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ORIGINAL PAPER

Correction of the electric resistivity distribution of Si wafers using selective neutron transmutation doping (SNTD) in MARIA nuclear research reactor

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Abstract. The result of the electric resistivity distribution modification in silicon wafers, by means of selective neutron transmutation doping (SNTD) method in the MARIA nuclear research reactor at Świerk/Otwock (Poland) is presented. Silicon wafer doping system has been fully designed for the MARIA reactor, where irradiation took place. The silicon wafer resistivity distribution after SNTD has been measured by the capacity voltage (C-V) method. In this article we show first results of this correction technique. The result of the present investigation is that the planar resolution of the correction process is about 4 mm. It is the full width at half maximum (FWHM) of the resistivity distribution produced by thermal neutrons irradiation of Si wafer through a 3 mm hole in the Cd-mask.

Key words: silicon wafer • thermal neutron doping • silicon resistivity homogeneity • silicon resistivity heterogeneity • neutron transmutation doping (NTD) • selective neutron transmutation doping (SNTD) • MARIA nuclear research reactor • C-V measurement

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Introduction

The homogeneity of the electric resistivity of silicon wafers is one of the most important characteristics, which, however, is difficult to attain with high precision on the high-resistivity silicon wafers using standard production methods, including the NTD method. Correction of resistivity distribution on such wafers is needed for many applications. Among them there are silicon detectors, which perfectly suit the pulse-shape analysis method, e.g. for nuclide identification, provided that resistivity is distributed uniformly with heterogeneity not exceeding 1%. Price of silicon wafers critically depends on the scale of fluctuations in resistivity distribution.

Selective neutron transmutation doping of silicon-technique

Neutron transmutation doping of silicon ingots for correction of their resistivity distribution is well known [2]. New application of this method was proposed by A. J. Kordyasz [3, 4] as the selective neutron transmutation doping (SNTD), for correction of resistivity distribution of high resistivity silicon wafers. This method applies the NTD process at selected regions of the silicon wafer and is based on the well known transmutation reaction:

$$ {}^{30}_{14}\mathrm{Si}(\mathrm{n},\gamma)\,{}^{31}_{14}\mathrm{Si} \xrightarrow[2.624\mathrm{h}]{\beta^-} {}^{31}_{15}\mathrm{P} \quad (1)$$

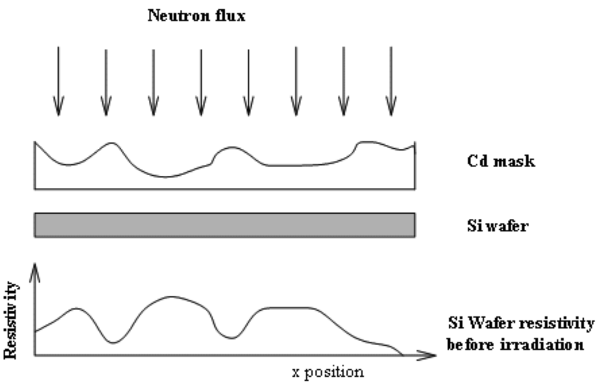

Fig. 1. Selective neutron transmutation doping (SNTD) technique with Cd-mask, schematically.

The idea of SNTD is illustrated in Fig. 1. Neutron-induced transmutation of Si-atoms into the P-donors changes the electric resistivity of silicon. Cadmium is known to be an effective neutron shield. In the presented method, the initially uniform thermal neutron flux from a nuclear reactor is modified by a Cd-filter of thickness mechanically drilled (upper part of the figure) to produce the appropriate non-homogeneous neutron flux capable of smoothing up the non-uniform resistivity distribution of the silicon wafer (lower part of the figure). Transmutation level N_{NTD}, i.e. the concentration of ^{31}P due to NTD, depends on the neutron fluency (time integral of neutron flux) applied:

$$N_{\mathrm{NTD}} = N\sigma_0 \int \varphi \, dt \tag{2}$$

where: N – density of ^{30}Si nuclei; σ_0 – thermal neutron capture cross section for reaction (1), φ – thermal neutron flux; t – time. In general, the neutron flux may vary during irradiation and on-line flux monitoring is necessary. In the MARIA reactor there is a self-powered neutron detector which is used to measure the neutron flux in such cases.

