Irradiated Silicon for Microwave and Millimeter Wave Applications

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***Abstract*— Complex permittivity measurements of irradiated high-resistivity float-zone silicon have been performed in this letter from microwave to millimeter-wave frequencies employing three different resonance techniques. It has been proven that the irradiated silicon exhibits resistivity of the intrinsic silicon at temperatures larger than 295 K and the loss tangent due to phonon absorption reaches about 10^{-5} at room temperature. The total loss tangent of the room-temperature irradiated silicon is smaller than 6×10^{-5} at frequencies larger than 5 GHz. The real part of the complex permittivity of silicon linearly increases with temperature for $T > 200$ K.**

***Index Terms*— Microwave measurement, millimeter wave measurement, permittivity, silicon.**

I. Introduction

HIGH-RESISTIVITY float-zone (HRFZ) silicon is the material with one of the lowest electromagnetic (EM) losses at frequencies spanning from microwaves up to 10 THz [1]–[6]. It is commonly applicable in ionizing radiation detectors, thermography, automobile night vision systems, homeland security, and border patrol systems. There are two major absorption mechanisms in silicon, namely, crystal lattice oscillations related to phonon absorption and the absorption of free charge carriers. The former one is usually denoted with a loss tangent term, which is equal to $\tan\delta_e \approx 1.2 \times 10^{-5}$ [1], [7]–[12], whereas the latter one is usually represented with resistivity, ρ, leading to the following notation of the complex relative permittivity:

$$\varepsilon_r = \varepsilon'_r\left(1 - j\tan\delta_e - j\frac{\rho^{-1}}{\omega\varepsilon_0\varepsilon'_r}\right) \tag{1}$$

where ω is angular frequency, ε_0 stands for the vacuum permittivity, and ε'_r is the real part of the relative permittivity.

At frequencies much larger than 100 GHz, the loss tangent of silicon is mainly imposed by phonon absorption, $\tan\delta_e$, hence, resistivity is not that crucial. However, as the frequency decreases down to the microwave spectrum, the absorption of free charge carriers starts to play a dominant role in the total losses, as in (1). Therefore, to increase the performance of silicon at microwave frequencies, its resistivity should be as large as possible. Practically, it should be larger than $\rho = 10$ kΩcm, thus, enabling such applications of the HRFZ silicon as an advanced window material for high-power gyrotrons [13].

There are at least two methods of increasing the resistivity of silicon. The first method is doping the float-zone (FZ) silicon with gold, which acts as a deep-level donor/acceptor center. Its presence in both n- and p-type low-doped silicon crystals contributes to a significant increase in resistivity. Early work on this topic is presented in [13], whereas extensive studies of gold-doped silicon crystals have been described in [14]. Krupka *et al.* [2] have shown that gold doping of silicon increases resistivity 50 times from 2 to 100 kΩcm. Another method to increase the resistivity of the FZ silicon is irradiation with high-energy particles (e.g., deuterons, neutrons, protons, or electrons). It has been shown in [2] that the irradiation of the FZ silicon with high-energy deuterons (4.4 GeV), by employing the high-resolution photo-induced transient spectroscopy, results in the formation of deep-level defects with activation energies from 360 to 530 meV. Consequently, resistivity increases over 150 times up to the level of the intrinsic silicon (i.e., $\rho = 338$ kΩcm at 300 K), which is due to the compensation of shallow impurity donors with deep acceptors related to these defects. Irradiated silicon samples obtained at a laboratory scale have been already studied at a few microwave frequencies [1]–[3]. Currently, large irradiated HRFZ silicon wafers are commercially available.

According to [4], characterization of high-resistivity (HR) silicon at frequencies up to 20 GHz can be undertaken with a standardized method based on a split-post dielectric resonator (SPDR), which consists of two dielectric resonators usually

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made of a low-loss ceramics located in a cylindrical cavity, which allows exciting a $TE_{01\delta}$ mode with a circumferential electric field. Consequently, the insertion of a dielectric sheet in between the dielectric resonators modifies both the resonance frequency and the corresponding Q-factor, thus, enabling the extraction of the complex permittivity of a material under test (MUT). The extraction is undertaken by comparing raw measurement results with the EM model of the resonator. However, ultralow material losses in conjunction with relatively low energy filling factors and moderate conduction losses of the cavity prevent the use of SPDRs in the accurate characterization of the irradiated HRFZ silicon at larger frequencies.

