Measurements of Permittivity, Dielectric Loss Tangent, and Resistivity of Float-Zone Silicon at Microwave Frequencies

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***Abstract*—The complex permittivity and resistivity of float-zone high-resistivity silicon were measured at microwave frequencies for temperatures from 10 up to 400 K employing dielectric-resonator and composite dielectric-resonator techniques. At temperatures below 25 K, where all free carriers are frozen out, loss-tangent values of the order of 2×10^{-4} were measured, suggesting the existence of hopping conductivity or surface charge carrier conductivity in this temperature range. Use of a composite dielectric-resonator technique enabled the measurement of materials having higher dielectric losses (or lower resistivities) with respect to the dielectric-resonator technique. The real part of permittivity of silicon proved to be frequency independent. Dielectric losses of high-resistivity silicon at microwave frequencies are mainly associated with conductivity and their behavior versus temperature can be satisfactory described by dc conductivity models, except at very low temperatures.**

***Index Terms*—Conductivity measurement, dielectric losses, dielectric resonators, permittivity measurement, silicon, semiconductor materials measurements.**

I. INTRODUCTION

OVER THE past 50 years, there have been numerous attempts to measure the complex permittivity of semiconductors at microwave frequencies employing waveguides, resonators, and broadband dispersive Fourier transform spectroscopic technique [1]–[7]. For the most accurate measurement of the complex permittivity or conductivity of semiconductors at microwave frequencies, it is essential that the sample under test has no electrical contact with any metal part of the fixture that is used for measurements. Some microwave techniques offer the possibility of contactless measurements, e.g., cylindrical resonant cavities and waveguides operating in one of the TE_0 modes (usually the dominant one) [3]–[5]. For such structures, currents have only circumferential component and, thus, do not flow through the metal–semiconductor interface. Such a situation also occurs when the sample under test is separated from all metal parts of the fixture, e.g., [2]. In general, the complex permittivity of a semiconductor material is given by (1) as follows:

$$\varepsilon = \varepsilon_0 \left(\varepsilon_r - j\varepsilon_r'' - j\frac{\sigma}{\omega\varepsilon_0} \right) = \varepsilon_0\varepsilon_r(1 - j\tan\delta) \qquad (1)$$

where $\tan\delta$ is the effective dielectric loss tangent of the semiconductor given by (2) as follows:

$$\tan\delta = \tan\delta_d + \frac{\sigma}{\omega\varepsilon_0\varepsilon_r} \qquad (2)$$

where ε_o is the permittivity of the vacuum, ε_r is the relative real permittivity of the semiconductor, ω is the angular frequency, σ is conductivity, and $\tan\delta_d$ is the dielectric loss tangent associated with pure dielectric loss mechanisms (e.g., electronic and ionic polarization).

For doped semiconductors and for intrinsic semiconductors having energy gaps smaller than 1 eV, the dominant loss mechanism is related to the conductivity associated with free charge carriers up to high microwave frequencies and at temperatures that exceed the activation energy of dopands. For such materials, their dielectric loss tangent can be represented by the second term on the right-hand side of (2). High-resistivity float-zone silicon has found applications as a substrate material for various microwave devices such as transmission lines, filters, or antennas, especially at millimeter-wave frequencies [8]–[10]. Over the last years, significant progress has been achieved in growing silicon crystals with very large resistivities exceeding 10 kΩ cm; however, it is difficult to find in the literature or from manufacturers accurate measurement data of their complex permittivity, in particular as a function of temperature. This data is essential in the design of microwave devices. Such measurements are reported in this paper.

