samples without their destruction. The open waveguide ends are not radiating, that allow using the structures for nondestructive measurement of parameters of materials.

Fig. 1 The measuring complex

The resonator, basically, is the central region (I) (Figs. 2–4) and that dielectric volume, which is placed in this region. As a result, the measurements of local parameters of materials are possible to carry out.
In our work on developing the measuring technique the following problems have been solved:

- the development of computational electrodynamics model of the following waveguide junctions filled with the isotropic or anisotropic environment: 1) rectangular – rectangular waveguides (Fig.2) \(^{[1–3]}\), 2) cylindrical – radial waveguides (Fig.3) \(^{[4]}\), cylindrical – rectangular waveguides (Fig.4) \(^{[5]}\), with focus on determination of electric properties of crystal dielectrics placed at the junction. To obtain the system of linear algebraic equations (SIAE) it was modified a known mode-matching technique \(^{[6]}\); electromagnetic fields in each of junction of partial waveguides (II, III) (Fig. 3) were presented as a decomposition on the damped eigen waveguide modes, and – as the field superposition of these waveguides in junction region (I) (which is their common region). The electromagnetic field in this representation term wise satisfied boundary conditions on metal and region boundaries, that reduced, as a result, to an infinitive SIAE of the second order. The resonance frequencies of modes were determined from the condition that the determinant of the truncated indefinite SIAE equaled zero. To solve obtained SIAE the numerical algorithm on the basis of Muller’s method and software were designed (program language is Fortran – Windows’–appendix Fortran Power Station 4.0) \(^{[2]}\);

- the theoretical and experimental study of the resonant phenomena occurred in the described structures \(^{[1–5, 7]}\);

- the classification of the eigen modes in such structures, the determination of their frequency ranges and interrelation with all possible classes of resonances \(^{[2, 3]}\). Depending on geometrical and electrodynamical parameters at structures are possible to assign the following resonance modes:

1) the natural resonances of a waveguide junction on damping modes, for which the attenuation of all waveguides on resonant frequency is characteristic feature;

2) the natural waveguide–dielectric resonances, which are realized in the following case: the resonance wave is propagation in the central region with dielectric (I), and all wave modes of waveguides (II, III) are attenuation;

3) the quasinatural resonances, which exist in a waveguide junction under the condition of at least one wave propagation, and a higher type of waves are resonant \(^{[3]}\);

- the development of techniques for measuring permittivity tensor components and dielectric losses of transparent and opaque crystals;

- the design of microwave measuring complex.
The data acquisition and control device were made using a microprocessor kernel; it provides communication between components of the device and the PC and realization of automatic adjustments algorithms. This interfacing device can communicate with the PC using any of 3 possible communication channels: USB, RS-232, IRDA. It also has 16 discrete lines for input-output that can be used as an incomplete analogue of LPT–interface, 8 analogue inputs for connection of detectors and other sensors, 6 analogue outputs for controlling frequency and amplitude of the frequency sweep oscillator (FSO), other peripheral components and the bias. The microcontroller has FLASH–memory that allows operatively changing the configuration of the complex. Some algorithms of a digital signal processing of signals from detectors and analogue multipliers, and also a digital automatic power control (APC) were implemented in the device.

The windows based software of the complex has been designed in a modular manner so that the measuring complex is capable to solve various problems and can be easily expanded to new operation modes. The data acquisition software block for real time processing was written in MATLAB.

