

# Ultra-Low Absorption Measurement in Dielectrics in Millimeter- and Submillimeter-Wave Range

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**Abstract**— A measurement of ultra-low absorption of microwave radiation in dielectrics is reported. Two Fabry–Perot resonators with  $\geq 600\,000$  quality factors, fully general-purpose interface bus programmable millimeter-wave frequency synthesizer with 10–15-mW continuous wave (CW) power level, 100-Hz frequency resolution from 78 to 118 GHz, and corresponding hardware and software for signal processing were used. The  $\pm 500$ -Hz accuracy of resonance curve width measurements was reached. This high accuracy allowed loss tangent measurement as small as  $10^{-6}$ – $10^{-7}$  in dielectric samples with a thickness of  $\sim 0.5$  mm. A convenient method of measurements of almost arbitrary plane parallel samples has been developed and described. Practical applications such as development and control of thin low-loss resonant windows of powerful ( $\sim 1$ -MW CW) gyrotrons used in thermonuclear experiments, precise reflection coefficient of metals measurements, as well as other applications are discussed. The existence of such technique up to frequencies exceeding 1 THz makes measurements described at the whole millimeter- and submillimeter-wave bands affordable.

**Index Terms**— Backward wave oscillators, dielectric losses, Fabry–Perot resonators, millimeter- and submillimeter-wave spectroscopy, millimeter-wave measurements, submillimeter-wave measurements.

## I. INTRODUCTION

IN MILLIMETER- and submillimeter-wave bands, the open Fabry–Perot resonators are uniquely suitable for studying dielectric properties of low-loss materials. The absorption measurement in this case comes to measurement of quality factor  $Q$  of the empty ( $Q_0$ ) and loaded ( $Q_L$ ) resonator. An especially simple relation between absorption characterized by loss tangent ( $\tan \delta$ ) and quality factor is for the resonator completely filled by dielectric (e.g., gas)

$$\tan \delta = 1/Q_L - 1/Q_0. \quad (1)$$

There is a sufficient variety of measurement and calculation techniques on the base of Fabry–Perot resonators to obtain the dielectric properties of solid or liquid samples. However, in any case, all the information about the absorption in dielectrics is contained in a broadening of resonance curve width of loaded resonator in comparison with the empty one.

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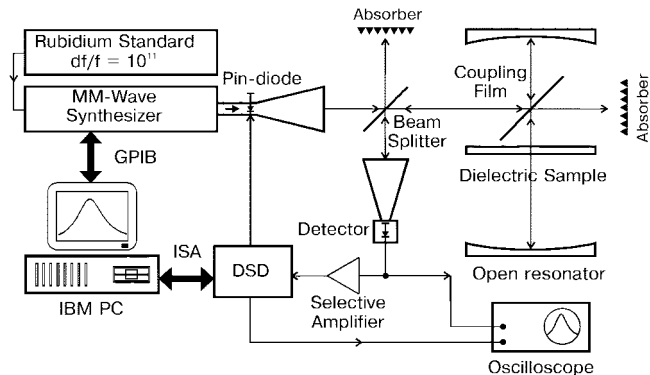


Fig. 1. The block-diagram of experimental setup.

For achieving the highest sensitivity of absorption measurements, the highest  $Q$ -value of Fabry–Perot resonators together with the precise methods of the resonance curve width measurements should be developed. Thus, this paper is devoted to approaching the highest resonator  $Q$ -value, as well as the greatest possible accuracy of its measurements.

Let us consider analogous papers published earlier. One of the most similar to this paper is [1], which was also done in a 100-GHz frequency range, where the resonator with  $Q = 110\,000$  and the sixth harmonic of a microwave synthesizer as a radiation source were used. Komiya *et al.* [1] reached 10% accuracy in the measurements of the loss tangent of the order of  $10^{-4}$ . In [2], which is related to this topic, the high- $Q$  ( $\sim 100\,000$ ) hemispherical Fabry–Perot cavity together with stabilized synthesized phase locked 60-GHz Gunn oscillator source and superheterodyne receiver enabled Afsar *et al.* to measure loss tangent value as low as  $10\ \mu\text{rad}$ .