II. MEASUREMENTS TECHNIQUES

Two measurement setups were used in this study and are shown in Fig. 1. In the first setup [see Fig. 1(a)], the sample under test was situated on a small single crystal quartz support inside a cylindrical cavity and the whole structure was mounted on the cold head of a closed cycle Gifford–McMahon cryocooler for low-temperature measurements or in an oven for elevated temperature measurements. Adjustable coupling mechanisms were used to control coupling coefficients from both ports of the resonator. The resonators were attached to the network analyzer via semirigid coaxial cables. In the second setup [see Fig. 1(b)], the same cylindrical cavity and single crystal

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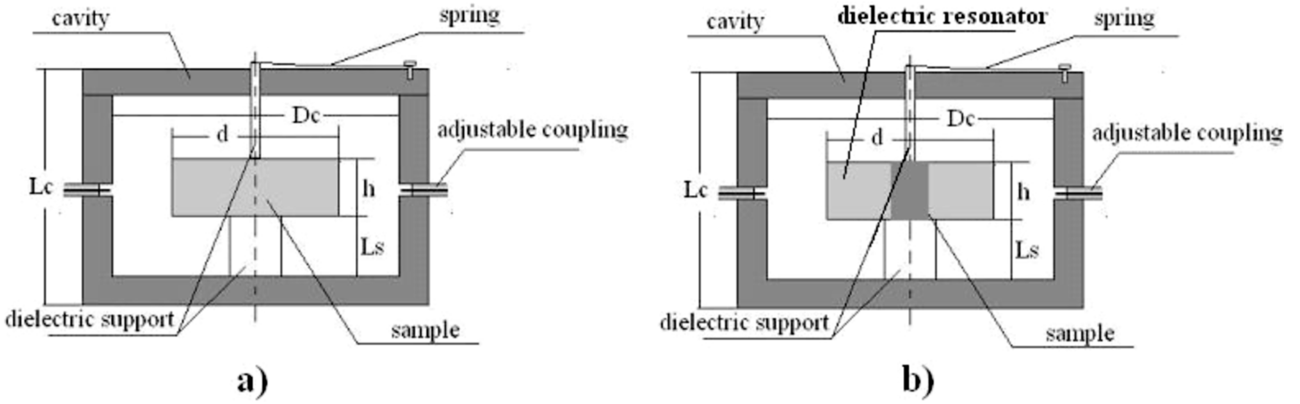

Fig. 1. Sketch of measurement setups used in experiments. (a) Sample under test situated in cylindrical cavity. (b) Sample under test situated inside cylindrical dielectric resonator.

quartz support from the first configuration has been used, but the sample under test had a reduced diameter and was situated inside a ceramic dielectric ring resonator. The purpose of using a dielectric resonator configured in this way was to reduce the electric energy filling factor in the sample and, therefore, increase the Q factor due to conductor losses in the silicon sample.

In the second measurement setup, the contribution to the Q factor due to losses in the sample increased by one order of magnitude compared to the first measurement setup. This allows measurements of dielectric loss tangents one order of magnitude larger than those that can be measured in the first setup. High-resistivity silicon has dielectric loss tangents of the order of $10^{-2} - 10^{-3}$ at room temperature and microwave frequencies. Therefore, techniques that have been used for measurements of its complex permittivity were the same as that used for dielectrics [11]–[14]. Most of the measurements were performed using the quasi-TE_{011} mode. This appears as the second resonance in the first measurement setup or the first resonant peak in the second. For some cryogenic measurements, other higher order modes have also been employed. In order to find the relationship between the measured resonant frequencies, Q factors the real part of the relative permittivity, and dielectric loss tangent, it is necessary to rigorously solve Maxwell's equations for the structure under test. This was done using rigorous mode-matching and Rayleigh-Ritz techniques described in detail in [11]. In this paper, only the most important aspects of resonant techniques are explained. If the effective dielectric loss tangent of the sample is smaller than 0.1, then the resonant frequencies depend on the real part of the permittivity and the dimensions of the resonant structure. In such a case, the real part of the permittivity can be evaluated from the measured resonant frequency of a specific mode taking into account thermal expansion of the resonant structure and the sample under test. The dielectric loss tangent of the sample can then be evaluated from