On the other hand, the N_{NTD} level required to change the specific resistivity of silicon specimen from initial ρ_{in} to target value ρ_{out} depends on the initial type of conductivity (donor/acceptor) and is given by:

$$\begin{aligned} N_{\mathrm{NTD}} &= \frac{1}{\rho_{\mathrm{out}} e \mu_e} - \frac{1}{\rho_{\mathrm{in}} e \mu_e} \quad \text{for n-type silicon} \\ N_{\mathrm{NTD}} &= \frac{1}{\rho_{\mathrm{out}} e \mu_e} + \frac{1}{\rho_{\mathrm{in}} e \mu_p} \quad \text{for p-type silicon} \end{aligned} \tag{3}$$

where: e – electron charge; μ_e, μ_p – mobility of electrons and holes respectively.

To get appropriate correction of the specific resistivity one has to calculate the transmutation level using formula (3) and apply the thermal neutron fluency specified by (2).

The SNTD process was successfully tested by the irradiation of silicon wafer by thermal neutrons through a Cd neutron filter [3]. The whole silicon wafer doping system shown in Fig. 2, was designed for the MARIA reactor, where the experiment was carried out. The reactor facility used for commercial NTD of silicon ingots has been adapted for that purpose. Specially constructed distance tube, set in the reactor core, was designed to get a more homogeneous and isotropic neutron beam. There are two parameters important for silicon NTD: the average density of thermal neutron flux and neutron flux homogeneity within the silicon. First parameter determines the time of silicon irradiation according to formula (2). This parameter was measured by a sequence of irradiations of Al-Au (0.1%) and Al-Co (0.1%) foil detectors, on the top of distance tube (see Fig. 2). Average density of thermal neutron beam on the top of irradiation channel is approximately 2×10^{12} $\mathrm{n \cdot cm^{-2} \cdot s^{-1}}$ with the MARIA reactor operating at 20 MW thermal power. Second parameter, thermal neutrons density distribution, determines the quality of the silicon wafers irradiation process. Every two irradiated points, not covered by neutron mask, after all should have a similar fluency to set homogeneity of silicon doping. Measurement of this parameter was done by the irradiation of the set of 37 Al-Au 0.1% detectors regularly located on the target surface. Maximum deviation of thermal neutrons density distribution was 17% (Fig. 3). Further improvement of neutron field homogeneity has been achieved by rotation of silicon wafers during irradiation.

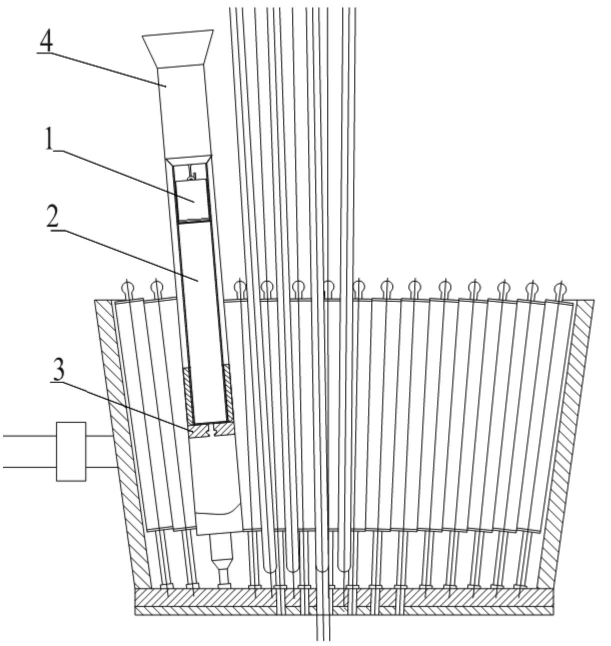

Fig. 2. Experimental setup in the MARIA nuclear research reactor core. 1 – container with silicon wafer and Cd-mask; 2 – distance tube; 3 – graphite cork; 4 – NTD of silicon ingots assembly.