A combination of the Fabry–Perot resonator with  $Q$  as high as 600 000 and a broad-band (78–118 GHz) precisely controlled and powerful enough frequency synthesizer as a radiation source used in the present work permitted us to reach the new level of sensitivity of absorption measurements. It corresponds to  $\pm 500$ -Hz accuracy of resonance width measurements, which permits us to detect absorption as low as loss tangent of  $\sim 10^{-8}$  in the case of a completely filled cavity. Sensitivity for various practical cases is calculated in Section III.

## II. EXPERIMENT

The block diagram of experimental setup is shown in Fig. 1 and the main parameters of resonators are shown in Table I.

TABLE I  
THE RESONATOR PARAMETERS

	Resonator with "glass" mirrors		Resonator with "brass" mirrors <sup>c</sup>		
Resonance Frequency (MHz)	109 200	115 600	108 500	153 600	194 590
Resonance Width (experiment) (MHz)	0.174	0.181	0.172	0.228	0.348
$Q$ -value of Resonator (experiment)	625 000	630 000	630 000	670 000	560 000
Resonator Length (mm)	300	300	397	418	391
Mirror Curvature Radii (mm)	1200.0	1200.0	240.450	240.450	240.450
Mirror Diameters (mm)	110	110	120	120	120
Coupling Film Thickness (mm)	0.006	0.006	0.012	0.012	0.012
Calculated Coupling Losses <sup>a</sup>	$10 \cdot 10^{-5}$	$11 \cdot 10^{-5}$	$38 \cdot 10^{-5}$	$74 \cdot 10^{-5}$	$120 \cdot 10^{-5}$
Calculated Atmospheric Absorption <sup>b</sup>	$3 \cdot 10^{-5}$	$5 \cdot 10^{-5}$	$5 \cdot 10^{-5}$	$7 \cdot 10^{-5}$	$26 \cdot 10^{-5}$
Reflection Losses (experiment)	$97 \cdot 10^{-5}$	$99 \cdot 10^{-5}$	$98 \cdot 10^{-5}$	$120 \cdot 10^{-5}$	$140 \cdot 10^{-5}$
Calculated Reflection Losses <sup>a</sup>	$87 \cdot 10^{-5}$	$90 \cdot 10^{-5}$	$87 \cdot 10^{-5}$	$104 \cdot 10^{-5}$	$117 \cdot 10^{-5}$
Reflection Coefficient (experiment)	0.99903	0.99901	0.99902	0.99880	0.99860

- a) For calculation of coupling and reflection losses we used the next values of:  
teflon refractive index  $n = 1.414$ , and silver conductivity  $\sigma = 5.71 \cdot 10^{17} \text{ s}^{-1}$  (at  $20^\circ \text{ C}$ ).  
b) The calculation of atmosphere absorption losses was made according to [16].  
c) Measurements of  $Q$ -value with "brass" mirrors were made for resonator described in [6].

The microwave synthesizer [3] of 78–118-GHz frequency range is used as a source of radiation. The resonator is coupled with a synthesizer and detector via quasi-optical 3-dB splitter and the coupling Teflon film placed under angle of  $45^\circ$  to the resonator axis. A plane parallel sample of a dielectric is situated at the center of the resonator perpendicular to its axis [4].

Let us write the quality factor of the empty resonator  $Q_0$  in the form

$$1/Q_0 = P_{\text{refl}} + P_{\text{coupl}} + P_{\text{diffr}} + P_{\text{atm}} \quad (2)$$

where  $P_i$  are fractional relative losses of energy during one wave traversing through a resonator due to reflection in mirrors, coupling, diffraction, and absorption in the atmosphere.

It is well seen from (2) that, for getting the high  $Q$ -factor, it is necessary to maximize the reflection coefficient of mirrors and minimize coupling, diffraction, and atmospheric losses. The methods used for this are described below, and the parameters of resonators obtained are listed in Table I.