$$\tan\delta = \left(Q_u^{-1} - Q_p^{-1}\right)/p_{es} \tag{3}$$

where Q_u is the measured unloaded Q factor of specific mode of resonator containing the sample under test, Q_p is the Q factor associated with parasitic losses in the cavity including metal wall losses and dielectric losses in the dielectric resonator and in the single crystal quartz support, and p_{es} is the electric energy filling factor for the sample under test (ratio of the electric energy stored in the sample to the electric energy stored in whole resonator)

$$p_{es} = \frac{\iiint\limits_{Vs} \varepsilon_s |\mathbf{E}|^2 dv}{\iiint\limits_{Vt} \varepsilon(v) |\mathbf{E}|^2 dv} \tag{4}$$

where Vs is the volume of the sample, Vt is the volume of the whole resonant structure, $\varepsilon(v)$ is the spatially dependent permittivity inside the whole resonant structure, and ε_s is the permittivity of the sample.

The Q factor due to parasitic losses can be found from the following formulas:

$$Q_p^{-1} = Q_d^{-1} + Q_c^{-1} \tag{5}$$

where $Q_d^{-1} = p_{\mathrm{DR}} \tan\delta_{\mathrm{DR}} + p_{qs}\tan\delta_{qs}$, $p_{\mathrm{DR}}(p_{qs})$ is the electric energy filling factors in dielectric resonator and in the single crystal quartz support, respectively, $\tan\delta_{\mathrm{DR}}, (\tan\delta_{qs})$ are the dielectric loss tangents of the dielectric resonator and the single crystal quartz support, respectively, and Q_c is the Q factor due to conductor losses in metal cavity walls

$$Q_c^{-1} = R_S/G \tag{6}$$

where R_S is the surface resistance of metal cavity walls at a given frequency, G is the geometric factor, which is defined as

$$G = \omega \frac{\iiint\limits_{Vt} \mu_0 |\mathbf{H}|^2 dv}{\iint\limits_{S} |\mathbf{H}_\tau| ds} \tag{7}$$

S is the internal surface of the cavity, and $\mathbf{H}_\tau$ is the component of the magnetic field tangential to the internal surface of the cavity.

The Q factor associated with parasitic losses can be evaluated from measurements made of the unloaded Q factors of the resonator without the silicon sample and of the empty cavity versus temperature. These measurements allow assessment of the surface resistance of the cavity walls and, in the second, experimental setup of the losses in the dielectric resonator, as a function of temperature. Losses in the single crystal quartz support are negligible due to the small electric energy filling factor value

TABLE I
RESULTS OF ROOM-TEMPERATURE MEASUREMENTS. $Dc = 24.0$ mm, $Lc = 16.12$ mm, $d = 15.455$ mm (*7.97 mm), $h = 6.0$ mm, $Ls = 4.26$ mm

Mode	f(GHz)	Q_u	Q_p	p_e	tanδ
TE_{011}	6.685	550	38950	0.9605	0.001866
HE_{111}	6.797	1134	18030	0.4346	0.001901
*TE_{011}	4.824	4036	16200	0.0885	0.002101

in it and the extremely low dielectric losses of quartz (below 2×10^{-5}). Once the material properties of metal cavity walls and dielectric parts in the cavity are known, the geometric factor and electric energy filling factors in the sample are numerically evaluated. It should be mentioned that, for high-resistivity silicon, the parasitic losses in the measurement setups are much smaller than losses in the silicon sample since effective dielectric loss tangent of silicon is usually larger than 10^{-3}, except at very low temperatures. In this case, the effective dielectric loss tangent can be determined with approximately the same precision as a Q-factor measurement, i.e., approximately 1%–2%. When dielectric loss tangent values in silicon samples are of the order of 10^{-5} or less (at very low temperatures), it is still possible to measure them very precisely by employing modes having large azimuthal mode indices, i.e., the so-called whispering-gallery modes. By employing whispering-gallery-mode techniques, dielectric loss tangents as low as 10^{-9} have been measured on high-purity single-crystal sapphire samples at cryogenic temperatures [12].

