Modern Resonator Spectroscopy at Submillimeter Wavelengths

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***Abstract*— Classical resonator spectroscopy methods have been realized for the first time in the submillimeter wave range. A resonator spectrometer was developed earlier at the IAP RAS on the basis of an open Fabry–Perot resonator excited by the radiation of a backward wave oscillator whose frequency is stabilized by the phase lock loop system. This spectrometer is successfully used for high-precision measurements of the dielectric properties of solid, liquid, and gaseous dielectrics as well as for the metal and coating reflection measurements in the 36–370 GHz range. In this paper, we report an extension of the upper limit of the spectrometer operation frequency to 520 GHz. Features of operation of the main spectrometer systems in the extended frequency range are analyzed. The broadband measurements of absorption in modern MPCVD diamonds are presented. A continuous record of the absorption spectrum of the laboratory atmosphere in the 350–500 GHz range obtained for the first time by the high-sensitivity microwave method is demonstrated. Further prospects for extension of the spectrometer range to terahertz frequencies are discussed.**

***Index Terms*— Atmospheric spectra, chemical vapor deposition (CVD) diamond losses, high-accuracy measurements, resonator spectrometer, submillimeter waves.**

I. Introduction

THE RESONANCE methods of investigations are widely used in precision spectroscopy in all frequency ranges due to their high sensitivity. The resonance systems have different specific features in each range. In the millimeter (MM) and submillimeter (SubMM) ranges, classical open Fabry–Perot resonators with an empty-resonator Q-factor of about 10^6 and finesse $\sim 3 \cdot 10^3$ (for reasonable sizes) are used for the gaseous and condensed media investigations [1]–[6]. The principle of sample absorption measurement by a resonator spectrometer is the following. Measured values are the resonance curve widths of the loaded and empty resonator. The difference of these values is directly connected with the sample absorption value. The same idea is used in the reflectivity investigations. In this case, we also measure the resonator curve widths of an empty resonator and upon replacement of one of the resonator mirrors by the reflector studied.

In both cases, the measured values used for the sample parameter calculation are the resonance frequencies and resonance curve widths. It is well known that the frequency measurements are the most accurate ones. This fact in combination with a high Q-factor (i.e., long effective radiation path length inside the resonator) defines the high sensitivity and accuracy of the method.

The present work based on the previous development is to our knowledge the first advancement of high-sensitivity classical resonator spectroscopy to frequencies of up to 520 GHz.

The resonator spectrometer earlier developed at the IAP RAS, methods of measurement, and the results of studying various materials at frequencies ranging from 40 GHz to 370 GHz are described in detail in [2]–[4] and [7]–[21]. In Section 2, the main features of the spectrometer are briefly discussed.

In Sections 3 and 4, a detail description of the resonator excitation system and the frequency phase lock system, which were essentially modified for operation in the 350–520 GHz frequency range, is presented.

In Section 5, the first results obtained using the spectrometer in the extended frequency range are reported.

In the conclusions, the further prospects for extension of the spectrometer operation frequency range to the terahertz frequencies are analyzed.

II. Resonator Spectrometer

Backward wave oscillators (BWOs) are used in the spectrometer as the sources of coherent microwave radiation with continuous frequency tuning. The sources are equipped with a precise digital frequency stabilization and control system based on the phase lock loop (PLL) system with the microwave synthesizer harmonics used as the reference signal. It is worth mentioning that for broadband high-sensitivity MM-SubMM spectroscopy, BWOs remain the most convenient and reliable sources of radiation. A quasi-optical line forming the Gaussian beam is used for the resonator excitation in a wide frequency range. The resonator is connected with the radiation source and detector by a coupling film.

The resonance curve is recorded by a fast step-by-step frequency scanning system without loss of the oscillation phase during the frequency switching. The curve width is determined from a fit by model function based on the Lorentz profile. The averaging of resonance widths obtained from upward

Manuscript received May 28, 2012; revised July 18, 2012; accepted August 21, 2012. Date of publication September 4, 2012; date of current version December 6, 2012. This work was supported in part by the Russian Foundation for Basic Research and the Russian Federal Special Program "Scientific and Scientifically-Pedagogical Staff of Innovative Russia" in 2009–2013. The associate editor coordinating the review of this paper and approving it for publication was Dr. Francis P. Hindle.