A remedy for poor sensitivity in the measurement of ultralow-loss samples can be whispering gallery (WG) modes, which look similar to dielectric resonators exploited in SPDRs. However, the main difference is that the MUT shaped into a cylinder acts as a resonator itself. According to [15], WG modes are the modes with a large azimuth order, which results in a strong concentration of EM energy inside the MUT (as large as 90%), mainly at the circumference of the sample. Consequently, a quality factor of the measured resonance transmission curve is imposed mainly by the loss tangent of the MUT. In the case of low-loss materials with $\tan\delta < 10^{-4}$, the corresponding Q-factor can be well over $Q = 10^4$. Krupka *et al.* [16] indicate that, similarly to SPDRs, complex permittivity can be determined by comparing measurement results with the EM model of the resonator developed with the aid of the mode-matching technique.

Both of the aforementioned methods operate at a discrete frequency or in the case of WG modes at a few frequencies at most. Consequently, the attempt to fit measurement results with (1), in order to distinguish dielectric and conductive losses, is loaded with relatively large uncertainty. According to [6], it can be alleviated by using a Fabry–Perot open resonator (FPOR), which operates at Gaussian $TEM_{0,0,q}$ modes, where q is a longitudinal mode order. The MUT in the form of a dielectric sheet has to be located at the maximum of the electric field present in the beam waist of a given mode. The use of the FPOR at consecutive $TEM_{0,0,q}$ modes allows performing resonant measurements in a very broad frequency bandwidth. Karpisz *et al.* [17] applied a double-concave FPOR to the characterization of various dielectric and semiconductor materials in the 20–110 GHz frequency range. Due to the large volume of the FPOR, its Q-factor can be as large as 3×10^5, which makes it very sensitive to ultralow-loss materials. However, the main limiting factor in the measurement of materials, like the HRFZ silicon, is the low-energy filling factor. For that reason, a plano-concave FPOR, which is half of the double-concave FPOR presented in [17], has been applied in this letter [18]. The whole characterization has been fully automated, so that the measurement time at 20 frequency points in a 20–50 GHz range was about 10 min.

The use of the aforementioned three resonant methods opens up the way for the characterization of the HRFZ silicon in the 1–50 GHz range and beyond, thus, enabling well-defined estimation of both resistivity, ρ, and the loss tangent due to phonon absorption, $\tan\delta_e$. In addition, the setup with WG modes is applied in this letter to determine the temperature dependence of the permittivity and resistivity of the HRFZ silicon.

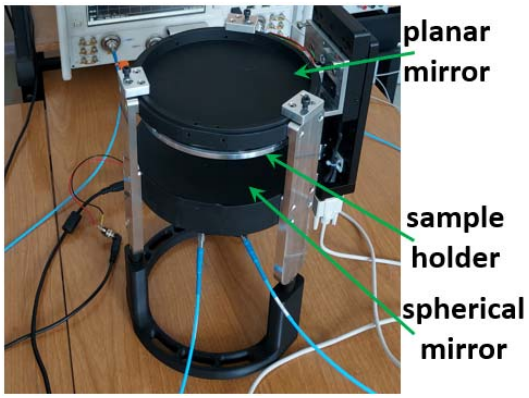

Fig. 1. Plano-concave FPOR.

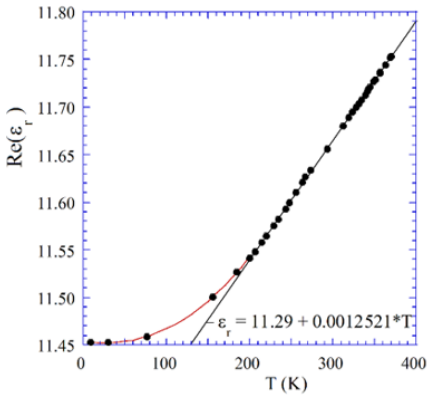

Fig. 2. Real part of permittivity of bulk-irradiated HRFZ silicon sample ($d = 13.32$ mm, $h = 2.025$ mm) versus temperature (measurement data from [3]).