The upper limit for loss-tangent measurements is associated with the lowest Q-factor values that can be effectively measured, and the value of the electric energy filling factor. In our resonant cavity, the minimum value of the measurable Q factor was approximately 100. The electric energy filling factors for the two measurement setups are shown in Table I, together with room-temperature measurements results for the two samples.

As can be seen in Table I, the use of the quasi-HE_{111} mode enables the measurement of losses approximately twice as large as that obtainable using the quasi-TE_{011} mode. This is related to an electric energy filling factor value for the HE_{111} mode, which is twice as high as that of the TE_{011} mode. An electric energy filling factor in the sample can be further reduced by employing a dielectric ring resonator, as in the second measurement setup. In this case, the electric energy filling factor in the sample is reduced by an arbitrary number, which depends on the permittivity and external diameter of the dielectric resonator. The last row of Table I shows that, for the second measurement setup, the electric energy filling factor has been reduced by a factor of 10.

III. RESULTS OF EXPERIMENTS

A. *Measurements Employing Silicon Sample as the Dielectric Resonator*

Measurements at room temperature have been performed on two bulk cylindrical p-type silicon samples having diameters of 15.455 mm (Sample #1) and 15.457 mm (Sample #2) and a height of 6.0 mm. The surfaces of the samples were mechanically polished, but not to optical quality. The real part of the relative permittivity ε_r was extracted for both samples at different frequencies by employing higher order TE_0 modes. The results of these measurements showed the relative permittivity to be independent of frequency. In permittivity determination, the thermal expansion of the silicon samples and copper cavities were taken into account. Results of permittivity measurements as a function of temperature are shown in Fig. 2.

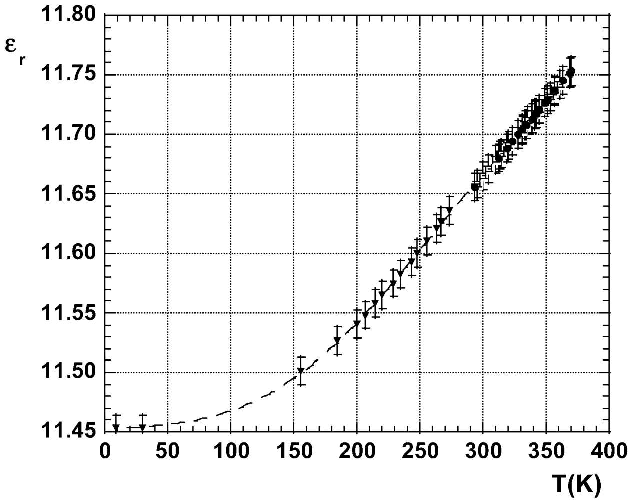

Fig. 2. Permittivity versus temperature for sample with $d = 15.455$ mm. Experimental data points include results extracted from measurements employing quasi-TE_{011} mode in two cavities (6.62 and 6.69 GHz) and the third TE_0 mode (11.5 GHz) in the second cavity.

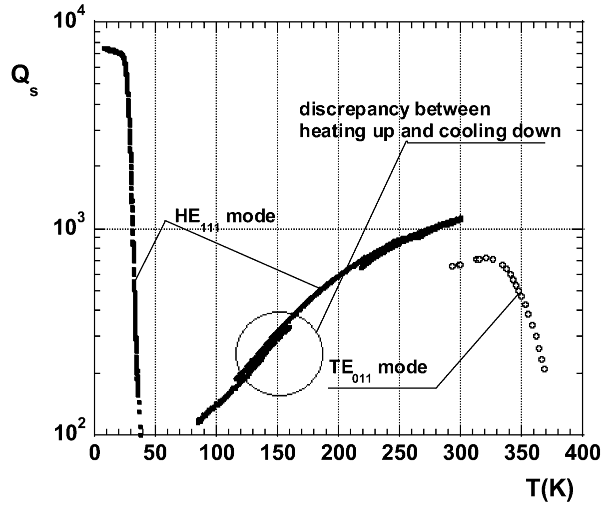

Fig. 3. Q factors in the sample versus temperature for silicon sample #1 with $d = 15.455$ mm measured at frequencies of approximately 6.8 GHz (HE_{111} mode) and 6.62 GHz (TE_{011} mode).