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Digital Object Identifier 10.1109/JSEN.2012.2215315

and downward frequency scans is used to avoid distortions related to the resonator central frequency drift during the curve recording. Multiple records are used for the accuracy and sensitivity improvement. After averaging of about 1000 scans, the accuracy of measurement of the resonance curve width amounts to 20 Hz for the resonance width ~100 kHz. This accuracy corresponds to the minimum detectable variation of the gas absorption coefficient ~0.002 dB/km. For dielectrics, this corresponds to the absorption in a plate of 1 mm thickness with a loss tangent of about 10^{-7}.

III. Resonator Excitation System and the Resonance Response Recording

Fundamental TEM_{00q} modes of the Fabry–Perot resonator, where q is the longitudinal mode number, $q = 2L/\lambda$ (L is the resonator length and λ is the wavelength), are employed. A small-angle round smooth horn is used for transformation of the main generator waveguide mode TE_{01} to a quasi-optical Gaussian beam for the resonator excitation. All other modes produced by the BWO or generated in the supply waveguides transform to parasitic resonator modes such as TEM_{mnq}. These modes have resonance frequencies usually differing from the fundamental modes TEM_{00q} and only litter the resonator spectrum. The amplitudes and the number of these parasitic modes characterize the non-effectiveness of the waveguide mode to the quasi-optical beam transformation. They also characterize the quality of the resonator connection with the quasi-optical system. In fact, the radiation power spent for the parasitic mode excitation is wasteful loss of the BWO power. The quasi-optical line of the spectrometer makes it possible to excite the TEM_{mnq} modes at a level constituting less than 10% of the TEM_{00q} modes at all operation frequencies.

Let us consider characteristic features of the resonator excitation system in the spectrometer frequency sub-bands. The 180–260 GHz and 260–370 GHz sub-bands are characterized as intermediate. Starting from these frequencies, the BWO has an oversized output waveguide with 1.2×2.4 mm^2 cross section. However, high purity of the radiation mode (the BWO producer claims that more than 90% of the output power is generated in the TE_{01} mode) permits one to use after the BWO output flange a smooth waveguide junction to the waveguide with 0.55×1.1 mm^2 cross section, which is close to the main one at these frequencies. In these frequency ranges, standard waveguide elements can be used as in the lower-frequency sub-bands. The relatively high BWO output power (up to 0.1 W) permits using commercial point-contact detectors of the MM wave range [22], [23] for the resonator responses recording with a sufficient signal-to-noise ratio.

The main peculiar features of the frequency ranges above 370 GHz are, first of all, decreased (down to several milliwatts) BWO output power and reduced sensitivity of the point-contact detectors due to parasitic capacitances that rapidly increase in the diodes with increasing frequency. Because of the high oversizing of the output waveguide, the BWO emission becomes multimode and has polarization components on both waveguide sides. This effect additionally reduces the useful BWO output power. Due to high losses, the standard commercial waveguide elements of the main cross section are absent. Thus, for the frequency range 350–520 GHz covered by an OB-76 BWO, a quasi-optical line (see the photo in Fig. 1.) was developed. The only waveguide element close to the “monomode” one is mode filter (2), which is made as a smooth waveguide junction from the output waveguide with 1.2×2.4 mm^2 cross section to a 0.55×1.1 mm^2 waveguide. The waveguide is further transformed into horn (3) which forms a quasi-optical beam with a diameter of about 15 mm. Beam splitter (4) directs approximately 80% of the BWO power to the PLL system, which is described in the next section.

