

EXPERIMENTAL OBSERVATION OF FUNDAMENTAL MICROWAVE ABSORPTION IN HIGH-QUALITY DIELECTRIC CRYSTALS

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This paper is devoted to the experimental study of small microwave losses ($\tan \delta \approx 10^{-4}$ - 10^{-6}) in high-quality dielectric single crystals. Wide-temperature-range measurements of the loss tangent at frequencies 9-72 GHz have indicated the existence of fundamental (lattice) and extrinsic (due to imperfections) microwave absorption in sapphire and YAG crystals. Strong distinctive temperature dependence of the lattice absorption ($\tan \delta \sim T^\alpha$, $\alpha = 4$ or $\alpha = 5$ for different crystal symmetries) allows to identify it with the "photon + phonon \rightarrow phonon" interaction due to lattice anharmonicity.

1. Introduction

Since resonant absorption in normal¹¹ solid dielectrics takes place in the optical and infrared areas of the spectrum, it has been considered for a long time that dielectric losses at low frequencies, including the microwave band, have nothing in common with the fundamental lattice properties and are fully determined by defects and impurities. Perhaps this is the reason for $\tan \delta$ having become a more technical parameter referring rather to a *solid material* and not to a *solid substance*. This concept has been indirectly backed up by the divergency of experimental data on $\tan \delta$ of various solids. For instance, for sapphire the reported values of the loss tangent varied from 1×10^{-3} to 4×10^{-6} [1-3].

Design and research of high- Q solid state microwave resonant systems [4-10] in which Q is directly limited by the dielectric loss in a solid, have brought about a question: what is the fundamental limit of $\tan \delta$ in perfect solids, i.e. what is the least possible

value of dielectric relaxation in the ideal crystal lattice?

Dielectric relaxation in an ideal crystal can be regarded as a result of interaction between an electromagnetic field and the phonon system of the dielectric. The ac electric field disturbs the equilibrium in the ensemble of phonons, and the following relaxation is accompanied by energy dissipation. In harmonic approximation only resonant interaction of the field with optical phonons can exist, both time and spatial variations of the field being essential. At low frequencies the ac field is quasi-uniform: for the wavelength λ this means $\lambda \gg 1/|k|$, where k is the wave vector of the optical phonons. Thus there is no interaction between the field and phonons in harmonic approximation. And so the low-frequency dielectric relaxation in the ideal lattice should be of anharmonic origin.

Most of the theoretical papers concerning low-frequency EM absorption in solids contain the evaluation of various defects' contribution to $\tan \delta$. In most cases the dielectric relaxation produced by the defects is modelled by a direct excitation of acoustic phonons at the frequency of the ac electric field: the direct

¹¹ By normal we mean a dielectric which does not possess piezo- or ferroelectric properties.

extrinsic losses. Only a small number of the publications deal with the lattice absorption mechanism. A complete theory of dielectric losses due to anharmonicity of the ideal lattice is proposed by Gurevich [11-13]. He analyzed the lattice anharmonicity for various crystal symmetry classes and obtained both analytic formulae and numerical estimates for $\tan \delta$ of ideal crystals. He showed specifically that fundamental absorption in crystals with the centre of symmetry should be much less than that in non-symmetrical ones. The main contribution into $\tan \delta$ at frequencies $\omega \ll kT/\hbar$ should be made by the process of admixture of an ac field photon to an acoustic phonon with the following birth of a greater energy phonon. In symmetrical crystals the two phonons in the interaction must belong to different acoustic branches of the phonon spectrum. Since the former is determined by the type of crystal symmetry, the values and analytic expressions for $\tan \delta$ differ for different crystal symmetry classes. We reproduce here the fundamental loss tangent formulae for hexagonal and cubic (rhombohedral) crystals with the centre of symmetry; the approximation is correct for temperatures T far below the Debye temperature T_D ($T \ll T_D$):

$$\tan \delta \approx \eta \frac{\omega(kT)^3}{\epsilon \rho v^3 \hbar^2 (kT_D)^2} \quad (1)$$

(hexagonal symmetry),

$$\tan \delta \approx \eta \frac{\omega^2(kT)^4}{\epsilon \rho v^3 \hbar (kT_D)^2} \quad (2)$$

(cubic and rhombohedral symmetry).