It is observed that results of experiments are very smooth with experimental errors in the range of 0.1%. The experimental errors predominantly depend on dimensional uncertainties of the samples under test. Results of measurements of the Q factor and dielectric loss tangent versus temperature for bulk silicon samples are presented in Figs. 3–6. In the temperature region of 100–250 K, losses are associated with conductivity due to free holes. In this region, all Boron atoms are ionized, and the conductivity, and therefore, the dielectric loss tangent, depends on the mobility of the holes [16]. Measurements between 36–100 K

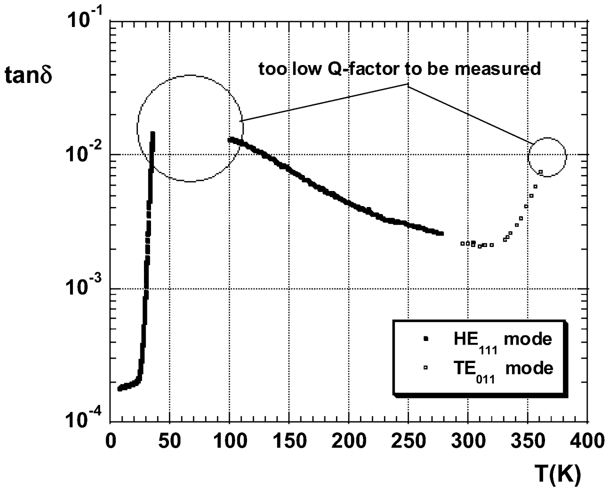

Fig. 4. Effective dielectric loss tangent versus temperature for silicon sample #1 with $d = 15.455$ mm measured at frequencies of approximately 6.8 GHz (HE_{111} mode) and 6.62 GHz (TE_{011} mode).

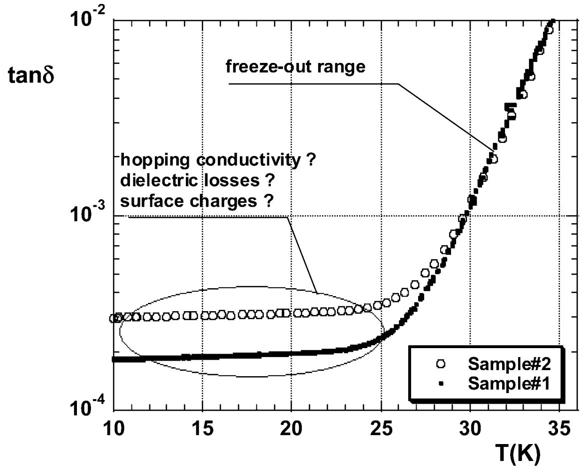

Fig. 5. Effective dielectric loss tangent versus temperature at low-temperature region for two silicon samples at frequency of approximately 6.8 GHz (HE_{111} mode).