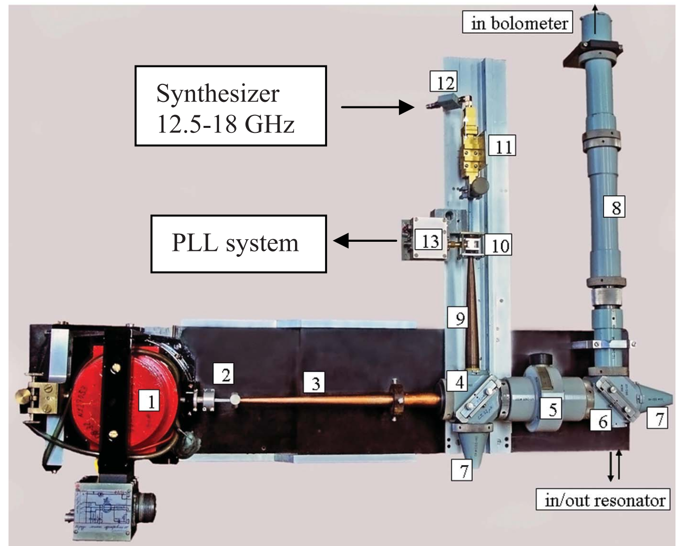

Fig. 1. Photo of the quasi-optical line. (1) BWO in the magnet. (2) Waveguide junction from output waveguide with 1.2×2.4 mm^2 cross section to the waveguide with 0.55×1.1 mm^2 cross section. (3) Horn. (4) Beam splitter for the PLL system. (5) Attenuator. (6) 3-dB divider. (7) Terminating loads. (8) Transmission line of resonator response to bolometer. (9) Horn. (10) Multiplier-mixer of the PLL system. (11) Multiplier-amplifier of the microwave synthesizer signal in the PLL system. (12) Isolator 8–18 GHz. (13) Preamplifier (HEMT) of intermediate frequency (IF) in the PPL system.

Beam splitter (4) is connected with variable attenuator (5), which is used for the optimization of the power coming to the detector. The next is diplexer (6), which is a 50% beam power divider. One half of the power is directed to the resonator, and another half is directed to terminating load (7). The radiation coming from the resonator passes through diplexer (6) and is directed through transmission line (8) to the detector.

We would like to note that components 4–8 have been assembled from the excellent SubMM waveguide constructing kit “Oliva” [24].

As was mentioned above, the sensitivity of the available point-contact detectors was found to be insufficient for the spectrometer operation at frequencies above 350 GHz. Thus, a liquid helium-cooled InSb bolometer produced by “QMC Instruments” [25] was employed. The bolometer provided a signal-to-noise ratio of the resonator response records more than two orders of magnitude higher in comparison with the point-contact detectors and permitted measurements using a radiation power attenuation of about 20 dB (element (5) in Fig.1).

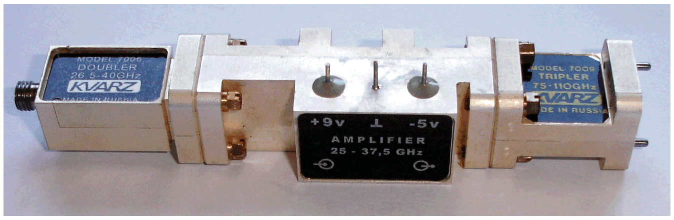

Fig. 2. Photo of the frequency multiplier used in the PLL system.

IV. Frequency Stabilization System

The general idea of frequency stabilization and operation is the same as that described in [12]. The PLL system of BWO generators uses the conventional frequency stabilization configuration using the harmonics of a centimeter-wave ($\sim$10 GHz) synthesizer ("Agilent" E8257D) as a reference signal. The reference signal is mixed with the BWO radiation in the multiplier-mixer [element (10)]. In the low-frequency sub- bands, the mixers [26] based on planar GaAs Shottky diodes are employed. The harmonic generation efficiency falls down with increasing harmonic number, and at frequencies above $\sim$400 GHz, at the 40$^{\text{th}}$ and higher harmonic numbers, the generated power becomes less than is necessary for stable operation of the PLL system.

The attempts to use the multiplier-mixer based on a GaAs/AlAs superlattice planar diode [27] also yield no results. Such a mixer really generates the harmonics with high numbers more effectively than the Shottky-based mixer. However, due to the negative differential conductivity range on the current-voltage characteristic of its nonlinear element, this mixer generates not only the reference-signal harmonics, but also its own wide-band signals saturating the IF amplifier in the PLL system. The superlattice diode-based mixer turned out to be very sensitive to the geometrical alignment and to the reference signal power, which were necessary to adjust at each resonator eigen mode frequency. All these peculiarities made it impossible to use the mixer because the spectrometer requires stable operation of the PLL system for many hours without any tuning.

The problem was resolved by employing an active frequency multiplier [28], [29] converting the microwave synthesizer frequency up to the 75–110 GHz frequency range (see the photo in Fig. 2). This device is the consecutive connection of a 13.25–20 GHz passive frequency doubler, a 25–37 GHz power amplifier, and a passive frequency tripler. The input connector is an SMA coaxial connector and the output is a waveguide with 1.25×2.5 mm^2 cross section. The multiplier output power varies smoothly between 2.2 and 4.5 mW at frequencies 75–110 GHz with an input power of about 100 mW.

The frequency multiplier was connected to the multiplier-mixer based on a GaAs planar Shottky diode. The multiplier-mixer construction is similar to that used for the phase stabilization of SubMM BWO radiation [30], [31]. Such a design made it possible to reduce the harmonic number to 4–5 in the multiplier-mixer and thus ensured stable operation of the PLL system without tuning in the whole 350–520 GHz frequency range.