- 1) The diffraction losses are the easiest to reduce to the negligible value in comparison with the other ones by increasing the mirror diameters and size of a coupling film. In our experiments, the diffraction losses are really negligible.
- 2) The well-known and traditional scheme for us, where the radiation is coupled into and out of the resonator by thin Teflon film placed inside a cavity, provides very broad-band coupling and maximum ( $\geq 20 \text{ dB}$ ) suppression of high-order resonator modes [4]. As a result, maximum signal-to-noise ratio ( $S/N$ ) could be reached. The coupling coefficient can be varied conveniently by changing the film thickness (coupling films are placed in

an easily removable frame). We used Teflon films with thicknesses of 6 and 12  $\mu\text{m}$ . It was found that, for our case, the further decrease of resonator coupling led to sharp reduction of  $S/N$  without considerable increasing of  $Q$ -value.

- 3) To increase the reflection coefficient, optically polished spherical glass and brass mirrors were coated first by silver and then by aluminum by means of vacuum vaporization in a special chamber evacuated by an oil-free pump down to the pressure  $\simeq 6 \cdot 10^{-5} \text{ Pa}$ . The thickness of silver layer was  $\simeq 0.5 \cdot 10^{-3} \text{ mm}$  and the aluminum one was  $\sim 10^{-5} \text{ mm}$ .

Closeness of calculated and measured reflection losses (difference is approximately 10%) presented in Table I is extremely encouraging to use the method described here for solving a very important problem of reflection coefficient measurements of high conductive metals, alloys, and thin-film coatings. The solution of this problem by other methods is quite difficult (e.g. [5] and [6]), while the difference between calculated and measured reflective losses appears worthy of investigation.<sup>1</sup>

Final parameters of our resonators are listed in Table I. Since, as can be seen, the diffraction losses are negligible ( $\sim 10^{-5}$ ), the coupling losses are already ten times less than the reflection losses and silver is one of the best microwave reflecting materials, the further increase of  $Q$  value is possible only with the use of cryogenically cooled mirrors. However, this is quite expensive and complicated way. To increase resonator length ( $L$ ) is also not a straightforward solution for

<sup>1</sup> It could be due to several reasons, such as a grain structure of silver layer produced by vacuum vaporization, anomalous skin effect, as shown in [7], or uncertainties of calculated conductivity values at that frequency range.

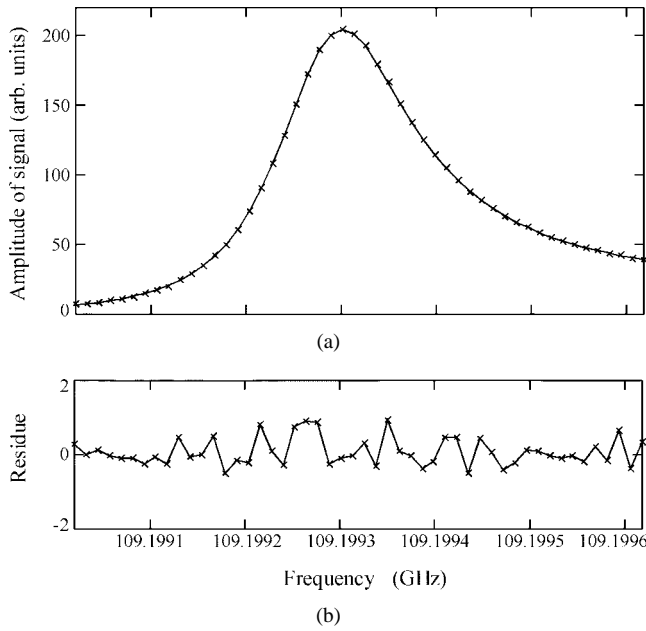


Fig. 2. (a) Fit of the resonance curve at 109 GHz to the function  $a_0 + a_1 \cdot (f - f_0) + L(f)$ , where  $L(f)$  is a Lorentzian line shape. (+ +: experimental values, —: fit calculation), residue of the fit is shown in (b). Frequency scan step is 12.2 kHz, 50 record points, integrating time per point is 15/86 s, resonance curve width obtained from fit is  $\Delta f = 174 \pm 0.78$  kHz ( $Q \approx 627\,000$ ). The asymmetrical curve shape is due to the interference of microwave beams on the detector.

the issue. Thus, as a result, we have got a practical limit of the quality factor of the Fabry–Perot resonator with the noncooled mirrors. It varies from  $Q = 560\,000$  up to  $Q = 670\,000$  for different resonator geometries and working frequencies (80–200 GHz).