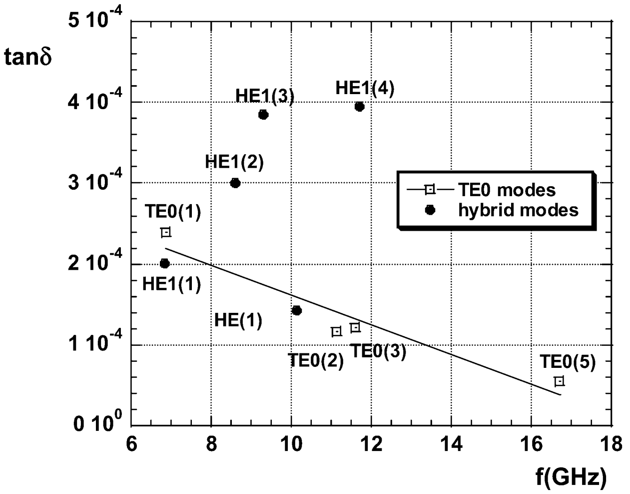

Fig. 6. Effective dielectric loss tangent of sample #2 at 10 K evaluated using Q factors of different modes.

on bulk silicon samples were not possible due to the Q-factor values being less than 100.

Hysteresis effects are observed in the sample. This is manifested by measuring different Q factors at the same temperature when the temperature is increasing compared to when it was decreasing. The increase in losses at temperatures above 310 K are related to the generation of free charge carriers through the energy gap, and above 350 K, high-resistivity silicon becomes essentially intrinsic. In the temperature range of 25–40–K, hole freeze-out effects can be observed. It is not clear what the origin of dielectric losses are below 25 K. To some extent, losses in this temperature range may be attributed to hopping conductivity [15], nonuniform dopand distribution in the sample, and accumulation of charge carriers on the surface of samples or dielectric losses. Additional measurements of losses at 10 K versus frequency have been performed employing several higher order modes. Results of these measurements are shown in Fig. 6 where the first number in the description of the modes denotes an azimuthal mode index, while the second one, in parenthesis, denotes the sequence of the mode on the frequency scale. In other words, both radial and axial mode indices are combined into one as they are not integer numbers for our resonant structure. In the literature, these indices are often denoted by Greek characters to underline this feature, or alternatively, such modes are termed "quasi." It is seen that dielectric loss tangent values measured with TE_0 modes decrease with increasing frequency. This result supports the assumption about conductive loss mechanisms such as hopping conductivity. It should be mentioned that for TE_0 modes, the electric field in the cavity (and sample) only has an azimuthal component that is tangential to all sample surfaces. This is not true for measurements based on the hybrid modes that have all three spatial components of the electric field, some of them perpendicular to the sample surfaces. The spatial distribution of the electric field for hybrid modes is complicated and unique for each mode. If conductivity is nonuniform or anisotropic in the sample volume, it may lead to different values of measured dielectric losses for each mode, as seen in Fig. 6.

It should also be noted that measurement errors employing TE_0 modes at 10 K are small, around 2% or less, because all resonances have Q factors of the order of a few thousand, and they are well separated in frequency and easily identified. Measurements performed in the same cavity on a single-crystal MgO sample at 10 K had shown dielectric loss tangent values below 1×10^{-6}.

B. Measurements Employing Composite Dielectric-Resonator Technique

For these measurements, the diameter of sample #1 is reduced creating sample #3. Sample #3 has a diameter of 7.97 mm and a height of 6.00 mm. A cylindrical ring dielectric resonator was manufactured from $\mathrm{Ba(Zn_{1/3}Ta_{2/3})O_3}$ (BZT) ceramic. It has the same height as that of the silicon sample, an external diameter of 16.14 mm, and an internal diameter of 8.00 mm. The permittivity of the ceramic dielectric is 29.86 and the dielectric loss tangent $\tan\delta$ is 5.9×10^{-5} at 10 GHz.

