Use of Whispering-Gallery Modes and Quasi-TE_{0np} Modes for Broadband Characterization of Bulk Gallium Arsenide and Gallium Phosphide Samples

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***Abstract*—The complex permittivity of bulk crystals of semiinsulating gallium arsenide (GaAs) and gallium phosphide (GaP) were measured over the frequency range from 4 to 30 GHz and at temperatures from 30 up to 300 K employing whispering-gallery-mode (WGM) and quasi-TE_{0np}-mode dielectric-resonator techniques. At temperatures about 40 K, dielectric loss tangent values were below 10^{-6} for GaAs and below 10^{-5} for GaP. The use of several WGMs, as well as TE_{0np} modes excited in the same test sample enabled a broad frequency range of measurements (one decade). The real part of the permittivity of GaP and GaAs proved to be frequency independent at microwave frequencies. The dielectric loss tangents of GaAs and GaP increase with temperature and frequency.**

***Index Terms*—Dielectric losses, gallium compound permittivity measurement, semiconductor materials measurements.**

I. Introduction

DIELECTRIC properties of both gallium arsenide (GaAs) and gallium phosphide (GaP) have been measured by many researchers at various frequencies from megahertz to infrared, and the results of those measurements are referred to in many books devoted to A–III–B–V semiconductors, e.g., [1]. Extensive studies of dielectric properties of GaAs at microwave frequencies have been undertaken by Courtney [2]. He performed measurements using the TE_{011}-mode dielectric-resonator technique. Frequency variations were achieved by employing seven samples of different diameters cut from one bulk crystal. The lengths of these samples were progressively shortened to provide series of measurements over the frequency range from 3 to 36 GHz. The thermal coefficient of permittivity and the dielectric loss tangent were measured over the temperature range from 300 to 380 K. Although the results obtained by Courtney until now are considered to be among the most accurate, currently available techniques allow higher precision and more sensitive measurements to be made, especially at cryogenic temperatures where dielectric loss tangents become very small. The whispering-gallery-mode (WGM) [3], [4] and the quasi-TE_{0np}-mode [5] dielectric-resonator techniques are the most accurate for measurements of very low-loss dielectrics. At microwave frequencies and cryogenic temperatures, the contribution of conductivity to the total dielectric losses in semiinsulating ($\rho > 10^6\ \Omega \cdot \mathrm{cm}$) GaAs and GaP is negligible. Thus, these materials can be treated as dielectrics over this frequency range.

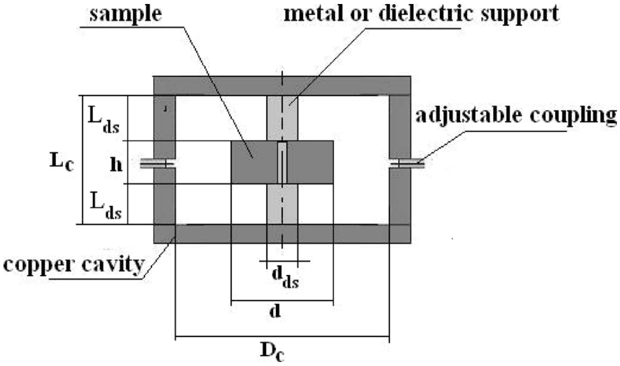

Fig. 1. Sketch of measurement setup used in experiments.

An accurate knowledge of the dielectric properties of GaAs and GaP is essential to the design of microwave devices that can be manufactured employing these materials.

II. Measurements Techniques

In our measurement setup (Fig. 1), the sample under test was housed in a cylindrical metal cavity employing metal or low-loss dielectric supports. The entire structure was mounted on the cold head of a closed-cycle cryocooler for the low-temperature measurements. Dielectric supports were only used for the room-temperature measurements employing quasi-TE_{0np} modes. Adjustable coupling mechanisms were used to control coupling coefficients on both ports of the resonator. The resonators were connected to a network analyzer via semirigid coaxial cables.