The millimeter-wave synthesizer used in our experiments is from the “family” based on Russian backward wave oscillators (BWO’s). These synthesizers are manufactured by the “KVARZ” Institute of Electronic Measurements covering, in summary, a 53–178-GHz frequency range, and are one of the best radiation sources in that wave-band. A detailed description of them is given in [3]. The synthesizer used in our experiments has a minimal frequency step of 100 Hz, the signal from external atomic frequency standard (5 MHz,  $\delta f/f \sim 10^{-11}$ ) is used as the reference clock. The output power of the synthesizer is 10–15 mW. The synthesizer phase noise measured by manufacturer does not exceed  $-80$  dBc/Hz at 100 kHz away from carrier frequency. Another type of experiment proving spectral purity of the synthesizer radiation was observation in [3] of narrow saturation dip (“Lamb dip”) with  $10^{-7}$  fractional width on the top of the Doppler-broadened  $\text{H}_2\text{S}$  spectral line. Effect of source phase noise on our measurements was insignificant. In Figs. 2 and 3, residuals of the fits of experimental points of the resonance curve to the Lorentzian profiles constitute less than 1% of the peak value. All the synthesizer functions were computer controlled via National Instruments’ general-purpose interface bus (NI GPIB) interface. The synthesizer radiation was amplitude modulated by a p-i-n diode placed in the waveguide section between the synthesizer and a horn.

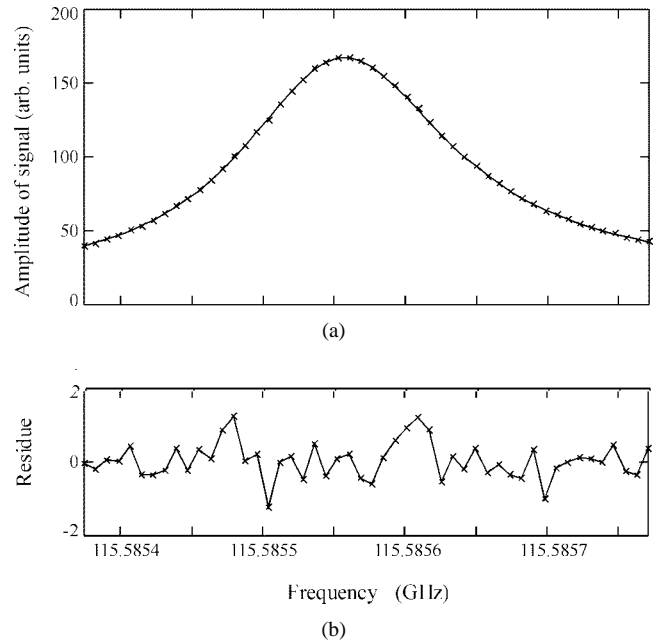


Fig. 3. (a) Fit of the resonance curve to a Lorentzian line shape and the (b) residue of the fit at 115 GHz. Frequency scan step is 8.1 kHz, 50 record points, integrating time 10/86 s,  $\Delta f = 181.52 \pm 1.32$  kHz ( $Q \approx 637\,000$ ). This trace record demonstrates the minimum distortion of the resonance curve achieved by the tuning of the quasi-optical tract.