One more feature of the spectrometer operation has to be pointed out. This feature appeared when a fast and high-sensitivity radiation detector was used. High-frequency BWOs operated above 200 GHz use AC line voltage for the cathode filament. This leads to the parasitic modulation of the BWO output power with the supplied power frequency. This modulation is very small, but it becomes noticeable if sensitivity of the receiver is high. In our spectrometer, this power modulation leads to a periodic shape distortion of the resonator response and, correspondingly, an increase in the absorption measurements error.

The DC voltage used for the cathode filament could eliminate this modulation, but this would drastically shorten the lifetime of the quite expensive BWOs. Using the DC filament with fast polarity commutation every few minutes could be another acceptable solution. However, the implementation of this idea entails essential complication of the BWO power supply and, correspondingly, reduces its reliability. Besides, such a method does not guarantee the preservation of the BWO lifetime indicated in the passport.

To solve the unwanted modulation problem, the resonance curve recording regime was changed by changing the fast frequency scanning parameters determined by a radio-frequency (RF) digital synthesizer. Since the minimum time between frequency switchings of our RF synthesizer is 60 μs and cannot be reduced, we decreased the number of frequency steps within one scan and, correspondingly, increased the step size maintaining the same scan width. In this way, the duration of each individual scan was reduced until it became much less than the modulation period. In this regime, the resonance curve distortions are insignificant and can be taken into account at the record processing stage. The number of points reduction at the resonance curve resulted in increased statistical error in the curve width measurement. To eliminate this problem, the number of scans was increased accordingly to preserve the 20-Hz measurement accuracy of the resonance curve width, even in the presence of the parasitic modulation.

V. Dielectrics Absorption Investigations

The results of the radiation absorption investigation for a CVD diamond output window designed for the gyrotron with megawatt power in continuous-wave operation are presented in Fig. 3. The frequency range extension confirmed the presence of the absorption scattering mechanism in CVD diamonds.

From the previous studies it follows that the main absorption mechanism in CVD diamonds at MM waves (at room temperature) is due to the extrinsic conductivity caused by defects, impurities, and non-diamond inclusions [32]. The loss tangent frequency dependence of this mechanism is reverse frequency dependence: tan $\delta \sim 1/f$ at up to several terahertz. However, a deviation from the 1/f law was observed at frequencies above $\sim$180–200 GHz [33].

It was assumed [33] that the additional losses are due to radiation scattering by structural inhomogeneities (namely, microcaverns between crystallites). This mechanism was expected to become the main absorption mechanism at the

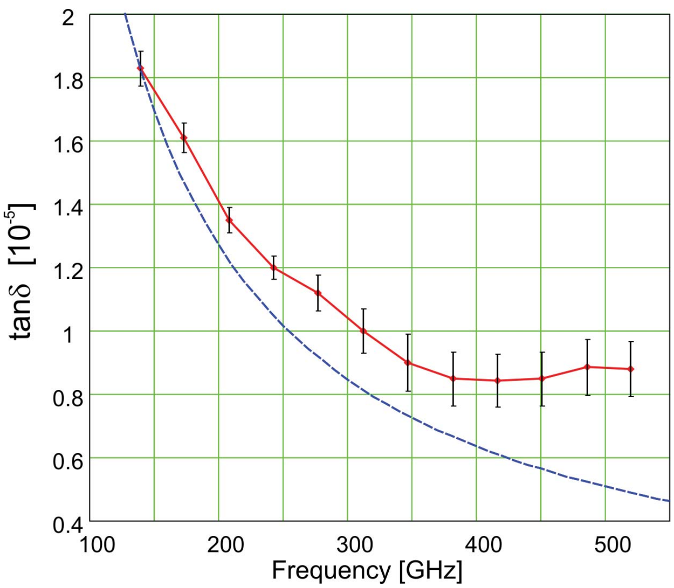

Fig. 3. Loss tangent frequency dependence of the CVD diamond window. Thickness = 1.8 mm. Refractive index = 2.380. Upper curve: loss tangent frequency dependence of the CVD diamond window. Lower curve: approximation∼1/f based on the low-frequency point.