In order to find the relationship between the measured resonance frequencies and the real part of the relative permittivity, it is necessary to rigorously solve Maxwell's equations for the structure under test. This was done using Rayleigh–Ritz and mode-matching techniques, which are described in details in [3] and [4]. The dielectric loss tangent for the samples under test was evaluated using (1) as follows:

$$\tan\delta = \frac{(Q_u^{-1} - Q_p^{-1})}{p_e} \tag{1}$$

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where

Q_u	measured unloaded Q factor of a specific mode in the resonator containing the sample under test;
Q_p	Q factor associated with parasitic losses in the cavity including the metal wall losses and dielectric losses of any other support materials not under test;
p_e	electric energy filling factor of the sample under test (the ratio of the electric energy stored in the sample to the electric energy stored in whole resonator) defined as

$$p_e = \frac{\iiint\limits_{Vs} \varepsilon |\mathbf{E}|^2 dv}{\iiint\limits_{Vt} \varepsilon(v) |\mathbf{E}|^2 dv} \tag{2}$$

where

Vs	volume of the sample;
Vt	volume of entire resonant structure;
$\varepsilon(v)$	spatially dependent permittivity inside the entire resonant structure (absolute value);
ε	absolute permittivity of the sample.

The Q factor due to parasitic losses can be found from

$$Q_0^{-1} = Q_d^{-1} + Q_c^{-1} \tag{3}$$

where

Q_d^{-1}	$= p_{ds} \tan \delta_{ds}$;
p_{ds}	electric energy filling factor of dielectric supports;
$\tan \delta_{ds}$	dielectric loss tangent of the dielectric supports;
Q_c	Q factor due to conductor losses in metal cavity walls related by

$$Q_c^{-1} = \frac{R_S}{G} \tag{4}$$

where

R_S	surface resistance of metal cavity walls at a given frequency;
G	geometric factor, defined as

$$G = \omega \frac{\iiint\limits_{Vt} \mu_0 |\mathbf{H}|^2 dv}{\iint\limits_{S} |\mathbf{H}_\tau| \, ds} \tag{5}$$

where

S	internal surface of the cavity;
H_τ	component of the magnetic field tangential to the internal surface of the cavity.

Formulas (1)–(5) were used to determine the dielectric loss tangent from measurements performed on the quasi-TE_{0np} modes. The Q factors associated with parasitic losses for the WGMs with large azimuthal mode indices were extremely large in both GaAs and GaP samples. In such cases, parasitic losses were neglected in (3).

III. ERROR ANALYSIS

A. Permittivity

Permittivity is determined as a numerical solution of a nonlinear equation resulting from the application of a specific method intended for rigorous electromagnetic analysis of the resonant structure under test. In general, for low-loss dielectric samples, permittivity is a function of the resonance frequency of a specific mode and dimensions of the resonant structure. Additionally, if sample supports are made of dielectric, the permittivity of the dielectric supports should be taken into account. This can be formally written as follows:

$$\varepsilon_r' = F(d, h, f_m, D_c, L_c, d_{ds}, L_{ds}, \varepsilon_{rds}') \tag{6}$$

where

ε_r'	relative permittivity of the sample;
ε_{rds}'	relative permittivity of the dielectric support, meaning of all other variables, as seen in Fig. 1.

Assuming that the electromagnetic analysis technique provides accurate solutions of (6), one can evaluate the total B-type uncertainty (nonstatistical uncertainty including calibration errors) of the real part of permittivity from (7). We can write

$$\begin{aligned}\left(\frac{\Delta\varepsilon_r'}{\varepsilon_r'}\right)^2 = {} & S_d^2\left(\frac{\Delta d}{d}\right)^2 + S_h^2\left(\frac{\Delta h}{h}\right)^2 + S_{fm}^2\left(\frac{\Delta f_m}{f_m}\right)^2 \\ & + S_{Dc}^2\left(\frac{\Delta D_c}{D_c}\right)^2 + S_{Lc}^2\left(\frac{\Delta L_c}{L_c}\right)^2 \\ & + S_{ds}^2\left(\frac{\Delta d_s}{d_s}\right)^2 + S_{Ls}^2\left(\frac{\Delta L_s}{L_s}\right)^2 + S_{\varepsilon s}^2\left(\frac{\Delta \varepsilon_s}{\varepsilon_s}\right)^2\end{aligned} \tag{7}$$

where $S_x = (\partial\varepsilon_r'/\partial x)(x/\varepsilon_r')$—the sensitivity coefficient is related to the quantity x (where the subscripts found in Fig. 1 are denoted by x). Δf_m is the uncertainty in the measurement of the mode frequency f_m.