A silicon point-contact diode was used for microwave signal detection. The signal from the detector was amplified, digitized, and demodulated by an amplifier and a digital synchronous detector (DSD) mounted in a plug-in data acquisition board for an IBM PC AT compatible computer. Description of intelligent DSD will be done elsewhere. A resonance curve obtained by frequency scan through resonance band of a resonator was fitted to the Lorentzian profile by the least-squares method. The software used was developed earlier for studies of molecular spectral lines collisional broadening [8] and will be described elsewhere.

A record of the resonance curve obtained and its fit to Lorentzian shape together with residual of the fit are depicted in Figs. 2 and 3. To fit the experimental curve captured from the synchronous detector, the following model function has been used:

$$S(f) = L(f) \cdot e^{i\phi} + A_1 \cdot (f - f_0) + A_0 \cdot e^{i\theta} \quad (3)$$

where  $L(f)$  is the Lorentzian function,  $A_1$  is the term describing power dependence versus frequency and interference effects,  $A_0$  is the baseline which represents averaged synchronous noise and integrated white Gaussian noise, and  $\phi$  and  $\theta$  are the phases for finding which special algorithm was used.

After the curve fitting, we have obtained central frequency, width, and amplitude of the resonance.

It should be noted that a resonance curve could also be observed in a fast linear-frequency-sweeping regime of the synthesizer. Power of the generator and sensitivity of the detector have permitted us to observe resonance curves on an oscilloscope screen. High sweeping speed gives the advantages

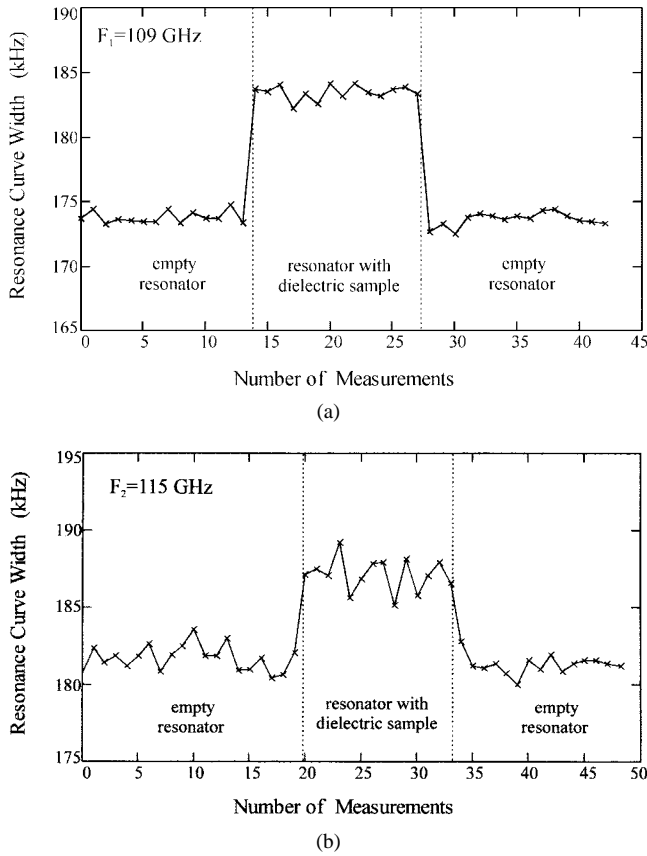


Fig. 4. The statistics of resonance curve width values over many curve records for cases of an empty resonator, with dielectric sample inside, and empty resonator again. (a) Integrating time 15/86 s, fit error 0.78 kHz, mean values of resonance curve widths are:  $\Delta f_{\text{empty}1} = 173.75 \pm 0.44$  kHz,  $\Delta f_{\text{sample}} = 183.43 \pm 0.54$  kHz,  $\Delta f_{\text{empty}2} = 173.6 \pm 0.54$  kHz. (b) Integrating time 10/86 s, fit error 0.68 kHz, mean values of resonance curve widths are:  $\Delta f_{\text{empty}1} = 181.73 \pm 0.82$  kHz,  $\Delta f_{\text{sample}} = 187.13 \pm 1.05$  kHz,  $\Delta f_{\text{empty}2} = 181.31 \pm 0.6$  kHz.

at the adjusting of the installation. For example, we can reduce line-shape distortion, which one is due to interference of quasi-optical beams on the detector (see Fig. 2), as is shown in Fig. 3.

