ULTRALOW ABSORPTION IN SILICON CARBIDE IN THE MILLIMETER-WAVE RANGE

E. E. Chigryai, B. M. Garin, R. N. Denisyuk, D. S. Kalenov, I. P. Nikitin
Kotel'nikov Institute of Radio-engineering and Electronics of Russian Academy of Sciences

The paper is received on October 19, 2016

Abstract. Dielectric properties of high-purity semi-insulating single-crystal 6H silicon carbide are investigated in the millimeter-wave (MM) range. A method and a setup are developed for measuring ultralow dielectric loss in the long-wavelength part of the millimeter-wave band at frequencies below 80 GHz in samples with relatively small transverse size (~20 mm) and arbitrary thickness (~1 mm or less). The method and the setup are based on a semisymmetric open resonator with spherical mirror with radius of curvature of 40 mm. The value of loss tangent measured at 69.4 GHz and room temperature is $\tan\delta \sim 6\cdot 10^{-5}$, which is the lowest value ever observed in this material. This value is significantly lower than that observed in well-known low-loss materials such as single-crystal sapphire and quartz but exceeds the loss value in the best samples of CVD diamond. However, SiC has lower production cost compared to the latter. The loss mechanisms in silicon carbide are revealed. At low frequencies <20 GHz, the loss is due to absorption by free charge carriers. In the region 60–400 GHz, the loss can be attributed to the intrinsic lattice loss due to two-phonon absorption processes.

Key words: silicon carbide, dielectric loss, open resonator**.**

1. Introduction

The problem of low-loss materials in the millimeter-wave range of electromagnetic waves is one of key problems in radio engineering and electronics. In particular, this applies to high-power electronics, including the application in high-power gyrotron windows (including megawatt continuous-wave gyrotrons used in the ITER project) [1], high-Q resonators, etc.

In many low-loss materials, the lower theoretical limit for the loss tangent $\tan\delta$ is determined by the intrinsic lattice loss (ILL) due to the two-phonon absorption in the corresponding ideal crystal [2, 3]. It follows from the theory that the low values of $\tan\delta$ can be expected in crystals with low values of lattice anharmonicity, high sound velocity, high symmetry of the crystal structure, and high lattice heat conductivity. On the basis of these criteria, it was predicted that the lowest ILL can be obtained in crystals with the diamond structure, including diamond and silicon [4, 5]. These materials are characterized by lowest loss tangent, down to $\tan\delta \sim 2{\cdot}10^{-6}$ in the millimeter-wave band at room temperature [4, 5]. These characteristics made it possible the design of megawatt continuous-wave gyrotrons for the ITER project. The windows in these gyrotrons are made of polycrystalline diamonds grown with the use of chemical vapor deposition (CVD) technology.

Silicon carbide crystals are related to diamond. In these crystals, the unit cell contains carbon and silicon atoms from the same group IV, in contrast to diamond, where the unit cell contains two identical carbon atoms. This crystal can exist in different polytypes, which may contain different crystal structures, including cubic and hexagonal structures. Silicon carbide is one of the most promising materials for the design of hardware components for various microwave devices operating at higher temperatures and radiation levels compared with the existing analogs, including millimeter-wave and terahertz-band devices; it can find application in microwave technology, including heterostructures in high-power diodes, transistors, thyratrons, etc., as well as in high-power electronics. Silicon carbide is quite promising for application as output windows in high-power microwave oscillators, including gyrotrons with sub-megawatt output power levels, since the production of this material is much cheaper compared with CVD diamonds.

In the early 2000s, the CREE (USA) launched a manufacturing technology for large single-crystal samples of high-purity semi-insulating silicon carbide [8]. The production of this material is much cheaper compared with the production of CVD diamonds.

Earlier [9–11], the authors investigated millimeter-wave loss in 4H silicon carbide with alternating layers of hexagonal and cubic lattices.

In recent years, facilities have been developed in Russia that allow one to grow large single-crystal samples of high-purity semi-insulating 6H silicon carbide in which a layer of hexagonal lattice alternates with two layers of cubic lattice [12,13].

In [13], the authors for the first time measured loss in 6H SiC grown at Svetlana-Elektronpribor (St. Petersburg) at frequencies of 6.4 GHz and 120–360 GHz at room temperature.