For WGMs, the dominant part of the electromagnetic energy is concentrated in a small area near the lateral surface of the sample. The electric energy is distributed in such a way that approximately 96% is inside the dielectric and only approximately 4% is in the air/vacuum space inside the cavity. The larger the azimuthal mode number, and the larger the permittivity of the sample, the better confinement of the electric field inside the sample.

For WGMs, electromagnetic fields are negligibly small in the relatively large region near the resonator axis. As a result, the supports may be manufactured from arbitrary material, but provided their diameter is smaller than 1/3 the diameter of the sample, without any measurable change in the WGM resonance frequencies.

The cavity is also constructed such that the dielectric sample is located well away from both the lateral surface and end plates of the cavity. In such cases, (7) can be simplified by neglecting terms related to the dielectric supports and the dimensions of the cavity, and is written as

$$\left(\frac{\Delta\varepsilon_r'}{\varepsilon_r'}\right)^2 = S_d^2\left(\frac{\Delta d}{d}\right)^2 + S_h^2\left(\frac{\Delta h}{h}\right)^2 + S_{fm}^2\left(\frac{\Delta f_m}{f_m}\right)^2. \quad (8)$$

B. Dielectric Loss Tangent

The B-type uncertainty of the dielectric loss tangent determination can be evaluated from (1). A detailed analysis leads to the following formula:

$$\left(\frac{\Delta\tan\delta}{\tan\delta}\right)^2 = S_{Qu}^2\left(\frac{\Delta Q_u}{Q_u}\right)^2 + S_{Qp}^2\left(\frac{\Delta Q_p}{Q_p}\right)^2 + S_{pe}^2\left(\frac{\Delta p_e}{p_e}\right)^2 \quad (9)$$

where

$$S_{Qu} = -\frac{Q_p}{Q_p - Q_u}$$
$$S_{Qp} = \frac{Q_u}{Q_p - Q_u}$$
$$S_{pe} = -1.$$

One can notice that the relative errors of the dielectric loss tangent determination increase to infinity when the total measured unloaded Q factor (Q_u) approaches the value of the Q factor due to parasitic losses (Q_p). However, for high-order WGMs, the Q factors due to parasitic losses become very large so their inverse can be neglected, even for the lowest loss materials like sapphire ($\tan\delta$ of sapphire is the order of 10^{-9} at cryogenic temperatures). In such cases, $S_{Qu} \approx -1$ and $S_{Qp} \approx 0$. Thus, the relative uncertainties in the dielectric loss tangent determination are similar to the relative errors in the unloaded Q-factor measurements (the electric energy filling factor is usually determined with a relative uncertainty similar to that in the real part of permittivity).

Unfortunately, it is commonly observed that the resonance line shape of WGMs is not Lorentzian because of mode degeneration. For this reason, the relative uncertainty in the unloaded Q-factor measurements for WGMs can be as large as 10%, as compared to only 1% uncertainty for well-behaved quasi-TE_{0np} modes. On the other hand, the presence of parasitic losses for quasi-TE_{0np} modes makes measurement of extremely low-loss dielectrics practically impossible, while the relative errors in dielectric loss tangent measurements, using WGMs, remain constant for arbitrarily low dielectric loss tangent values.

IV. RESULTS OF EXPERIMENTS

Measurements were performed on both a cylindrical GaAs sample with a diameter of 25.39 ± 0.01 mm and a height of 6.25 ± 0.01 mm and on a cylindrical GaP sample with a diameter of 48.12 ± 0.03 mm and a height of 5.00 ± 0.01 mm. The sample dimensions were measured at room temperature (296 K), while the dimensions of these samples at other temperatures were evaluated by taking into account the temperature-dependent thermal expansion coefficients for both materials [6], [7]. The samples were mechanically polished on all surfaces. The internal dimensions of the cylindrical cavities used in WGM measurements are as follows. The cavity used with the GaAs sample has a diameter of 34.6 mm and a height of 25.8 mm. The cavity used with the GaP sample has a diameter of 60.0 mm and a height of 43.0 mm. The cavity used for the quasi-TE_{0np}-mode measurements of both the GaAs and GaP samples has a diameter of 60.0 mm and a height of 30.14 mm.