To check the reproducibility of getting experimental data, the repeated measurements of resonance curve parameters have been done. In Fig. 4, a long (more than 40 min) time record of many measurements of the resonance curve width of an empty, loaded, and again empty resonator is shown. This figure demonstrates very high stability and reproducibility of resonance width measurements observed in our experiment over a period of several hours.

Atmospheric perturbation involves many factors which could be responsible for resonance central frequency shifting and resonator  $Q$ -value changing. For example, the absorption and refractive index of air depend on humidity, temperature, and frequency band. In our case, to prevent air movement through the open resonator, it was enclosed within a plastic bag. Atmospheric absorption losses [9] were taken into account in calculations (see Table I). To reduce influence of the atmospheric environment, one can use a resonator under vacuum or fill the cavity with dried air.

To minimize resonator length dependence on temperature changes, the resonator skeleton was made of INVAR alloy (thermal expansion coefficient about zero at room temperature).

Nevertheless, the central resonator frequency perceptibly drifted during a time interval of several minutes. To minimize frequency drift influence on the width measured, it is necessary to decrease the time of the resonance curve record. However, on the other hand, this leads to the accuracy deterioration of the resonance curve width determination [see Fig. 4(b)].

Let us quickly consider an acquisition algorithm of the resonance curve trace. A computer program makes a step-by-step frequency scan over a selected frequency range. The maximum record size was  $p = 560$  points at the frequency domain. Amplitude modulation of millimeter-wave radiation with frequency  $F_{\text{mod}} = 170$  Hz and selective amplifier of  $F = 170$  Hz and  $\Delta F \simeq 6$  Hz were used. A sampling rate  $F_{\text{sample}} = 85$  Hz of the DSD was chosen. A signal capture for each frequency step consists of ensemble averages of  $N$  accumulation events, which are signals integrated over sampling interval in time domain by the DSD. Thus, the total integrating time is  $\tau = N/F_{\text{sample}}$  and the total time record of the curve is  $T = p \cdot \tau$ . A curve width evaluation error depends on the number of frequency scan points,  $S/N$  ratio, and integration time  $\tau$ . Thus, the accuracy is determined by the time record of the curve, but the time record is limited by the frequency drifting. In our experiments, an optimum resonance curve record time was found to be about 10 s. In this case, the fitting error and standard deviation of the width over many measurements were about the same.

Finally, we have gotten the result:  $\pm 500$  Hz of the absolute accuracy of measurements of resonance curve width at 110-GHz frequency. This value corresponds to 15 curve records (total measurement time was 130 s).

### III. DISCUSSION

The limit sensitivity obtained in the experiment corresponds in accordance with (1) to the  $\tan \delta \leq 10^{-8}$ , minimum detectable absorption for the case of a completely filled dielectric (e.g. gas) cavity. For the interpretation of the result obtained in terms of measurable absorption in solid (or liquid) dielectric sample situated in the resonator, a real case must be considered.

We use the method when a plane parallel dielectric placed in the resonator perpendicular to its axis has “resonant” optical thickness, i.e., constituting exactly an integer number of half-wavelengths ( $\lambda/2$ ) at one of resonator eigenfrequencies

$$t \cdot n = m \cdot (\lambda/2) \quad (4)$$

where  $t$  is the dielectric plate thickness,  $n$  is the refractive index, and  $m$  is the integer number.

The advantage of this method is the full use of the resonator technique possibilities of absorption measurements and rather simple expressions (for  $\Delta = 1$  [10]) for calculation of loss tangent from experimental data [4], [11].

The simplified expression for  $\tan \delta$ -value [4] is

$$\tan \delta = (L/et)(1/Q_1 - 1/Q_0)$$

or

$$\tan \delta = (L/t)(1/Q_2 - 1/Q_0) \quad (5)$$

where  $\epsilon$  is the dielectric permittivity,  $Q_0$  is the quality factor of the empty resonator, and  $Q_1$  and  $Q_2$  are quality factors of the loaded resonator for the cases when sample surfaces coincide correspondingly with minima or maxima of the standing wave in the resonator.

