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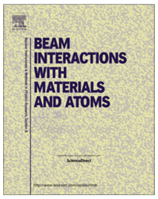

Electrical properties of as-grown and proton-irradiated high purity silicon

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ABSTRACT

The complex permittivity of as-grown and proton-irradiated samples of high purity silicon obtained by the floating zone method was measured as a function of temperature at a few frequencies in microwave spectrum by employing the quasi TE_{011} and whispering gallery modes excited in the samples under test. The resistivity of the samples was determined from the measured imaginary part of the permittivity. The resistivity was additionally measured at RF frequencies employing capacitive spectroscopy as well as in a standard direct current experiment. The sample of as-grown material had the resistivity of ~85 kΩ cm at room temperature. The sample irradiated with 23-MeV protons had the resistivity of ~500 kΩ cm at 295 K and its behavior was typical of the intrinsic material at room and at elevated temperatures. For the irradiated sample, the extrinsic conductivity region is missing and at temperatures below 250 K hopping conductivity occurs. Thermal cycle hysteresis of the resistivity for the sample of as-grown material is observed. After heating and subsequent cooling of the sample, its resistivity decreases and then slowly (~50 h) returns to the initial value.

1. Introduction

The electrical properties of silicon irradiated with high-energy particles, such as protons, significantly change due to the damage of its crystal lattice [1,2] and such material exhibits extremely high resistivity. Many experimental techniques [3–8] have been used to study the electrical properties of silicon after irradiation. These techniques include Hall effect measurements [9,10], photo-induced current transient spectroscopy [11,12], the pocket pumping method [13] and the microwave split post dielectric resonator method [14]. Studies of irradiation-induced defects in silicon have been also carried out by deep level transient spectroscopy (DLTS), electron paramagnetic resonance (EPR), Fourier transform infrared absorption (FTIR), and photoluminescence spectroscopy (PL). The properties of radiation defect centers determined from these studies were reported e.g. in the references [14–21]. The high resistivity of irradiated silicon is usually explained by the free charge carrier compensation resulting from the appearance of deep energy level centers related to the irradiation defects. The oxygen-related shallow donors were observed earlier in silicon grown by floating zone (FZ) and Czochralski (Cz) techniques, as well as in epitaxial silicon subjected to irradiation with a high fluence of high-energy particles [22,25]. The activation energies of these centers determined from the high-resolution photo induced transient spectroscopy (HRPITS) studies were in the range from 10 to 30 meV. It is believed that irradiation-induced shallow donor centers are associated with electrically active small aggregates of oxygen atoms that are formed during irradiation [22–25]. The diffusivity of oxygen strongly depends on the concentrations of irradiation-induced point defects in silicon [26,27]. The well-known oxygen-related thermal donors that are formed in Czochralski grown silicon by its annealing at 450 C are also identified as oxygen aggregates [28]. Because the native point defect concentrations in an as-grown single crystal are by orders of magnitude lower than those that are formed in an irradiated material, a higher temperature is needed to make the diffusivity of oxygen atoms sufficient for the formation of these aggregates [26,27]. The difference in the electronic properties of the thermal donors results from the different size of the oxygen aggregates.

Detailed studies of the electrical properties of irradiated silicon are important for two reasons. Firstly, high-resistivity silicon detectors are commonly used in nuclear power stations and particle colliders and their properties deteriorate after irradiation, so it

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is essential to improve their performance. Secondly, after the irradiation with high fluences of high-energy particles, the resistivity of lightly doped silicon becomes very high and such material can find applications in microwave and terahertz technology.

In this work we have performed comparative studies of the electrical properties of the highest purity as-grown and proton-irradiated floating zone silicon as a function of temperature. The major measurement technique which has been used was the contactless dielectric resonator technique which allowed us to measure permittivity and the total dielectric loss tangent of the two kinds of high purity silicon samples. The bulk resistivity of the samples was determined from the measured dielectric loss tangent values. Additionally, the resistivity of the irradiated sample was measured versus temperature by employing standard direct current experiment, at room temperature, by means of capacitive spectroscopy at RF frequencies.

2. Theory

Electromagnetic properties of a semiconductor are specified by the complex relative permittivity (1)

$$\varepsilon_r = \varepsilon_r' - j\varepsilon_r'' - j\frac{1}{\rho\omega\varepsilon_0}, \tag{1}$$

where

ε_0 is the permittivity of vacuum,
ε_r – the relative complex permittivity,
ε_r' – the real part of the relative complex permittivity,
ε_r'' – the imaginary part of the relative complex permittivity,
ω – the angular frequency,
ρ – resistivity.