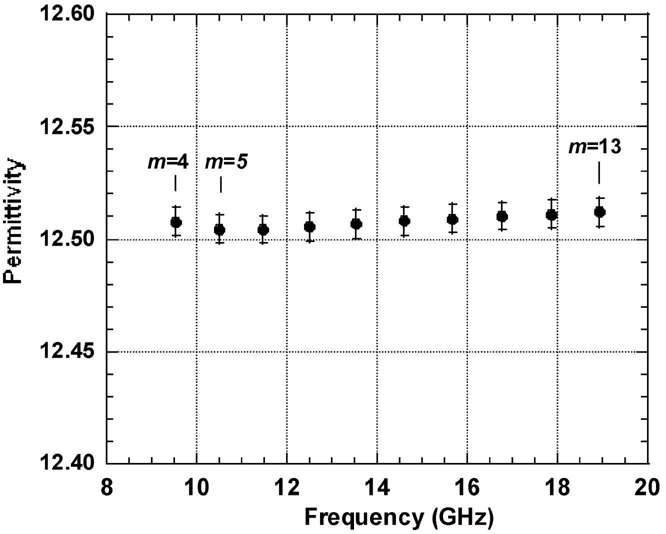

Fig. 2. Permittivity versus frequency for GaAs sample at 41 K evaluated from the resonance frequencies of WGMs belonging to the $N1$-mode family. The parameter m denotes the azimuthal mode number.

Initially the real part of the relative permittivity was determined for both samples on the basis of measurements of the resonance frequencies of the quasi-TE_{011} modes. Once permittivity values were determined, the resonance frequencies of the higher order quasi-TE_{0np} modes and of the WGMs were computed. The resonance frequencies and Q factors for all those modes were then measured. Such a measurement procedure was performed at room temperature and at the lowest temperature that was achieved in the cryocooler (approximately 41 K, depending on the cavity used). Finally, the real part of permittivity was evaluated on the basis of those measurements. Results of the permittivity determinations were very consistent and frequency independent. The highest accuracy of the permittivity determination was obtained at the lowest cryogenic temperatures because, at these temperatures, the resonance lines are very narrow and the influence of temperature instabilities on the measured frequencies is minimized. The results of the permittivity determination for GaAs based on the subsequent measurement of ten WGMs belonging to the $N1$-mode family are shown in Fig. 2.

One can notice that the standard deviation with respect to the average permittivity value is approximately 0.003, which is less than 0.03%. The total relative uncertainty of the permittivity determination can be evaluated from (8). In Table I, the sensitivity coefficients of permittivity are shown for two WGMs having extreme azimuthal mode numbers $m = 4$ and $m = 13$. For all other modes, with $4 < m < 13$, S_d and S_h take intermediate values between those that are shown in Table I.

It is seen that $S_d + S_h \approx 2$ and $S_{fm} > 2$. One can prove that the sensitivity coefficient S_{fm} is related to the electric energy filling factor by the expression $S_{fm} = 1/(2p_e)$.

TABLE I
SENSITIVITY COEFFICIENTS OF PERMITTIVITY FOR THE GaAs SAMPLE

m	S_d	S_h	S_{fm}
5	1.169	0.831	2.109
13	1.744	0.256	2.022

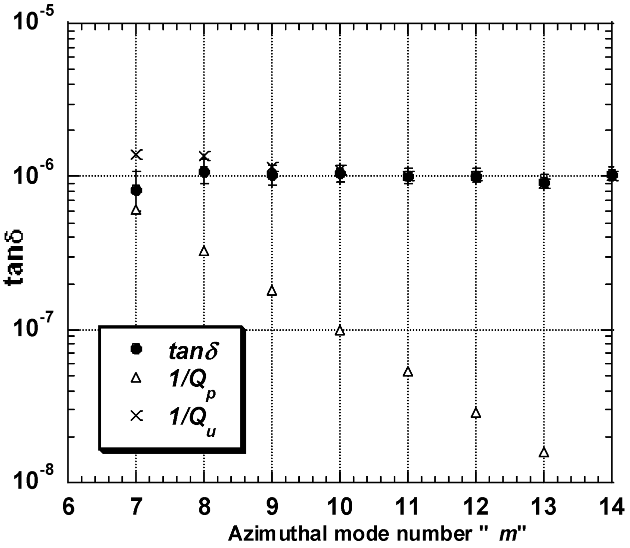

Fig. 3. Dielectric loss tangent for the GaAs sample at 41 K evaluated from resonance frequencies of WGMs belonging to the $N1$-mode family (the same as in Fig. 2).