Recalled the definition of quality factor  $Q = f/\Delta f$ , then

$$\tan \delta = (L/tf\epsilon)(\Delta f_1 - \Delta f_0)$$

or

$$\tan \delta = (L/tf)(\Delta f_2 - \Delta f_0)$$

where  $f$  is the frequency and  $\Delta f_0$ ,  $\Delta f_1$ , and  $\Delta f_2$  are the widths of resonant curves corresponding to  $Q_0$ ,  $Q_1$ , and  $Q_2$ .

At first sight, this case may look like a very specific one, but as a matter of fact, this technique can be applied to the measurements of the dielectric properties (refractive index  $n$  and  $\tan \delta$ ) of most plane parallel dielectrics including liquids. We propose to tune the sample to the "resonance" case not by changing its thickness, which is cumbersome and sometimes not possible, but by tuning the synthesizer frequency. The criterion of the "resonance" condition is the independence of the resonance frequency upon a position of dielectric along the resonator axis; a dielectric plate displacement in the limits of several half-wavelengths will not change the resonance frequency of a resonator. However, the displacement from the position where sample surfaces are in the minima of standing wave to the position where they are in its maxima results in the loss increasing in the sample by  $\epsilon$  times. Otherwise, this leads to decreasing the  $Q$  value of a resonator [4], [11].

Following (5), let us estimate loss tangent values, which we have a possibility to measure by our apparatus with  $\pm 10\%$  accuracy (what corresponds to  $\sim 7$ -kHz resonance curve width difference of loaded and empty resonator and  $\sim 0.7$ -kHz standard deviation of measuring values) for some practical cases as follows:

- 1) for sapphire or silicon plate (refractive indexes  $n$  are 3.07 and 3.42, respectively) of  $\sim 3$ -mm thickness (this is a real thickness for a gyrotron window chosen to withstand the atmospheric pressure at  $\sim 80$ -mm diameter):  $(\tan \delta)_{\min} (10\%) \simeq 6.9 \cdot 10^{-7}$  and  $(\tan \delta)_{\min} (10\%) \simeq 5.5 \cdot 10^{-7}$ , respectively;
- 2) for diamond plate ( $n = 2.38$ ) of the real thickness 0.57 mm:  $(\tan \delta)_{\min} (10\%) \simeq 6 \cdot 10^{-6}$ .

This accuracy should be more than sufficient for loss tangent measurements at the present state of low-loss materials development like sapphire ( $\tan \delta = 2 \cdot 10^{-4}$ ) [12] and Si ( $2.5 \cdot 10^{-6}$ ) [13]. At the same time, we have had great progress in diamond development in the last two years: losses in chemical vapor deposited (CVD) diamonds were reduced down to  $8 \cdot 10^{-6}$  [14]. Thus, the accuracy reached in this paper is well suited for measurements of diamonds at the present level, but without significant reserves.

#### IV. CONCLUSION

The level of sensitivity in the measurements of loss tangent achieved in this paper exceeds the earlier one obtained in [1] by more than two orders. On the one hand, that is due to constructing a high- $Q$  resonator, on the other hand, it was defined in a greater degree by using the powerful precise microwave synthesizer as a radiation source, together with the software developed for studies of the pressure line broadening, and it is found to be very effective. Thus, the goal of this paper stated in Section I and the new level of sensitivity of absorption measurements have both been achieved. It is worth noting that, at the present time, the technique used in this paper is extended well above 1 THz [3], [15], and the measurements of dielectric properties of ultra-low-loss materials are now available in whole millimeter- and submillimeter-wave bands.

#### ACKNOWLEDGMENT

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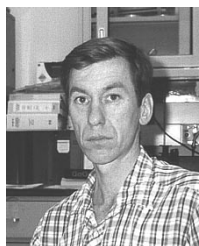
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