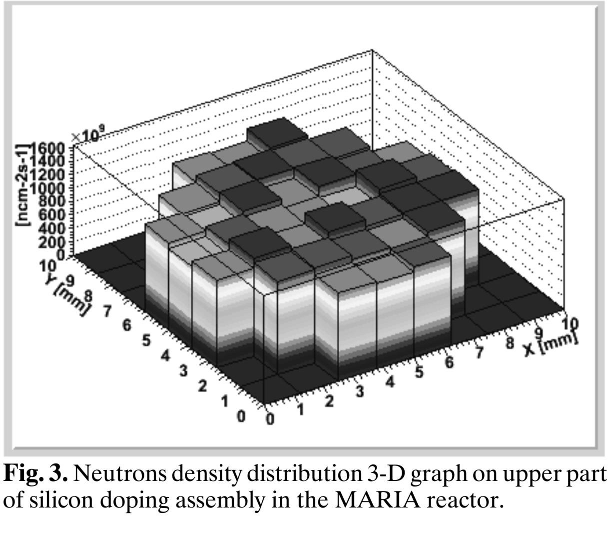

Fig. 3. Neutrons density distribution 3-D graph on upper part of silicon doping assembly in the MARIA reactor.

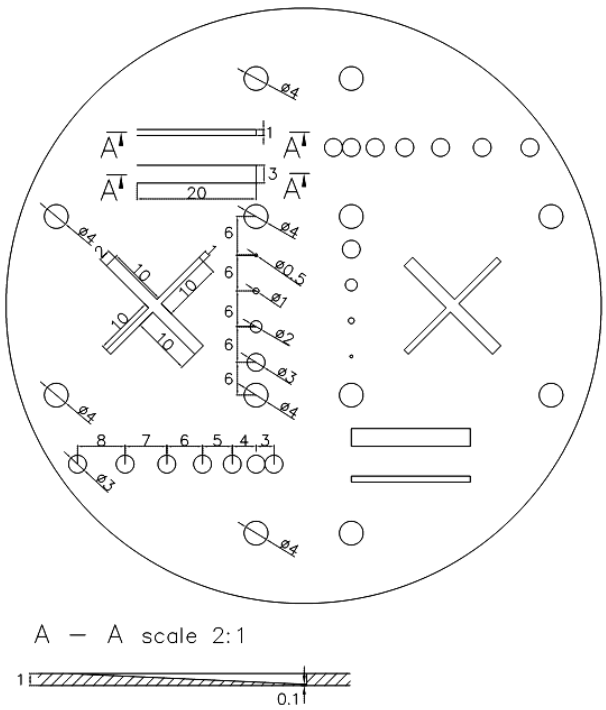

Fig. 4. Cd-mask made from a 1 mm thick cadmium sheet with open holes, and rectangular openings, and Cd wedges with a minimal thickness of 0.1 mm. Sizes are in mm.

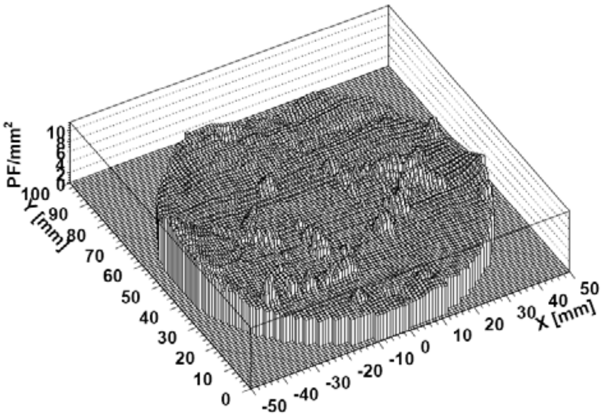

Fig. 5. Capacitance distribution of the silicon wafer after irradiation by thermal neutron flux through Cd-mask in the MARIA reactor (interrelation between the capacitance and the electric resistivity is given by Eq. (4)).

Silicon wafer, closed in a special container, was covered by the thermal neutrons absorbing, cadmium mask 1 mm thick, which is shown in Fig. 4. For research purposes, circular holes and crosses have been drilled in the mask, to let neutrons penetrate the silicon wafer where target resistivity was expected to be lowered. The main goal of this research was to measure how the electric resistivity changes depending on the distance between holes, size of holes, broadness of rectangular openings and thickness of the neutron mask.

Measurement of resistivity distribution of Si wafer

Silicon wafer used in this research has been manufactured at the Institute of Electronic Materials Technology (ITME, Warsaw, Poland). It was made from n^+-type substrate layer, antimony doped, with an average resistivity of 0.01 Ω·cm, 400 μm thickness and a high resistivity n-type epitaxial layer, with an average resistivity of 4000 Ω·cm and 128 μm thickness. We had just this silicon wafer to carry out the experiment.