Taking into account the dimensional uncertainties of the samples under test and the uncertainties of the resonance frequencies, the total uncertainty in the permittivity was evaluated and was found to be smaller than 0.2% for all WGMs with $m > 4$ in both GaAs and GaP samples. In the uncertainty evaluation, the absolute error of the resonance frequencies measurements Δf_m was assumed to be 5 MHz. This is a rather conservative assumption, and it corresponds to the maximum difference between two split modes of an otherwise degenerate pair (mode splitting results from imperfect geometry—the lack of perfect axial symmetry and all modes with $m \neq 0$ are split). In Fig. 3, the results of dielectric loss tangent determination for the GaAs sample are shown.

The uncertainties in the dielectric loss tangent have been numerically evaluated assuming a relative uncertainty in the unloaded Q-factor measurements of 10% and a relative error in the Q factor due to parasitic losses of 15%. Such a conservative assumption for the evaluation of the latter is related to the uncertainty of the surface resistance determination at cryogenic temperatures. However, it is clearly visible in Fig. 3 that parasitic losses can be neglected for the WGMs where $m > 9$. In such cases, the dielectric loss tangent can be simply evaluated from

$$\tan\delta = \frac{1}{(Q_u p_e)} \approx \frac{1}{Q_u}. \tag{10}$$

Indeed the curves corresponding to $\tan\delta$ and $1/Q_u$ in Fig. 3 overlap for $m > 9$. One should also note that the diameter of the copper cavity that was used for the GaAs measurements at cryogenic temperatures was relatively small. The distance between the lateral surface of the GaAs sample and the lateral surface of the cavity was only approximately 4.6 mm. For this reason, the Q factors due to parasitic losses were not as large as they would had been if a cavity with a larger diameter was used.

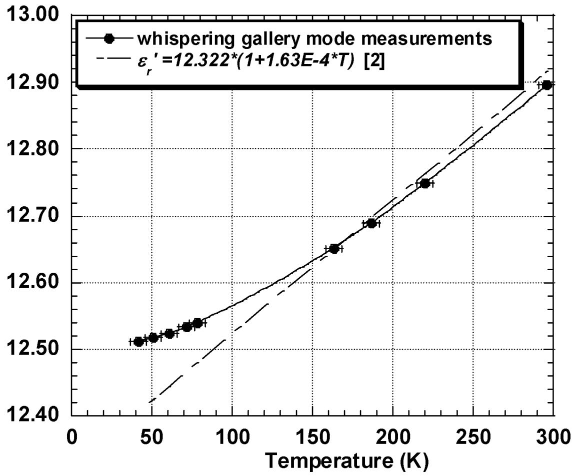

Fig. 4. Permittivity versus absolute temperature for the GaAs sample. The experimental data points were extracted from measurements of the WGM with frequencies near 18.9 GHz ($N1,\ m = 13$ WGM).

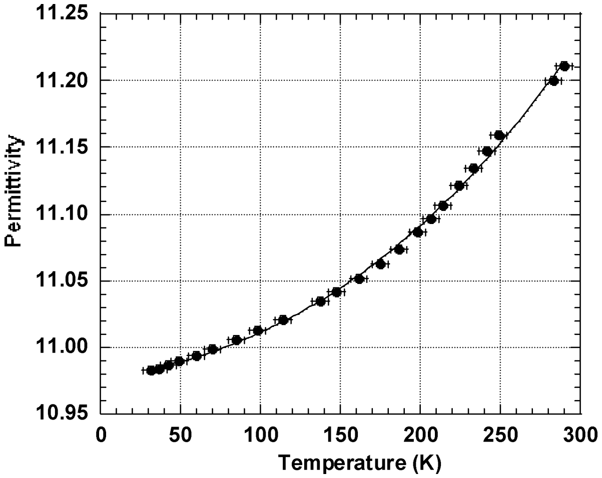

Fig. 5. Permittivity versus absolute temperature for the GaP sample. The experimental data points were extracted from measurements of the WGM with frequencies near 10.8 GHz ($S1,\ m = 11$ WGM).