Here ϵ is the dielectric permittivity of the crystal, ρ the density and v the mean sound speed; η is the dimensionless anharmonicity parameter which is equal to 10-100.

The absolute value of fundamental losses, as predicted by this model, is considerably lower than the standard technical level of the "loss-free" dielectric: $\tan \delta = 1 \times 10^{-6}$. For sapphire (near-hexagonal symmetry: $\epsilon \approx 10$, $\rho = 4 \text{ g/cm}^3$, $v = 6 \times 10^3 \text{ cm/s}$, $T_D = 1047 \text{ K}$ [1]) the predicted value of the loss tangent is $\tan \delta \approx 1 \times 10^{-6}$ at $T = 300 \text{ K}$, $\omega = 2\pi \times 10^{10} \text{ rad/s}$ (parameter η is assumed to be equal to 100), and is

rapidly decreasing with the fall of temperature as T^3 . This is why the observation of fundamental MW absorption requires first the employment of a special high-sensitivity measurement technique. And second, high-quality single crystals should be studied, in which the small lattice absorption is not masked by the effect of imperfections.

2. Measurement technique and experimental results

Anomalously low values of microwave $\tan \delta$ in single crystal alumina were first discovered in 1977 [14]. The quality-factor measurements of dielectric-loaded superconducting resonators (SCR), consisting of a sapphire base with deposited lead or niobium film, showed that the loss tangent in sapphire does not exceed 2×10^{-8} at $T = 2 \text{ K}$ (frequency near 3 GHz). Development of the technique enabled the authors of ref. [4] to establish a new upper limit of the sapphire's loss tangent under the same conditions: $\tan \delta \leq 1.5 \times 10^{-9}$ (3 GHz, $T = 2 \text{ K}$). Similar results were obtained later by Strayer, Dick and Tward [6], who reproduced the described measurement technique. They observed, in addition, a substantial difference (up to two orders) in $\tan \delta$ below 4 K for the sapphire samples delivered from three different suppliers. The latest results on the sapphire's $\tan \delta$ below 4 K obtained by means of SCR, can be found in the publications of Blair and Jones [9] ($\tan \delta \leq 8 \times 10^{-10}$, frequency near 10 GHz, at 1.5 K) and of Thakoor et al. [15] ($\tan \delta \leq 5 \times 10^{-10}$, 3 GHz, 2 K).

The use of dielectric-loaded SCR constitutes a significant breakthrough in improving sensitivity of the $\tan \delta$ measurement. But its application is limited by the narrow temperature interval of superconductivity, while an effective physical study of fundamental microwave absorption requires the measurement of a $\tan \delta$ versus T plot in a wide range.

In this paper, we report the results of wide-temperature-range $\tan \delta$ measurements in sapphire and YAG between 3.5 and 300 K at frequencies 9, 36 and 72 GHz. These results were obtained with the help of dielectric ring resonators (DRR) based on the effect of full internal reflection [16,17].

The DRR is a toroid or a disk (short cylinder) cut of a massive bulk of dielectric. Schematically, it can

be regarded as a closed dielectric waveguide. If the radius of DRR and its surface quality are sufficient, the radiative losses due to scattering of the fringe EM field are negligible and the Q -factor is limited only by the loss tangent of the dielectric. Since the radiative losses depend on temperature very weakly (via the small temperature drift of DRR dimensions and dielectric permittivity), the DRR is a fine instrument for the high-sensitivity measurement of $\tan \delta$ in a wide temperature band.