### III. Measurement Technique

The algorithm of measuring permittivity tensor components and dielectric losses tangents (tgδ_∥, tgδ_⟂) in crystals consists of the following steps:

- based on rough estimation of the expected value of permittivity to measure select working frequency range;
- the measuring device with waveguide sections that corresponds to cross-section of crystals under study is joined in a straight-through scheme with the Network Analyzer with the sweep block for the working range; modes are identified and their resonant frequencies are measured;
- from numerical solution of SLAE that emerges in the theoretical model of the junction \(^2, 3\) the transversal and longitudinal components of the permittivity tensor are defined;
- the width of transmittance resonant curve \(\Delta f\) for H– and E– modes is measured at the level \(\alpha_0 -3\ dB\), where \(\alpha_0\) (dB) is the transmittance at resonant frequency \(f_0\);
- the value of loaded quality factor of working modes (\(Q_{EH}^{E,H} = f_0 / \Delta f\)) is calculated; based on it the unloaded (own) quality factor \(Q_{0}^{E,H}\) is calculated as \(Q_{0}^{E,H} = Q_{EH}^{E,H} \cdot \frac{- \alpha_0 / 20}{1-10^{-\alpha_0/20}}\);
- from the unloaded quality factors the dielectric losses along the corresponding directions (tgδ_∥ and tgδ_⟂) are calculated using expressions from [1].

#### A. Uniaxial Crystals with The Tetragonal Type of Symmetry

Among the crystals with this type of symmetry (syngony) \(^8\) we can mention quartz, leucosapphire, an artificial ruby, rutile, etc. Usually they are grown up in a form of a boule, having rectangular parallelepiped shape. Waveguide III (Fig. 2) is completely filled by dielectric, the permittivity tensor of which has a diagonal form \(\varepsilon_{xx} = \varepsilon_{yy} \neq \varepsilon_{zz}\) in the Cartesian system of coordinates oriented along the crystal (boule) axis (orientation of the optical axis is [001]). The boule edges should be aligned along the waveguide junction axis.

For this type of crystals determination of the tensor components is based on:

- measuring resonant frequency of \(H_{110}^\circ\) mode (\(f_1\));
- measuring resonant frequency of \(E_{110}^\circ\) mode (\(f_2\));
- numerical solution of SLAE \(^2, 3\) that yields components of permittivity tensor \(\varepsilon_{xx}\) and \(\varepsilon_{zz}\).

#### B. Uniaxial Crystals with The Hexagonal Type of Symmetry

In the crystal system of coordinates (crystallographic) \(\Sigma\) (\(\alpha = \beta = 90^\circ, \gamma = 120^\circ, a = b = \varepsilon_{xx} \neq c = \varepsilon_{zz}\) \(^8\) and in the orthogonal system of coordinates (crystalophysical) \(\Sigma'\) permittivity tensor looks accordingly:
Similarly to the previous case we need to measure resonant frequency of $E_{110}$– mode and that of $H_{110}$– mode and solve SLAE \[^2, 3\] in order to find $\varepsilon_{xx}$ and $\varepsilon_{zz}$.

IV. EXPERIMENTAL RESULTS

We have conducted experimental measurements of a number of dielectric samples (Tables 1–3) and compared the results to the samples certified by the GOST (government standard) technique. The measurement inaccuracy of dielectric permittivity of isotropic dielectrics was less than $\pm 0.5\%$, and that for anisotropic tensor components didn’t exceed $\pm 1\%$. Inaccuracy of dielectric losses tangent measurement was below $\pm 10\%$.

**TABLE I MEASURED AND REFERENCE VALUES OF $\varepsilon$ AND $Tg\delta$ FOR ISOTROPIC DIELECTRICS**

<table>
<thead>
<tr>
<th>dielectric</th>
<th>$f$, GHz</th>
<th>$\varepsilon_{meas.}$</th>
<th>$\varepsilon_{[9]}$</th>
<th>$Tg\delta_{meas.}$</th>
<th>$Tg\delta_{[10]}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>polycor BK-100</td>
<td>13.990</td>
<td>9.40</td>
<td>9.51</td>
<td>2.2 $\times$ 10^{-4}</td>
<td>2 $\times$ 10^{-4}</td>
</tr>
<tr>
<td>polycor BK-100</td>
<td>14.005</td>
<td>9.38</td>
<td>9.48</td>
<td>2.8 $\times$ 10^{-4}</td>
<td>2.6 $\times$ 10^{-4}</td>
</tr>
<tr>
<td>ceramics 22XC</td>
<td>13.725</td>
<td>10.28</td>
<td>10.4</td>
<td>2.5 $\times$ 10^{-4}</td>
<td>1.5 $\times$ 10^{-4}</td>
</tr>
<tr>
<td>crystal NaCl</td>
<td>8.846</td>
<td>5.75</td>
<td>5.80</td>
<td>1 $\times$ 10^{-4}</td>
<td>1 $\times$ 10^{-4}</td>
</tr>
</tbody>
</table>