Variable temperature permittivity measurements were performed employing only one WGM ($N1$ WGM with azimuthal index $m = 13$ for GaAs and $S1$ WGM with azimuthal index $m = 11$ for GaP). The results of the measurements for GaAs are shown in Fig. 4 and for GaP in Fig. 5. In Fig. 4, the results of the permittivity determination based on Courtney's measurements and the simple formula given in [2] (straight line) are also presented.

It is seen that agreement between our results and Courtney's results is excellent at temperatures above 200 K. Below 200-K temperature, variations of permittivity, as well as variations of the thermal expansion coefficient become smaller and smaller,

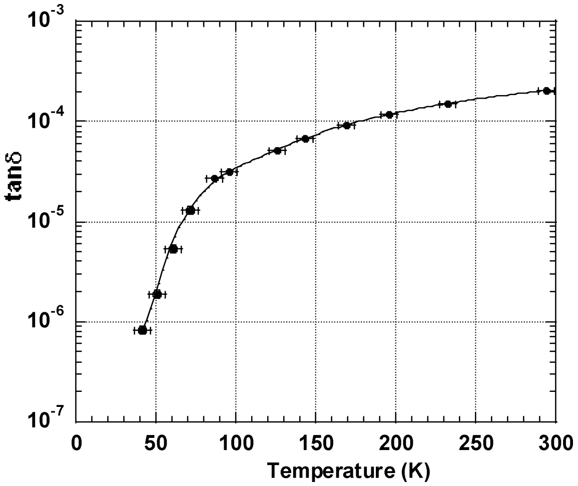

Fig. 6. Dielectric loss tangent versus absolute temperature for the GaAs sample measured at frequencies near 18.9 GHz ($N1,\ m = 13$ WGM) at temperatures below 80 K and near 12.4 GHz ($N1,\ m = 7$ WGM) at temperatures above 80 K). The horizontal error bars indicate the uncertainty associated with the sensor attached to the base of the copper cavity reading differently from the actual temperature of the crystal.

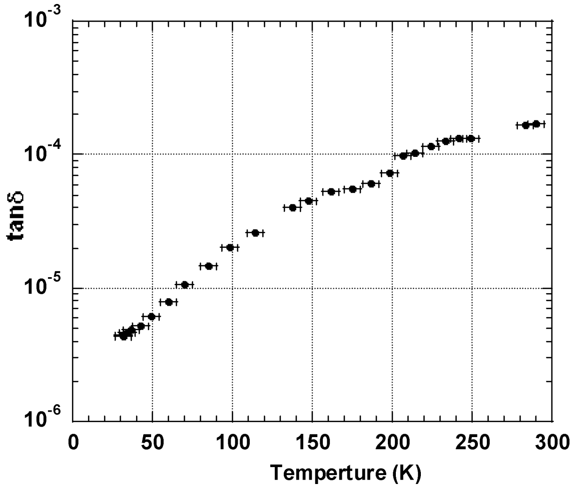

Fig. 7. Dielectric loss tangent versus absolute temperature for the GaP sample measured at frequencies near 10.8 GHz ($S1,\ m = 11$ WGM).

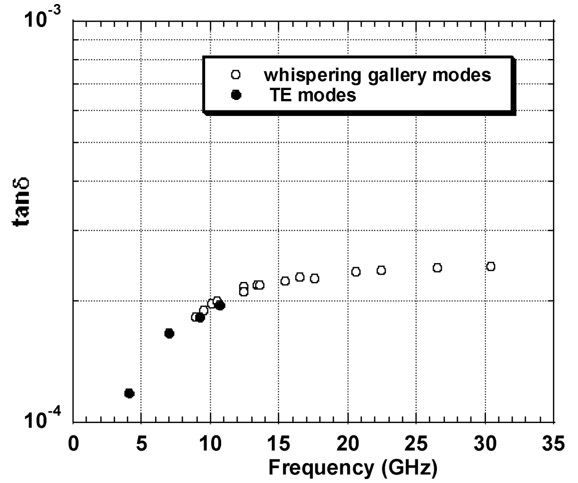

Fig. 8. Dielectric loss tangent versus frequency for the GaAs sample measured at room temperature employing various modes.