The Q -factor of DRR can be expressed via radiative losses and $\tan \delta$ in the following way:

$$Q^{-1} = Q_{\text{rad}}^{-1} + K \tan \delta(T).$$

Here Q is the quality factor for a given mode of DRR, Q_{rad}^{-1} describes the radiative losses of this mode, and K is a coefficient corresponding to a fraction of the mode's EM field energy enclosed inside the dielectric. Operational are the modes $TE_{n,1,0}$, $TH_{n,1,0}$ with high angular order n . The EM field of such modes is concentrated within the small circular area inside the dielectric evanescent to its cylindrical surface and drops exponentially outside the dielectric. Three main radiative mechanisms, contributing to Q_{rad}^{-1} , are the following [17]: (1) radiative losses due to the curvature of a dielectric guide, (2) radiative losses due to the surface irregularities, and (3) radiative losses due to the local dielectric constant variations (block structure of crystal).

The sapphire resonators used for the wide temperature-range $\tan \delta$ measurements were fabricated of large single crystal samples grown by the method of oriented crystallization. These samples possessed a concentration of dislocations between 10^2 and 10^4 cm^{-2} and a block structure with disorientation angle less than 1° . The surface of each resonator was polished to optical flatness (roughness less than $3 \mu\text{m}$); the difference between the principle crystal axis and geometrical axis of each resonator was not larger than 1° . All the resonators used in the experiment were of cylindrical shape, with a diameter of $\sim 10 \text{ cm}$ in the X-band (9 GHz), of $\sim 3 \text{ cm}$ in the Q-band (36 GHz), and of $\sim 12 \text{ mm}$ in the M-band (72 GHz).

Numerical computations, which were made in accordance with principles discussed in refs. [16,17] showed, that the total of the above three radiative mechanisms' contribution into Q_{rad}^{-1} did not exceed: $Q_{\text{rad}}^{-1} \leq 1 \times 10^{-11}$ for the X-band resonators,

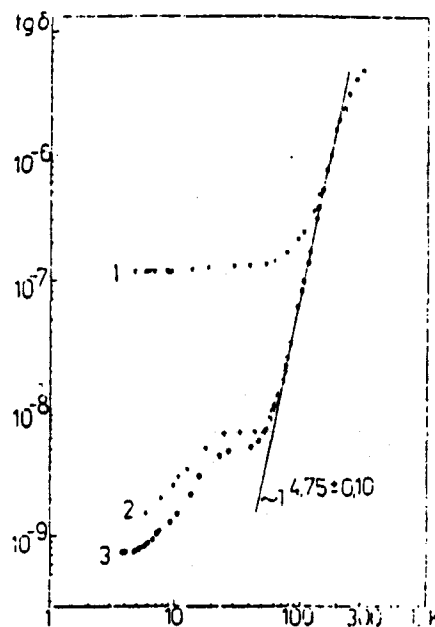


Fig. 1. Microwave absorption in sapphire, X-band (9 GHz).

$Q_{\text{rad}}^{-1} \leq 2 \times 10^{-10}$ for the Q-band resonators, and $Q_{\text{rad}}^{-1} \leq 1 \times 10^{-9}$ for the M-band resonators, used in the experiments. Coefficient K for the high-angular-order modes is close to 1: $K \geq 0.94$ for $n \geq 20$. We can conclude then that a total $\sim 10\%$ error of the $\tan \delta$ evaluation by the inverse value of the Q can be secured for: $Q^{-1} \approx 1 \times 10^{-10}$ in the X-band, $Q^{-1} \approx 2 \times 10^{-9}$ in the Q-band, and $Q^{-1} \approx 1 \times 10^{-8}$ in the M-band.

For cryogenic cooling, resonators were attached at the bottom of the copper liquid-helium tank, enclosed into the evacuated cryostat. Oscillation modes were driven by an inductive probe or by an open end of the dielectric-filled waveguide, placed at a distance of 1 to 10 mm from a resonator's surface.

The main results of $\tan \delta$ measurements on sapphire at 9, 36 and 72 GHz are presented in figs. 1-3. The different branches of the curves at low temperatures correspond to the resonators made of the samples varying in defect structure.