**TABLE II EXPERIMENTAL RESULTS FOR PERMITTIVITY OF MONOCRYSTAL SAMPLES**

<table>
<thead>
<tr>
<th>dielectric</th>
<th>$\varepsilon_{zz}$</th>
<th>$\varepsilon_{xx}$</th>
<th>reference data</th>
</tr>
</thead>
</table>
| Al$_2$O$_3$ (leuco-sapphire) | 11.51 | 9.22 | $\varepsilon_{zz} = 11.66$  
$\varepsilon_{xx} = 9.25$  
\[^{[11]}\] |
| SiO$_2$ (crystalline quartz) | 4.65 | 4.55 | $\varepsilon_{zz} = 4.60$  
$\varepsilon_{xx} = 4.44$  
\[^{[12]}\] |
| sintered quartz | 3.85 | - | $\varepsilon = 3.8$  
\[^{[12]}\] |
| ZnS (zink sulfide) | 8.70 | - | $\varepsilon = 8.5-9$  
\[^{[13]}\] |
| Y$_3$Al$_5$O$_{12}$ (yttrium aluminum garnet) | 10.77 | - | $\varepsilon = 10.7$  
\[^{[14]}\] |

**TABLE III EXPERIMENTAL RESULTS FOR DIELECTRIC LOSSES TANGENT OF MONOCRYSTAL SAMPLES**

| dielectric | $Tg\delta_{||}$ | $Tg\delta_{\perp}$ |
|------------|----------------|-------------------|
| LiTaO$_3$ (lithium tantalum) | 43.4 | 2.5 $\times$ 10^{-4} | 3.2 $\times$ 10^{-4} |
| Y$_3$Al$_5$O$_{12}$ (yttrium aluminum garnet) | 10.65 | - | 8 $\times$ 10^{-3} |
| crystal NaCl | - | 5.75 | 1 $\times$ 10^{-4} |

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V. CONCLUSIONS

It was found that the configuration of the considered waveguide junctions, waves $H$- and $E$- type can exist separately in cylindrical ramifications -rectangular waveguides, cylindrical - radial waveguides only if they are filled with an isotropic material. For junction of rectangular waveguides, the separate existence of waves is possible in the case of filling them with an anisotropic medium with a diagonal dielectric tensor.

The open waveguide ends are not radiating, that allows using the structures for nondestructive measurement of parameters of materials. The resonator, basically, is the central region (I) and that dielectric volume, which is placed in this region. As a result, the measurements of local parameters of materials are possible to carry out.

A nondestructive measurement technique for determination of permittivity tensor components and dielectric losses of uniaxial crystals with tetragonal and hexagonal symmetry types in microwave range was developed. The measurement errors of $\varepsilon$ and $tg\delta$ of isotropic and anisotropic samples were determined from the measurements of certified samples. The main contribution in total measurement error (regular and statistical) was due to manufacturing inaccuracies of waveguide sections and the samples under study. Relative measuring error of permittivity tensor components in range $\varepsilon = 2 - 20$ (when accuracy of manufacturing the waveguide sections and samples was $\pm 0.02$ mm) was numerically estimated to be $\pm 0.5\%$ for isotropic crystals and $\pm 1\%$ for uniaxial crystals. If the waveguides and the samples were manufactured with CNC machine tools of high accuracy (such as “Fanuc”) then the measurement error could be reduced down to 0.2\%. Relative measurement error of dielectric losses tensor components didn’t exceed $\pm 10\%$.

REFERENCES