For reference, measurements of parasitic losses versus temperature when a PTFE sample was inserted instead of the silicon sample were undertaken. The PTFE sample was used to keep the BZT resonator in a fixed position. This was necessary due to the vibrations of the close-cycle helium refrigerator. A room-temperature measurement showed that the Q factor and resonant

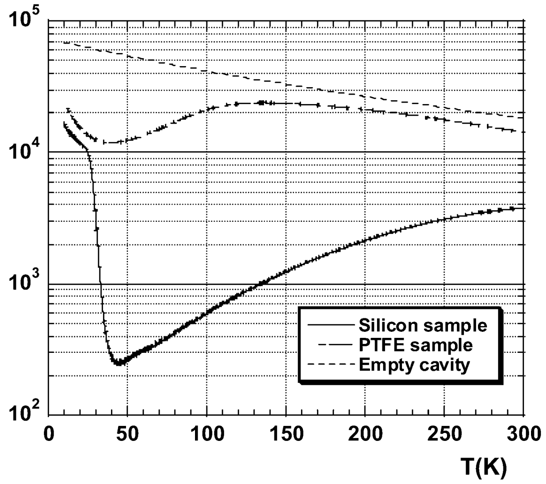

Fig. 7. Unloaded factors versus temperature for quasi-TE_{011} mode of BZT dielectric resonator containing silicon sample #3 and PTFE sample having $d = 7.97$ mm at a frequency of approximately 4.98 GHz (TE_{011} mode). Q factor of the empty cavity without BZT dielectric resonator. Support is additionally denoted via the dotted line.

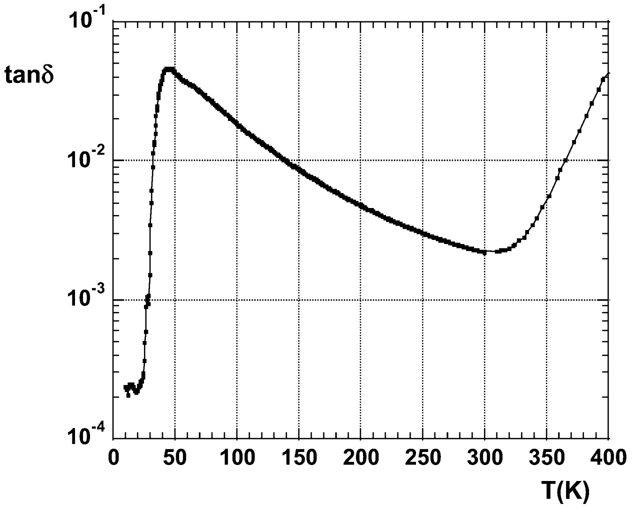

Fig. 8. Effective dielectric loss tangent versus temperature for silicon sample #3 at a frequency of approximately 4.98 GHz employing TE_{011} mode in composite dielectric resonator.

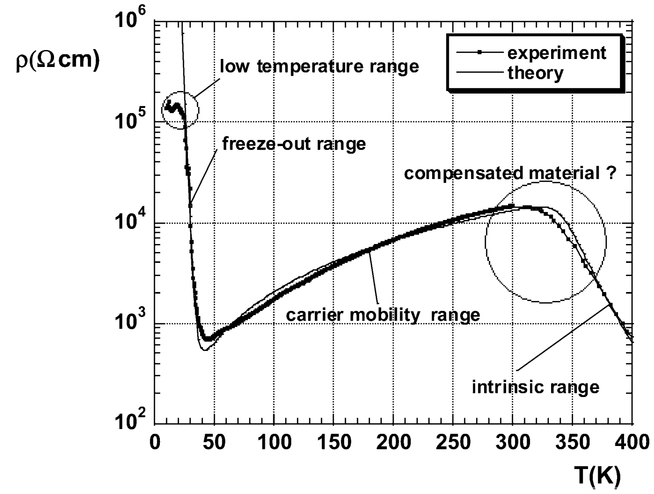

Fig. 9. Resistivity versus temperature extracted from measurements shown in Fig. 8 for silicon sample #3 at a frequency of approximately 4.98 GHz. Parameters of silicon assumed in computations of theoretical curve are shown in Table II.