The imaginary part of the permittivity contains two terms. The first one is associated with dielectric loss mechanisms and the second one with the material conductivity. The dielectric loss tangent is given by the formula

$$\tan\,\delta = \frac{\mathrm{Im}(\varepsilon_r)}{\mathrm{Re}(\varepsilon_r)} = \tan\,\delta_d + \frac{1}{\rho\omega\varepsilon_0\varepsilon_r'}, \tag{2}$$

where $\tan\,\delta_d = \varepsilon_r''/\varepsilon_r'$ denotes dielectric loss tangent associated with pure dielectric loss mechanisms (e.g. electronic and ionic polarization).

We have used both the quasi TE_{011} and the whispering gallery modes to measure each of our samples at a few frequency points. The electric energy filling factors p_e in the samples under test were larger than 0.91 for the whispering gallery modes and about 0.9 for the TE_{011} mode. For the whispering gallery modes the total measured *Q*-factor values Q_u were practically not affected by the conductor losses in the metal walls of the cavity. Therefore for these modes the dielectric loss tangent was determined from the formula $\tan\,\delta = 1/(p_eQ_u)$. Such a method was previously used to measure resistivity of gallium arsenide and gallium phosphide [29]. In our earlier paper [30], quasi TE_{011} mode was used for measurements of high resistivity silicon, however its sensitivity is not sufficient to accurately measure the dielectric loss tangent values that are smaller than 1×10^{-5}. Whispering gallery modes allow measurements of arbitrarily small losses and employing them dielectric loss tangent values that were smaller than 1×10^{-9} were measured on sapphire [31].

A schematic of the microwave cavity containing the sample under test is presented in Fig. 1a. 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 cryo-cooler for low and elevated-temperature measurements.

Additional measurements of the samples at room temperature have been performed in a capacitance dielectric test fixture (Fig. 1b) employing a multilayer model of the sample. In this model it is assumed that the sample under test consists of several layers having different thicknesses and resistivities. The capacitance and the *Q*-factor of the fixture containing the sample are measured over several decades of frequency. The thickness and resistivity of individual layers are obtained by applying optimization procedure which allows us to achieve the best fit of the model to the experimental data. Details of the measurement procedure are described in [32]. For the irradiated sample we have also performed resistivity measurements using a direct current setup which is depicted in Fig. 1c.

3. Experiments and discussion

Two high resistivity silicon samples were measured. The first one had diameter 13.28 mm and thickness 1.95 mm and the second, irradiated sample, had diameter 13.32 mm and thickness 2.052 mm. The first sample was made from the highest purity silicon obtained by the floating zone method by Topsil Semiconductor Materials A/S. The level of unintentional impurities (Phosphorus, Boron) in this material was smaller than $1 \times 10^{11}\,\mathrm{cm}^{-3}$ according to the information obtained from the manufacturer. On this sample majority conduction species were not able to be inferred using the available apparatus.

The second sample, also obtained by the floating zone method had a resistivity of 2000 Ω cm before irradiation. The semi-insulating properties of the sample were achieved by its irradiation with 23-MeV protons at the Karlsruhe Institute of Technology. The applied proton fluence was $5 \times 10^{14}\ n_{\mathrm{eq}}/\mathrm{cm}^2$.

Initial information on the electrical properties of both samples was obtained from measurements of the capacitance and *Q*-factor of the samples versus frequency in the capacitive dielectric test fixture (Fig. 1b). Assuming a 5 layer model of the sample and applying the optimization procedure we have obtained thickness and resistivity of subsequent layers [32]. Results are presented in Table 1. From results that are shown in Table 1 it is seen that the resistivity of the sample is not uniform in the direction perpendicular to the sample surfaces. For the first sample, further denoted as FZ85, the average resistivity is about 85 kΩ cm, while for the second – the irradiated sample, the average resistivity is about 520 kΩ cm at room temperature. The sequence of the layers cannot be determined with the capacitance technique but we suspect that the high resistivity layers correspond to the depletion regions in the vicinity of the surfaces of the samples. It is seen that for the irradiated sample the lowest resistivity fraction (504 kΩ cm) occupies 90% of the volume of the sample. For the FZ85 sample the lowest resistivity fraction (71 kΩ cm) occupies 70% of the volume of the sample (see Fig. 2).

The next experiments have been performed at microwave frequencies in the temperature range from 13 K to 365 K. The resonance frequencies and the unloaded *Q*-factors of a few different modes were measured as a function of temperature after cooling the fixture to the minimum achievable temperature of 13 K. Measurement results for the first sample at the temperatures of 13 K and 295 K are shown in Table 2.

As has been already mentioned for whispering gallery modes the total dielectric loss tangent can be obtained as $\tan\,\delta = 1/(p_eQ_u)$ while for the quasi TE_{011} mode the *Q*-factor due to conductor losses in metal cavity walls must be taken into account to determine the *Q*-factor Q_S depending on the losses in the sample and then the total dielectric loss tangent. In practice we have used appropriate software dedicated to determination of permittivity and the dielectric loss tangent in the quasi TE_{011}