At frequency of 6.4 GHz, a loss of $\tan\delta \sim 1.8 \cdot 10^{-4}$ is observed, while, at frequencies of 120–360 GHz, the loss tangent decreases with decreasing frequency from $2.3 \cdot 10^{-4}$ to $1.1 \cdot 10^{-4}$. These data imply that the lowest loss can be expected at frequencies <100 GHz.

In this work, we develop a method for measuring low dielectric loss at frequencies <100 GHz. We measure loss in 6H silicon carbide at frequency of 69.4 GHz, as well as the refractive index in the millimeter-wave band, and compare the results obtained with theory and discuss the loss mechanisms.

2. Method for measuring dielectric loss and refractive index

To measure the dielectric loss and the refractive index of low-loss plates, one applies resonance methods implemented in both waveguide [14] and free-space [15] configurations. In waveguide resonators, the dimensions of resonators themselves, samples, coupling apertures, and cavities for placing samples become critically small even at frequencies of about 30 GHz. Therefore, at frequencies above 30 GHz, one usually applies quasioptical open resonators with spherical mirrors for correcting the wave field.

2.1. Method for measuring a refractive index

The refractive index n of dielectric plates can be determined from the resonance frequencies of the sample. For an integer number of half-wavelengths across the sample, the phases of the waves reflected from the front and back faces of the sample are opposite. In this case, the frequency dependence of the reflection coefficient exhibits a series of minima. The reflection coefficient of the sample is given by

$$r = \frac{\frac{1-n}{1+n}\left(1-e^{2iknh}\right)}{1-\left(\frac{1-n}{1+n}\right)^2 \cdot e^{2iknh}} \tag{1}$$

Here $k = 2\pi f/c$ is the wavenumber, f is frequency, h is the plate thickness, and c is the velocity of light. At a reflection minimum, $e^{2iknh} = 1$, or $2knh = 2m\pi$. Therefore,

$$n = \frac{c \cdot m}{2h \cdot f}. \tag{2}$$

Here $f = f_m$ is the value of frequency at the reflection minimum, and m is an integer. If m cannot be determined from some prerequisites, then one can determine m from the values of frequencies at two neighboring reflection minima f_{m1} and f_{m2}.

$$m = \frac{f_{m1}}{f_{m2} - f_{m1}} \tag{3}$$

Measurement setup

To measure the resonance frequency of a sample, we used a reflectometer based on an oversized waveguide [16]. The field formed at the cross section of such a waveguide has a structure close to that of a plane wave, and the reflection from the open end is about –50 дБ. This fact allows one to carry out measurements with a sample placed at the cross section of the waveguide. The measurements were carried out on a network analyzer operating in the frequency band 118–178 GHz. The block diagram of the measurement setup is shown in Fig. 1.

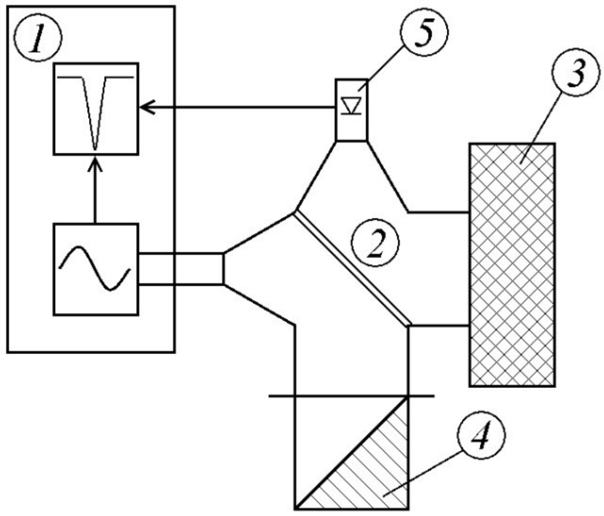

Fig. 1. Block diagram of the measurement setup: (*1*) network analyzer, (*2*) reflectometer, (*3*) sample, (*4*) matched load, and (*5*) detector.