The common feature of the three curves is the presence of a straight section between ~ 50 and $\sim 200 \text{ K}$ (in double logarithmic scale), where the $\tan \delta$ versus T plot can be well approximated by a power of T :

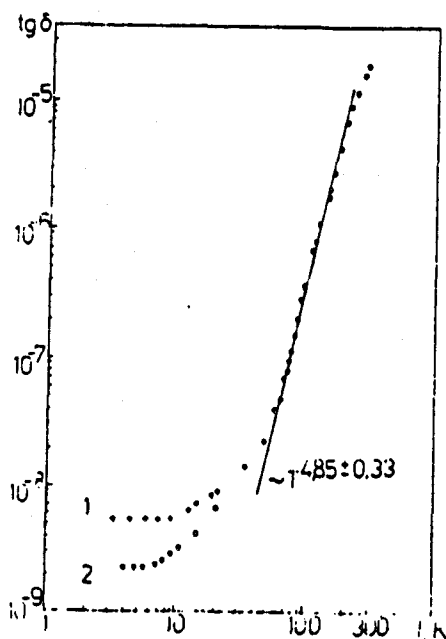


Fig. 2. Microwave absorption in sapphire, Q-band (36 GHz).

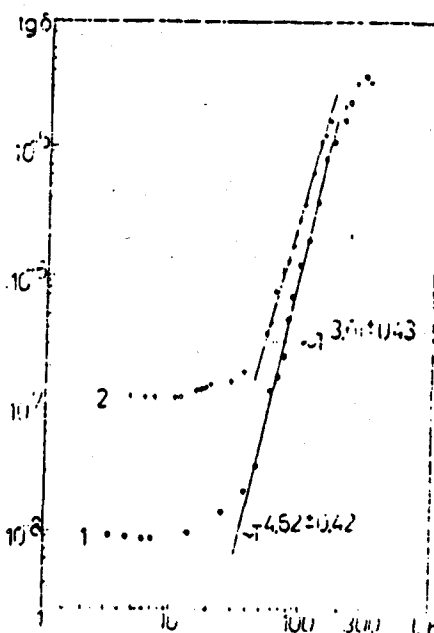


Fig. 3. Microwave absorption in sapphire (M-band, 72 GHz, curve 1), and in YAG (Q-band, 36 GHz, curve 2).

$\tan \delta \sim T^\alpha$.

This is a lower limit of the dielectric losses at a given frequency, which is independent of the variation of the defect structure and is evidently of fundamental origin. The linear regression analysis yielded the following values for the order index α :

$$\alpha = (4.75 \pm 0.10)_{0.9}, \text{ for } 9 \text{ GHz,}$$

$$= (4.85 \pm 0.33)_{0.9}, \text{ for } 36 \text{ GHz,}$$

$$= (4.62 \pm 0.42)_{0.9}, \text{ for } 72 \text{ GHz.}$$

These experimental values of α are close to the prediction (1) of the Gurevich theory [11,12] for hexagonal crystals, but the suspended mean value of α indicates a small significant difference with $\alpha_{\text{theor}} = 5$. In the area of the supposed fundamental loss the results obtained for the samples varying in structure do not differ significantly. In this area figs. 1 and 2 present the data obtained for one of the samples.

At low temperatures $T < 50 \text{ K}$ the value of $\tan \delta$ is greatly affected by the level of crystal perfection. The sapphire samples used in the experiment were tested

by the Berg-Barrette X-ray scattering method. The sample, corresponding to the curve 1 in fig. 1 revealed a well-resolved block structure with a disorientation angle of about 1° . This sample had the largest $\tan \delta$ at liquid-helium temperatures in the X-band: $\tan \delta = (1.2 \pm 0.2) \times 10^{-7}$ below 10 K. The rest of the samples under study displayed no obvious block structure (a disorientation angle of less than several angular minutes), but they differed in the growth rate set in the process of their crystallization. Curves 2 in fig. 1 and 1 in fig. 2 correspond to the samples grown at a crystallization rate of 8 mm/h, and the lowest curves 3 in fig. 1 and 2 in fig. 2 correspond to the rate of 4 mm/h. The helium-temperature $\tan \delta$ values in the faster-grown sample are approximately twice as large as those in the slower-grown one in both X- and Q-bands, being however considerably lower than in the sample with the well-resolved block structure. (The former had been grown at high crystallization speed.)