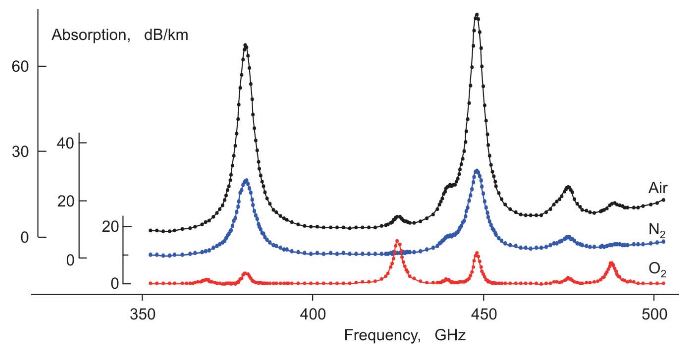

Fig. 4. Absorption spectra of air and main atmospheric gases mixed with water vapor at atmospheric pressure and room temperature. Measured values of gas's relative humidity constitute 6.6% for air, 1.9% for N_2, and 0% for O_2 (see the text).

terahertz frequencies. Our previous study confirmed that the diamond sample with a more perfect inner structure (having the minimum number of relatively large-size microcaverns) has an absorption closer to the theoretical curve in a range of up to 350 GHz. However, the studies of this sample in the 350–520 GHz range [34] clearly revealed the influence of the same mechanism (Fig. 3). Some "irregularity" of the loss tangent frequency dependence can be explained by the different number of large-size microcaverns in the resonator beam spot.

The resonator beam diameter changes with frequency. Its variation within the frequency range shown in Fig. 3 is about a factor of two. The total number of relatively large-size microcaverns, which mainly define the scattering, is not really large in a high-quality sample, so that the number of caverns within the spot changes irregularly. Thus, different amount of caverns participates in the radiation scattering at different frequencies.

Another example of the resonator spectrometer capabilities in the new frequency range is the broadband continuous records of spectra of the real atmosphere and its main constituents, namely, nitrogen and oxygen mixed with water vapour at room temperature. The spectra are presented in Fig. 4. It should be pointed out that these are the first atmospheric pressure spectra recorded in this range by accurate microwave techniques.

The measurement method is described in detail in [11]. The records clearly reveal the pressure-broadened absorption lines corresponding to the pure rotational transitions of molecular oxygen (O_2) at 368, 425, and 487 GHz and water molecule at 380, 439, 471, 474, and 488 GHz [35]. The points on the diagram correspond to the resonator eigen mode frequencies at which the absorption was measured. Gas humidity and temperature in the course of the experiment were monitored by a "TESTO-645" sensor [36] equipped with a high-precision humidity/temperature probe. Guaranteed accuracy of the relative humidity measurement is ±2% under experimental conditions. Despite the fact that during the "oxygen" spectrum recording the humidity meter indicated zero humidity, the water lines are clearly seen in the record. The relative humidity of the O_2 sample defined through the water line integrated intensity as described in [11] constituted 0.879(7)% during the spectrum recording. On the one hand, this value confirms the humidity sensor passport's accuracy. On the other hand, it demonstrates the possibility of developing a special humidity sensor on the basis of the resonator spectrometer with sensitivity exceeding the existing commercial products by several orders of magnitude. An additional advantage of such a humidity measurement method is volumetric measurements. All water molecules located in the volume between resonator mirrors contribute to the measured humidity, which is essentially different from the measurements at the point of location of the conventional sensor.

The obtained spectra are valuable for developing and testing of accurate radiation propagation models in the atmosphere applicable first of all for remote sensing. The detailed spectroscopic analysis of recorded spectra will be published elsewhere [37].

VI. Prospects for the Further Advancement Into the Terahertz Range

A systematic decrease of the BWO output power with increasing frequency in combination with a considerably increasing number of possible waveguide modes at the tube output leads not only to the reduction of useful power in the spectrometer, but also to strong amplitude-frequency dependences in the spectrometer waveguides. These drawbacks are supplemented by a decrease in the sensitivity of the multiplier-mixers and reduction of their efficiency of the beat-note generation for high harmonic numbers of the reference signal. All this will impede the continuous PLL system operation without tuning which is required for normal operation of the spectrometer. Nevertheless, the continuous records of broadband molecular spectra in the range near 1 THz performed with different kinds of a BWO-based spectrometer (e.g., [38] and references therein), which also employs a similar PLL system, have confirmed the fundamental possibility of the resonator spectrometer operation at up to about 1 THz.

Acknowledgment

The authors would like to thank A. M. Schitov and his colleagues for manufacturing the frequency multiplier for the phase lock loop system.

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