2.2. Method for measuring dielectric loss

The classical method of determining the tanδ of thin dielectric plates consists in measuring the Q factors of a resonator with (Q_1) and without (Q_0) a sample, respectively. These measurements allow one to determine the additional loss α_1 in the resonator caused by the introduction of a plate into the resonator. The compensation for the variation of the resonator length by moving the mirror allows one to determine α_1 in terms of the measured quantities [17].

$$\alpha_1 = \left(\frac{Q_0}{Q_1} - 1\right) \cdot \frac{Lk}{Q_0} \tag{4}$$

Here L is the resonator length, $k = 2\pi f/c$, f is the resonance frequency, and c is the velocity of light. To calculate tanδ, we represent the additional loss α_1 as the difference between the loss due to reflection from a plane metal mirror α_2 and from the boundary of the plate located near the mirror, α_0, i.e.,

$$\alpha_1 = \alpha_0 - \alpha_2 \tag{5}$$

The energy loss due to reflection has a simple meaning:

$$\alpha_0 = 1 - |r_0|^2, \qquad \alpha_2 = 1 - r_2^2 \tag{6}$$

The reflection coefficient r_0 is

$$r_0 = \frac{\dfrac{1-n^*}{1+n^*} - r_2 \cdot e^{2ikhn^*}}{1 - \left(\dfrac{1-n^*}{1+n^*}\right) \cdot r_2 \cdot e^{2ikhn^*}} \qquad (7)$$

Here $n^* = n(1 + i\tan\delta/2)$ is the complex refractive index of the plate, h is the plate thickness, and $\tan\delta = \varepsilon''/\varepsilon'$, where ε'' and ε' are the imaginary and real parts of the complex permittivity ε^*.

According to [18], the reflection coefficient r_2 is:

$$r_2 = \sqrt{1 - 2\left(\frac{f}{\sigma}\right)^{1/2}}. \qquad (8)$$

Here σ is the conductivity of metal. Table 1 presents the values of conductivity [19] and energy loss α_2 due to reflection from metal at frequency of 69.4 GHz.

Table 1.

	Metal	Conductivity, S/m	α_2
1.	Silver	$6.25 \cdot 10^7$	$6.6645 \cdot 10^{-4}$
2.	Copper	$5.88 \cdot 10^7$	$6.871 \cdot 10^{-4}$
3.	Aluminum	$3.85 \cdot 10^7$	$8.4914 \cdot 10^{-4}$

In [20], the authors presented measured values of loss due to reflection from metals. For example, for a copper plate processed by a diamond cutter with a copper content of 0.998, the loss due to reflection was measured to be $7.8 \cdot 10^{-4}$ at frequency of 79 GHz. This difference is related to the fact that calculations are made with the use of the static conductivity.

Dielectric loss $\tan\delta$ in the plate is determined by the solution of the equation

$$\alpha_0(\tan\delta) - \alpha_2 - \alpha_1 = 0 \qquad (9)$$

Here $\alpha_0(\tan\delta)$ is a function of $\tan\delta$ (current value of α_0, equal to $1 - |r_0(\tan\delta)|^2$, where $r_0(\tan\delta)$ is a current value of r_0, α_1 is defined in (4), and α_2 is a tabular value for a given metal.

Resonator

The method described was implemented on an open semisymmetric confocal resonator with sufficiently high Q, which is illustrated in Fig. 2.