As found experimentally [1], a growth rate directly determines the perfection parameters of the sapphire crystals: concentration of the residual doping atoms,

dislocations, as well as the residual lattice defects. It is reasonable to conclude then that the observed correlation between $\tan \delta$ and growth rate below 50 K is also due to the variation of the residual lattice imperfections level.

The smallest obtained value of $\tan \delta$ in sapphire was $(7 \pm 1) \times 10^{-10}$ in the X-band ($T \approx 4$ K, frequency is near 9 GHz).

The comparison of $\tan \delta$ values at three frequencies in the area of the proposed lattice losses $50 \text{ K} < T < 200 \text{ K}$ shows that the absorption increase with frequency is stronger than the linear one, as predicted by the Gurevich theory for hexagonal crystals. The $\tan \delta$ versus ω plot at a fixed temperature can be approximated by the function ω^β , where β does not depend on temperature within the interval 60–150 K. The estimate for β is $(1.7 \pm 0.2)_{0.7}$.

The frequency dependence of the dielectric losses at near-helium temperatures is near-linear: $\tan \delta \sim \omega^{\beta_1}$ where $\beta_1 = (0.89 \pm 0.12)_{0.7}$. The analysis is based on the comparison of the data obtained with DRR cut of the same sapphire specimen: curve 2 in fig. 1, 1 in fig. 2, and 1 in fig. 3.

Fig. 3 shows also the results of $\tan \delta$ measurements in a cubic crystal of the yttrium-aluminium garnet (YAG) in the Q-band (curve 2). Between 50 and 150 K the $\tan \delta$ versus T plot of YAG is proportional to T^{α_1} where $\alpha_1 = (3.61 \pm 0.43)_{0.9}$, in good agreement with the prediction (2) by the Gurevich theory for cubic crystals. The low-temperature saturation observed at $T < 50$ K is presumably of extrinsic origin. The Q-band DRR made of YAG had a cylindrical shape with a diameter of 33 mm.

3. Discussion

As the main result of this paper we stress the observation of a fundamental lower limit of microwave absorption in sapphire in a number of samples at different frequencies. The multiple reproduction of the experimentally obtained $\tan \delta \sim T^\alpha$ dependence in the temperature interval between 50 and 200 K, the absence of a significant difference between the estimates of α_{exp} through a wide range of microwave frequencies, as well as closeness of α_{exp} to the theoretical prediction [11,12] make it possible to deduce the fundamental, or intrinsic origin of the measured dielectric losses above 50 K.

The small significant variance of α_{exp} from the theoretical prediction for hexagonal crystals, $\alpha_{theor} = 5$, as well as the considerable disagreement of theory and experiment in the frequency dependence of the absorption ($\beta_{exp} = (1.7 \pm 0.2)_{0.7}$, contrary to $\beta_{theor} = 1$ for hexagonal crystals), should in our opinion not be regarded as a refutation of the theory [11–13]. First of all, the mismatch can be explained by the fact that sapphire, strictly speaking, does not belong to the hexagonal symmetry class. The sapphire lattice $\bar{3}m$ (D_{3d}) is of rhombohedral symmetry. But its acoustic spectrum does not differ much from that of a hexagonal crystal, and that gives reason to compare experimental results with prediction (1): $\tan \delta \sim \omega^1 T^5$. For rhombohedral crystals, formula (2) should be taken: $\tan \delta \sim \omega^2 T^4$. In the experiment we observed, in fact, an intermediate situation: $\tan \delta \sim \omega^{1.7} T^{4.75}$.