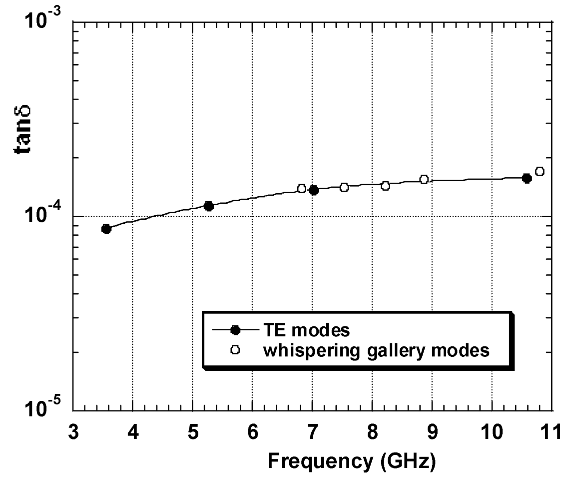

Fig. 9. Dielectric loss tangent versus frequency for the GaP sample measured at room temperature employing various modes.

which is typical for other single-crystal dielectrics such as sapphire, YAG [4], and silicon [8]. It should be mentioned, however, that Courtney performed his measurements over a temperature range from 300 to 380 K so conclusions from [2] are only valid over this temperature range.

Temperature variations of the dielectric loss tangent for both GaAs and GaP samples were measured using high-order WGMs. For the GaAs sample, at temperatures below 80 K, the WGM belonging to the $N1$-mode family with an azimuthal mode index $m = 13$ was used (the same as in the permittivity measurements). At temperatures higher than 80 K, this mode was not coupled well, and its resonance curve became noisy, thus a lower order ($m = 7$) WGM was used instead. One should notice that, at 41 K, the measured Q-factor value for the $N1$, $m = 13$ mode was more than one million, but at 78 K, it was "only" 32 000. The temperature variations of the dielectric loss tangent of GaP were smaller than those for GaAs so only one WGM ($S1,\ m = 11$) was needed over the entire temperature range.

The dielectric loss tangent measurements, as a function of temperature, are shown in Fig. 6 for GaAs and in Fig. 7 for GaP. The dielectric loss tangent was measured as a function of frequency using four quasi-TE$_{0np}$ modes and several WGMs excited in the same sample. Such an approach allowed us to avoid systematic errors related to different losses in different samples when several samples are used. For GaAs, at frequencies higher than 15 GHz, the dielectric losses become almost constant, as is seen in Fig. 8. For GaP, the dielectric losses increase very slowly with frequency above 8 GHz (Fig. 9). Such behavior is not typical for dielectrics. For most nonorganic dielectrics, e.g., sapphire, YAG, or microwave ceramic materials, the dielectric losses increase linearly with frequency. There are only a few nonorganic materials, e.g., single crystal quartz, with such behavior; however, many polymers have a similar dependence to GaAs and GaP.

V. CONCLUSIONS

The dielectric permittivity and loss tangents of GaAs and GaP have been determined from 30 to 300 K using both TE and WGM techniques. In both samples, these were also determined as functions of microwave frequency from 3 to 30 GHz for GaAs and from 3 to 11 GHz for GaP at room temperature. Additionally, the frequency dependence for GaAs was determined from 9 to 19 GHz at 41 K.

It has been shown that very accurate broad frequency range measurements of low-loss dielectrics can be performed using a combination of two measurements techniques, namely, WGMs and quasi-TE_{0mp} modes excited in the same sample of the material under test. The total relative measurement uncertainty for the real part of permittivity is better than 0.2%. It should also be emphasized that the sensitivity of the dielectric loss tangent determination from WGMs allows the measurement of materials with extremely low losses.

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