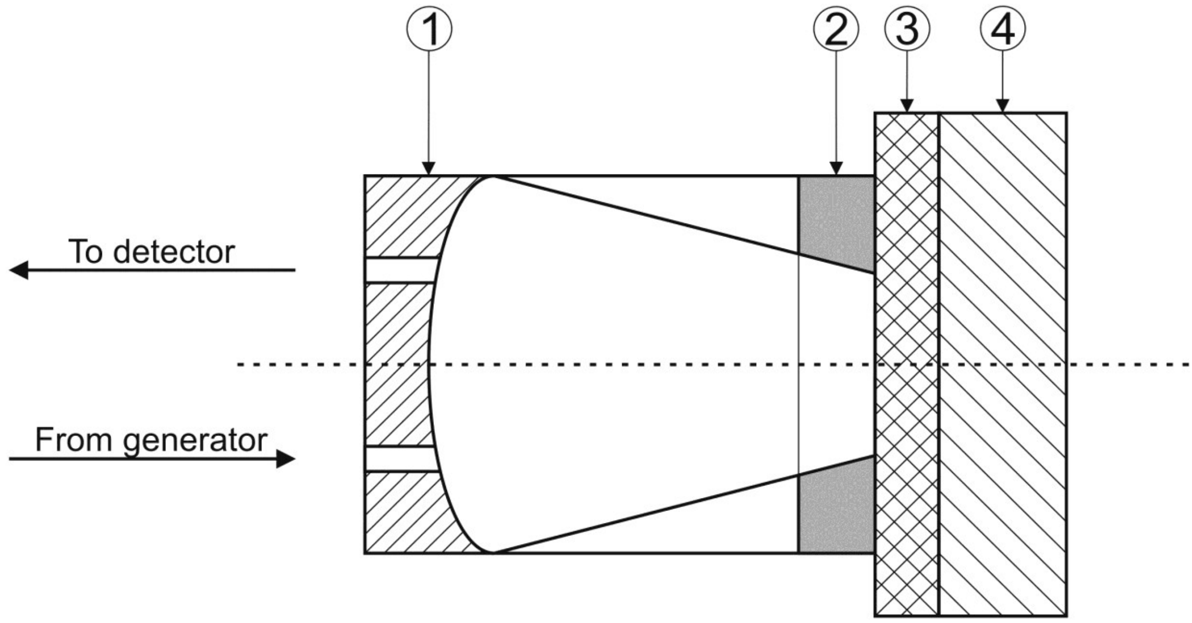

Fig. 2. Open resonator. (*1*) spherical mirror, (*2*) absorbing diaphragm, (*3*) sample, and (*4*) flat mirror.

The resonator consists of a spherical mirror with two coupling holes and a plane mirror. The plane mirror is situated at the end of an annular getinaks absorber, which serves for suppressing spurious oscillations in the resonator. The distance between the mirrors can be varied by a differential screw feed mechanism. Thus, one side of the resonator is free and is accessible for placing samples of various shapes and sizes. The coupling of the source and detector to the resonator is performed by circular holes with diameter of 1.25 mm and 20-mm-long circular waveguide sections with diameter of 3.2 mm. The holes and the waveguides are made in bulk metal of the spherical mirror. Microwave radiation is fed into the resonator through a waveguide of standard cross-section of 3.6×1.8 mm^2. The rectangular waveguide is well matched to the circular waveguide with diameter of 3.2 mm (SWR is not greater than 1.4).

The diameter of the spherical mirror is 40 mm, the radius of curvature is 40 mm, and the aperture diameter in the absorbing diaphragm is 25 mm. The distance between the mirrors is varied from 17 to 25 mm. The Q factor of the resonator with copper mirrors is 7000.

The setup allows the measurement of plane-parallel plates of arbitrary shape with transverse dimensions greater than the aperture diameter. The thickness of the samples should be about 1 mm or less.

3. Results and discussion

The measurements are carried out on a 6H-SiC sample with a thickness of 384 μm. The resonance frequency was measured to be 126.64 GHz. Hence, according to formula (2), the refractive index at $m = 1$ is $n = 3.0845$. The total error comes from the measurement errors of the sample thickness and the minimum frequency and does not exceed a few fractions of a percent (≈ 0.2–0.4 %).

Dielectric loss was measured in the above-described open resonator. A sample was placed on the output diaphragm of the resonator, and a copper mirror was placed on the sample. The resonator was fed by a Gunn oscillator with frequency of 69.4 GHz, and a Schottky diode was used as a detector. When a sample was placed into a resonator, we changed the resonator length to restore the resonance at the operating frequency. The ratio of Q factors of the resonator with and without a sample was 1.2. The resonator length at the resonance was 4.3228 cm. According to (4), $\alpha_1 = 1.795 \cdot 10^{-3}$. Table 1 shows that loss due to reflection from a copper mirror is $\alpha_2 = 6.9 \cdot 10^{-4}$. In this case, then value of $\tan\delta$ in equation (9) is $7.5 \cdot 10^{-5}$. If we assume that the loss due to reflection from a copper mirror is $7.8 \cdot 10^{-4}$, then $\tan\delta$ of silicon carbide at frequency of 69.4 GHz is $6.0 \cdot 10^{-5}$.