As for the numerical comparison of theory and experiment, there is a satisfactory agreement of the experimental data with the "hexagonal" prediction (1). The results of numerical comparison at $T = 78$ K are given in table 1 (the parameters for calculations are the same as those used above).

It should be mentioned, however, that for the correct application of the Gurevich theory to sapphire, special study of the sapphire's elasticity spectrum is required, and probably some development of the theory itself as well.

The experimental data about the extrinsic absorption in sapphire below 50 K do not permit a firm conclusion through which particular type of the defects it is produced. A weak-temperature dependence and an approximately linear $\tan \delta$ versus ω plot of the extrinsic dielectric losses make it possible to

Table 1

A numerical comparison of the experimental data on sapphire's loss tangent at $T = 78$ K with the theoretical prediction [11–13] for hexagonal and rhombohedral crystals.

Frequency (GHz)	$(\tan \delta)_{exp}$	$(\tan \delta)_{theor}$	
		hexagonal lattice	rhombohedral lattice
9	1.5×10^{-9}	1.5×10^{-9}	8×10^{-9}
36	1.1×10^{-7}	6×10^{-8}	1.4×10^{-7}
72	4.3×10^{-7}	1.2×10^{-7}	6×10^{-6}

suppose their relation to the chaotically distributed charged point defects [18]. But one cannot exclude a possible main influence of the 2D dislocations (block bounds), because this type of the defects, while producing the "direct" extrinsic absorption, requires the $\tan \delta \sim \omega/T^0$ plot as well [19]. We should stress here, that this mechanism has nothing in common with the scattering of a mode's EM field by the block-structure-caused ϵ variations. It is sufficient to say that the former model would require, at our experimental conditions, an inversed frequency plot: $Q^{-1} \sim \omega^{-2}$.

The correlation between the value of extrinsic absorption and the residual crystal imperfections gives reason to suggest an opportunity of obtaining an ultra-low microwave absorption in sapphire if the residual defect structure be expelled. Extrapolation of the experimentally found intrinsic absorption temperature plot gives the forecast of $\tan \delta \approx 5 \times 10^{-15}$ at $T=4$ K and a frequency 10 GHz, which corresponds to the DRR quality $Q \approx 2 \times 10^{14}$.

It should be mentioned here that practical design of such an ultra-high Q dielectric ring resonator would require, first, a qualitative breakthrough in crystal-growth technology, and second, a removal of numerous parasitic channels of dissipation. Nevertheless, it is a great experimental challenge, since the availability of 10^{14} quality resonators would allow to perform a number of new important physical experiments.

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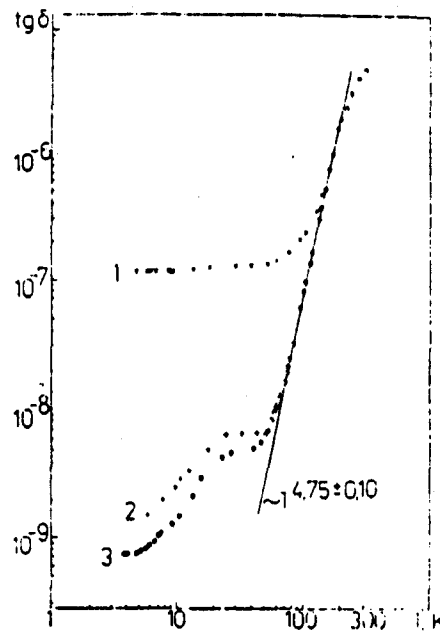


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The comparison of $\tan \delta$ values at three frequencies in the area of the proposed lattice losses $50 \text{ K} < T < 200 \text{ K}$ shows that the absorption increase with frequency is stronger than the linear one, as predicted by the Gurevich theory for hexagonal crystals. The $\tan \delta$ versus ω plot at a fixed temperature can be approximated by the function ω^β , where β does not depend on temperature within the interval 60–150 K. The estimate for β is $(1.7 \pm 0.2)_{0.7}$.