II. Experiments

The irradiated HRFZ silicon samples investigated in this letter have been manufactured by Topsil. Similar to in [3], either $TE_{01\delta}$ or WG modes have been induced in a cylindrical silicon sample having a diameter of 13.32 mm and a thickness of 2.025 mm. Silicon wafers measured in the SPDRs and in the FPOR had a diameter of 100 mm. In the FPOR shown in Fig. 1, only individual wafers were measured, whereas in the SPDRs wafers have been stacked to achieve better sensitivity of the loss tangent measurements. SPDRs, similar to those in [4], operating at 1.88, 2.45, and 5.14 GHz have been used in the experiments.

At first, the bulk-irradiated HRFZ silicon sample ($d = 13.32$ mm, $h = 2.025$ mm) has been characterized as a function of temperature taking into account thermal expansion coefficients of the sample and copper cavity. Measurement results of the real part of permittivity versus temperature obtained at 10.5 GHz are shown in Fig. 2. It can be noted that permittivity is almost temperature independent at very low temperatures. However, for $T > 200$ K, the real part of the relative permittivity can be fitted with

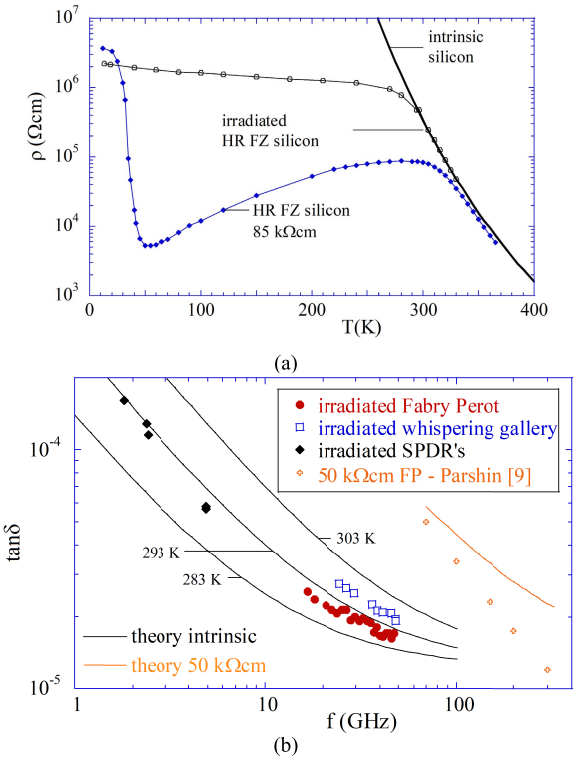

Fig. 3. (a) Measured resistivity of as-grown (r = 85 kWcm) and irradiated HRFZ silicon (Topsil) versus temperature (measurement data from [3]). (b) Measured and computed loss tangent of the irradiated HRFZ silicon. Literature data [9] are included for comparison.

$\varepsilon'_r = 11.29 + 1252.1 \times 10^{-6} \times T$. It should be mentioned that these relations are valid in the whole microwave and terahertz spectra. Uncertainty of the measurement of the real part of permittivity is better than ±0.5%.

The dielectric loss tangent and resistivity of that sample have been also determined and the results of those measurements are shown in Fig. 3(a). It can be noted that for $T > 295$ K, the resistivity of the irradiated HRFZ silicon can be fitted with the following formula:

$$\rho = 9.39 \times 10^{-5}(T/300)^{0.487} \exp(6594/T)(\Omega\text{cm}) \qquad (2)$$

which resembles the properties of the intrinsic silicon.