This value is less compared with the values obtained earlier in the 6H-SiC at frequencies of 6.4 and 120–360 GHz ($\tan\delta > 10^{-4}$) [13] and is the minimum of all the values observed in silicon carbide.

The results of measurements in the entire frequency range, including the earlier obtained results at $f = 6.4$ and 120–360 GHz in [13], as well as at $f = 69.4$ GHz in the present study, can be attributed to the following loss mechanisms. In the low-frequency range, the dominant loss mechanism is absorption by free charge carriers, which corresponds to the frequency dependence $\tan\delta \sim 1/f$ [6, 7]. In the range of 69–360 GHz, the loss increases with frequency, which can be attributed to the intrinsic lattice loss in this high-purity single-crystal sample, which is associated with two-phonon absorption processes [2–5].

4. Conclusions

We have developed, for the first time, a method and a setup for measuring ultralow dielectric loss in the long-wavelength part of the millimeter-wave band (at frequencies below 80 GHz) in plates with relatively small cross-sectional sizes (down to 20 mm). The method and the setup are based on an open semisymmetric resonator with spherical mirror with small radius of curvature of 40 mm.

The minimum value of dielectric loss was observed in silicon carbide in the millimeter-wave range at room temperature ($\tan\delta \sim 6{\cdot}10^{-5}$), which is the lowest value ever observed in this material in the millimeter-wave band. This value is an order of magnitude less than those observed in well-known low loss materials such as single-crystal sapphire and quartz but are higher than losses in the best samples of CVD diamond. Note, however, that the production of SiC is much cheaper compared with the latter one.

We have revealed the loss mechanisms n silicon carbide at the millimeter-wave range.

Acknowledgments

We are grateful to N.K. Travin (Svetlana-Elektronpribor, St. Petersburg) for supplying a sample of high-purity semi-insulating single crystal 6H-SiC.

This work was supported in part by the Russian Foundation for Basic Research, project No. 16-52-53140.

References

1. Thumm M., *Plasma Physics and Controlled Fusion*, vol. 45, pp. A143–A161, 2003.

2. Galdetskii A.V. and Garin B.M., Multiphonon absorption of IRE radiation in the crystals, caused by optical anharmonicity*, Preprint of Inst. Radio Eng. Electron., USSR Acad. Sci.*, No. 17(320), Moscow, 1981.

3. Garin B.M., Proc. 19th Int. Conf. on IR and MM Waves, Colchester, England, pp. 509–510, Sept. 1993.

4. Garin B.M., et al, Proc. 1th Ukrainian Symp. "Physics and Techniques of Millimeter and Submillimeter Radio Waves", Part 1. Kharkov, Ukraine, p. 86, 1991.

5. Garin B.M., Proc. Third Int. Conf. on Millimeter-Wave and Far-Infrared Science and Technol. Guangzhou, China, Aug.22–26, 1994, pp. 275–277.

6. Garin B.M., Parkhomenko M.P., Parshin V.V., Myasnikova S.E., Heidinger R., Danilov I., Molla J., Ralchenko V.G., Derkach V.N., and Tarapov S.I., Dielectric losses in CVD diamonds at frequencies 1kHz–200GHz and temperatures 70–800 K, Proc. Seventh Applied Diamond Conf./Third Frontier Carbon Technology Joint Conf., Tsukuba, Japan, Editors M. Murakawa, M. Miyoshi, Y. Koga, L. Schafer, and Y. Tzeng, USA, NASA, Vol. NASA/CP–2003-212319, Aug. 2003.

7. Parshin V., Garin B., Myasnikova S., and Orlenekov A., “Dielectric losses in CVD diamonds in the millimeter-wave range at temperatures 300–900 K”, *Radiophysics and Quantum Electronics*, 2004, Vol.47, No.12, pp.974-981.

8. Yu G., Dutta J.M., and Jones C.R., 2004, Potentials of SiC as a gyrotron window material, Conf. Digest 29th Int. Conf. on Infrared and Millimeter Waves and 12th Int. Conf. on Terahertz Electronics, Karlsruhe, Germany, 299-300.