The frequency dependence of the dielectric losses at near-helium temperatures is near-linear: $\tan \delta \sim \omega^{\beta_1}$ where $\beta_1 = (0.89 \pm 0.12)_{0.7}$. The analysis is based on the comparison of the data obtained with DRR cut of the same sapphire specimen: curve 2 in fig. 1, 1 in fig. 2, and 1 in fig. 3.

Fig. 3 shows also the results of $\tan \delta$ measurements in a cubic crystal of the yttrium-aluminium garnet (YAG) in the Q-band (curve 2). Between 50 and 150 K the $\tan \delta$ versus T plot of YAG is proportional to T^{α_1} where $\alpha_1 = (3.61 \pm 0.43)_{0.9}$, in good agreement with the prediction (2) by the Gurevich theory for cubic crystals. The low-temperature saturation observed at $T < 50$ K is presumably of extrinsic origin. The Q-band DRR made of YAG had a cylindrical shape with a diameter of 33 mm.

3. Discussion

As the main result of this paper we stress the observation of a fundamental lower limit of microwave absorption in sapphire in a number of samples at different frequencies. The multiple reproduction of the experimentally obtained $\tan \delta \sim T^\alpha$ dependence in the temperature interval between 50 and 200 K, the absence of a significant difference between the estimates of α_{exp} through a wide range of microwave frequencies, as well as closeness of α_{exp} to the theoretical prediction [11,12] make it possible to deduce the fundamental, or intrinsic origin of the measured dielectric losses above 50 K.

The small significant variance of α_{exp} from the theoretical prediction for hexagonal crystals, $\alpha_{theor} = 5$, as well as the considerable disagreement of theory and experiment in the frequency dependence of the absorption ($\beta_{exp} = (1.7 \pm 0.2)_{0.7}$, contrary to $\beta_{theor} = 1$ for hexagonal crystals), should in our opinion not be regarded as a refutation of the theory [11–13]. First of all, the mismatch can be explained by the fact that sapphire, strictly speaking, does not belong to the hexagonal symmetry class. The sapphire lattice $\bar{3}m$ (D_{3d}) is of rhombohedral symmetry. But its acoustic spectrum does not differ much from that of a hexagonal crystal, and that gives reason to compare experimental results with prediction (1): $\tan \delta \sim \omega^1 T^5$. For rhombohedral crystals, formula (2) should be taken: $\tan \delta \sim \omega^2 T^4$. In the experiment we observed, in fact, an intermediate situation: $\tan \delta \sim \omega^{1.7} T^{4.75}$.

As for the numerical comparison of theory and experiment, there is a satisfactory agreement of the experimental data with the "hexagonal" prediction (1). The results of numerical comparison at $T = 78$ K are given in table 1 (the parameters for calculations are the same as those used above).

It should be mentioned, however, that for the correct application of the Gurevich theory to sapphire, special study of the sapphire's elasticity spectrum is required, and probably some development of the theory itself as well.

The experimental data about the extrinsic absorption in sapphire below 50 K do not permit a firm conclusion through which particular type of the defects it is produced. A weak-temperature dependence and an approximately linear $\tan \delta$ versus ω plot of the extrinsic dielectric losses make it possible to

Table 1
A numerical comparison of the experimental data on sapphire's loss tangent at $T = 78$ K with the theoretical prediction [11–13] for hexagonal and rhombohedral crystals.

Frequency (GHz)	$(\tan \delta)_{exp}$	$(\tan \delta)_{theor}$	
		hexagonal lattice	rhombohedral lattice
9	1.5×10^{-8}	1.5×10^{-8}	8×10^{-9}
36	1.1×10^{-7}	6×10^{-8}	1.4×10^{-8}
72	4.3×10^{-7}	1.2×10^{-7}	6×10^{-8}