TABLE II
PARAMETERS OF SILICON ASSUMED IN THEORETICAL COMPUTATIONS OF RESISTIVITY

Acceptor ionization energy E_a(eV) (Boron)	0.045
Acceptor concentration N_a (cm^{-3})	1.05×10^{12}
Electron mobility v_e ($cm^2/(Vs)$)	$1430x(T/300)^{-2.3}$
Hole mobility v_h ($cm^2/(Vs)$)	$470x(T/300)^{-1.76}$
Energy gap E_g (eV)	$1.1785\text{-}9.025x10^{-5}xT\text{-}3.05\ x10^{-7}xT^2$
n_i - intrinsic carrier concentration at 300 K (cm-^{3})	$1.75x10^{10}$

frequency of the TE_{011} mode for the composite resonator with a PTFE sample is essentially the same as that without it. At cryogenic temperatures, PTFE losses are very low so that one can treat the Q-factor values of the resonator with a PTFE sample as being the same as the Q-factor values for an empty dielectric resonator. Additionally, the Q factor of the empty cavity without the BZT resonator and quartz support was measured in order to evaluate the surface resistance changes of the silver-plated cavity versus temperature. Results of Q-factor measurements and loss tangent determination for sample #3 are shown in Figs. 7 and 8. It is seen that by employing a composite dielectric resonator, measurements are possible in the whole temperature range using the quasi-TE_{011} mode. The upper limit on measurement temperature for the composite resonator is determined by the resonator construction rather than by the dielectric losses in silicon. For this structure, it is limited by the coaxial cables and the use of a tin solder.

Dielectric loss tangent values for samples #1 and #3 that are shown in Figs. 4 and 8 are similar when they are scaled with frequency. Measurements of dielectric loss tangents larger than 2×10^{-3} are more accurate, employing the composite dieletric-resonator technique, while measurements of loss tangents smaller than 1×10^{-3} are more accurate when the silicon sample stands alone as a dielectric resonator. In Fig. 9, resistivity values extracted from measurements of the loss tangents shown in Fig. 8 are presented. For comparison, we have evaluated theoretical resistivity values from well-known silicon resistivity models (e.g., see [15]). The best fit to experimental data has been obtained assuming the parameters of silicon given in Table II. It can be noticed that temperature dependence of resistivity, or dielectric loss tangent, can be satisfactory explained for temperatures larger than 25 K on the basis of well-understood theories of semiconductors. At temperatures above 25 K, thermal energy is sufficient for partial ionization of Boron dopands, which become fully ionized at temperatures close to 45 K. At temperatures from 45 to 300 K, the number of free carriers is approximately constant and conductivity decreases predominantly due to decreasing hole mobility (in p-type semiconductor). If the temperature increases to approximately 300 K, the number of additional free carriers generated through the forbidden energy gap becomes comparable to the number of holes due to ionized acceptors, and at temperatures above 350 K, high-resistivity silicon becomes essentially intrinsic with an approximately equal number of holes and electrons.

The origin of dielectric losses at temperatures below 25 K is not yet clear and requires further investigations.

IV. CONCLUSIONS

The dielectric losses of float-zone high-resistivity silicon have been measured between 10–400 K using two dielectric resonator measurement techniques. Using a novel composite dielectric-resonator configuration, measurements are possible even for samples with very low Q factors. The composite dielectric-resonator technique can be easily adopted for measurements of arbitrary semiconductors, even those having much smaller resistivity than the high-resistivity silicon used here, by the appropriate choice of dielectric-resonator dimensions. Measurements at temperatures as high as 600 K will be possible by using appropriate coaxial cables with welded connectors and silica insulation.

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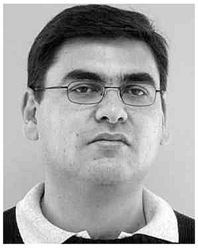

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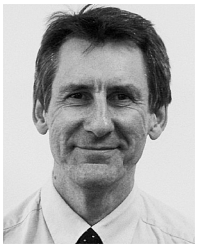

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