9. Dutta J.M. and Jones C.R., Precision Measurement of Low Loss Window Materials, Proc. Mater. Res. Soc. Symp., MRS Vol. 833, Editors Yong S. Cho, D. Shiffler, C. A. Randall, H. A.C. Tillman, and T. Tsurumi, pp. 63–71, 2005.

10. Dutta J.M., Yu G., Tang Y., Jones C.R., Parshin V.V., Garin B.M., and Polyakov V.I., "The mechanisms of absorption in high-gap semiconductors at millimeter and submillimeter wavelengths", Digest of the Joint 32nd Int. Conf. on Infrared and Millimetre Waves and 15th Int. Conf. on Terahertz Electronics (IRMMW-THz 2007), Cardiff, UK, Sept. 2–7, 2007, paper P. 875.

11. Garin B.M., Lower Loss Limits at Millimeter and Terahertz Ranges, Proc. Joint 29th Int. Conf. on IR & MM Waves and 12th Int. Conf. on THz Electronics, Karlsruhe, Germany, pp. 393–394, Sept.27–Oct.1, 2004.

12. V'yuginov V.N., Travin N.K., Venediktov O.V., Korovkina M.M., Tsatsul'nikov A.F., and Lundin V.V., Development of the base production technology for substrates of semi-insulating silicon carbide, *Sbornik trudov chetvertoi vserossiiskoi konferentsii "Elektronika i mikroelektronika SVCh"* [Collection of papers of the Fourth All-Russian Conf. "Microwave Electronics and Microelectronics"], St. Petersburg: LETI, 2015, pp. 79--81.

13. V'yuginov V.N., Volkov V.V., Rebrov A.N., Travin N.K., Venediktov O.V., Korovkina M.M., Grigor'ev A.D., Kozyreva O.A., Parshin V.V., Serov E.A., Garin B.M., and Serov N.V., Prospects of application of home-made substrates of semi-insulating SiC in the microwave band up to 450 GHz, *Tr. II Rossiisko-Belorusskoi konferentsii "Elementnaya baza otechestvennoi radioelektroniki":importozameshchenie i primeneni* [Proc. II Russian--Belarussian Scientific and Technical Conf. "Hardware Components of Domestic Radio Electronics": Import Substitution and Application], Nizhni Novgorod: Nizhni Novgorod Gos. Univ., 2015, pp. 30–35.

14. Parkhomenko M.P., Kalenov D.S., and Abakumov Yu.F., Resonator method for determining dielectric and magnetic parameters of *materials* and experimental setup in the millimeter-wave band based on this method. *Elektron. Tekh., Ser. 1: SVCh tekh.,* 2013, No. 2 (517) pp. 43--57.

15. Vlasov S.N., Parshin V.V., and Serov E.A., Methods for investigating thin dielectric films in the millimeter range. *Technical Physics*, 2010, Vol. 55, No. 12, pp. 1781–1787.

16. Yu.N. Kazantzev Yu.N, and Kharlashkin O.A., Wide rectangular low-loss waveguides, *Radiotekh. Elektron.,* 1971, Vol. 16, No. 6, pp. 1063−1065.

17. Chigryai E.E., Khokhlov G.I., and Nikitin G.I., Quasioptical beam splitters on thin dielectric films in the millimeter and submillimeter wave ranges. *Zhurnal Radioelektroniki - Journal of Radio Electronics,* 2016, No. 6. Available at http://jre.cplire.ru/jre/jun16/9/text.html.

18. Born M. and Wolf E., *Principles of Optics*, London: Pergamon Press, 1970, 4th ed.

19. Ginkin G.G., *Spravochnik po radiotekhike* [Handbook of Radio Engineering], Moscow–Leningrad: Gosenergoizdat, 1948.

20. Parshin V.V. and Serov E.A., Precise resonator methods for investigating dielectrics and metals in the 40–500 GHz frequency range and temperature interval of 4–900 K, *Sbornik trudov chetvertoi vserossiiskoi konferentsii "Elektronika i mikroelektronika SVCh"* [Collected papers of the Fourth All-Russian Conf. "Microwave Electronics and Microelectronics"], St. Petersburg, LETI, 2015, pp. 34--39.