According to Fig. 3(a), (2) is valid for $T > 330$ K in the case of a pure HRFZ silicon (with $\rho = 85$ kΩcm at room temperature). At lower temperatures ($T < 250$ K), the resistivity of the irradiated HRFZ silicon is almost temperature independent at the level over 1000 kΩcm, whereas for high-purity HRFZ silicon resistivity falls with the decrease of temperature down to 5 kΩcm at $T = 50$ K [see blue solid line in Fig. 3(a)]. It implies that the irradiated silicon has about 200 larger resistivity than the high purity one at $T = 50$ K. Significant changes in resistivity of the high-purity HRFZ silicon with temperature for 50 K $< T <$ 250 K are associated with the changes in charge carriers' mobility.

Expected values of the loss tangent have been evaluated for $T = 283$ K, $T = 293$ K, and $T = 303$ K using (1), assuming intrinsic resistivity as in (2), $\varepsilon'_r = 11.65$ and $\tan\delta_e = 1.2 \times 10^{-5}$. The outcome is shown in Fig. 3(b) (see black solid lines) together with our measurement data and supplemented with literature data [9]. Measurements employing SPDRs and FPOR have been performed at $T = 293$ K, while the measurements employing WG modes (see blue squares) were undertaken at $T = 298$ K. Measurement data in [9] were obtained from Fabry–Perot measurements on HR ultrapure gold-doped silicon with resistivity at the level of 50 kΩcm. Assuming that the loss tangent of silicon due to phonon absorption, $\tan\delta_e$, had been negligibly small, measurement results shown in Fig. 3(b) [9] could have been approximated with a straight line. However, the total loss tangent at higher frequencies is mainly driven by $\tan\delta_e$, so it cannot be neglected (see the orange solid line). Taking into account finite uncertainties of the applied measurement technique, it is difficult to estimate precisely that parameter.

The loss tangent of HR silicon evaluated from the ellipsometric measurements of the refractive index and the extinction coefficient at frequencies from 580 to 2550 GHz performed by Giles [8] lay in the range $\tan\delta = (2.3 - 4.1) \times 10^{-5}$ with the measurement uncertainties at the order of 2×10^{-5}. The refractive index obtained by Giles [8] at room temperature ($n = 3.4162 \pm 0.0002$) is in perfect agreement with the measurement data shown in Fig. 2, which confirms that the real part of the permittivity of silicon is nondispersive up to terahertz frequencies.

It should be mentioned that the most accurate loss tangent extraction is the WG mode measurements with the relative uncertainty of about 5% because the electric energy is predominantly concentrated in the sample and conduction losses in the metallic cavity are negligibly small. For the SPDR and FPOR techniques, the absolute uncertainty of measurements is in the order of about 1×10^{-5} because only a small part of the electric energy is stored in the MUT and metal wall losses are relatively large. As a result, the Q-factor of an empty fixture is relatively close to that of the sample. Apart from our measurements of the irradiated silicon employing WG modes up to 50 GHz [see blue squares in Fig. 3(b)], there are measurement results performed by Vogt and Leonhardt [19] at 620 GHz showing that a critically coupled WG mode resonator made of HR silicon ($\rho \approx 10$ kΩcm) had the loaded Q-factor at the order of 15 000. One can estimate, from their experiments, that the unloaded Q-factor was about 30 000 and the corresponding loss tangent of about 3×10^{-5}. More precise determination of the loss tangent of silicon related to pure dielectric losses, $\tan\delta_e$, can be established by employing WG mode measurements at higher terahertz frequencies.

III. Conclusion

Irradiated HRFZ silicon can find applications such as a very low loss material with the loss tangent smaller than 10^{-4} at $T < 300$ K at frequencies larger than 10 GHz. It should be mentioned that applications of the irradiated silicon are not recommended for $T > 300$ K due to the fact that its resistivity rapidly falls with temperature in that range and the process is irreversible. This is related to the relaxation of defects introduced by irradiation. The higher the temperature, the shorter the relaxation time when irradiated silicon loses its HR. Therefore, only low-temperature technological processes are allowed on the irradiated silicon wafers.

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