Time of silicon wafer irradiation was 9.4 h and the average thermal neutron beam density reached 1.7×10^{12} $\mathrm{n \cdot cm^{-2} \cdot s^{-1}}$. Measured irradiation fluence was 5.8×10^{16} $\mathrm{n \cdot cm^{-2}}$ and it has caused a resistivity change of the silicon wafer from approx. 4000 Ω·cm to approx. 700 Ω·cm. Complete annealing of radiation damage took place at elevated temperatures in a dry nitrogen atmosphere. Due to annealing, we have ground to expect, that the impact of structural defects on the results can be neglected. After annealing, capacitance of silicon wafer has been measured using the C-V method [5, 6], by bias Shottky diode formed by a mercury drop gravitationally pressed to the tested silicon surface. Measured capacitance resolution was 1 × 1 mm. The standard method has been used to determine resistivity from measured capacitance *C* with silicon dielectric constant [6] as a function of depletion layer thickness. Each point of the silicon wafer resistivity was calculated by the modified formula (1) of Ref. [1]:

$$\rho = \frac{0.68445}{C^2 \varepsilon_r \mu_e (V_0 + V)} \tag{4}$$

where ρ is the electric resistivity, *C* – measured capacitance, ε_r – silicon dielectric constant, μ_e – majority-carrier mobility (electrons), V_0 – Hg-Si barrier potential and *V* – applied bias potential.

A complex pattern of the Cd-mask, has been designed to check the surface resolution of the SNTD method. Result of the silicon wafer irradiation through Cd-mask is shown on the capacity diagram in Fig. 5. There are characteristic differences in silicon capacitance between places covered by the mask and places under holes in the mask. The results and comparison of the measured resistivity has been illustrated in further pictures.

Results of irradiation

The resistivity distribution along the selected cross sections of wafer surface seems to be the most convenient way to demonstrate the resolution of resistivity correction method. Analysis of first irradiation pattern is shown in Fig. 6. The irradiation of silicon through a 3 mm hole in the Cd-mask lowers the resistivity of the material down to about 300 Ω·cm. It was presented by the first-from-the-left peak which is produced by irradiation through the first-from-the-left hole in the Cd-mask. The FWHM of this peak is about 4 mm. For this reason, the peaks produced by last four holes overlap due to short distances between them (5, 4, 3 mm). Fig. 7 presents the modification of silicon wafer due the neutron irradiation through the holes with different diameters (4, 3, 2, 1, 0.5 mm). The FWHM for resistivity under holes with a diameter of 3 and 4 mm is almost the same, about 4 mm. The holes of the diameter below 2 mm almost do not modify the wafer resistivity. In order to test silicon resistivity as a function of the thickness of Cd-filter, the Cd wedge of 20 mm length

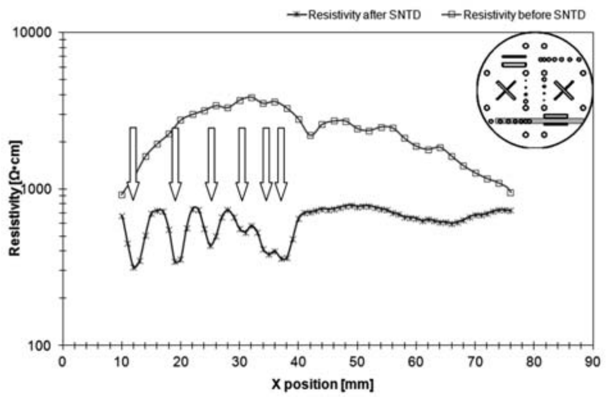

Fig. 6. Resistivity graph of silicon wafer section irradiated through neutron mask with 3 mm holes size in Cd-mask (line with crosses). Data for the analysis were taken along horizontal gray line marked on the Cd-mask design, (see the right upper corner of the picture). Distances (in mm) between centers of holes are 7, 6, 5, 4 and 3, respectively. (Unfortunately, the data for the most left 3 mm hole are not available). Arrows illustrate position of holes in the mask. Line with open squares is the wafer resistivity before neutron irradiation.

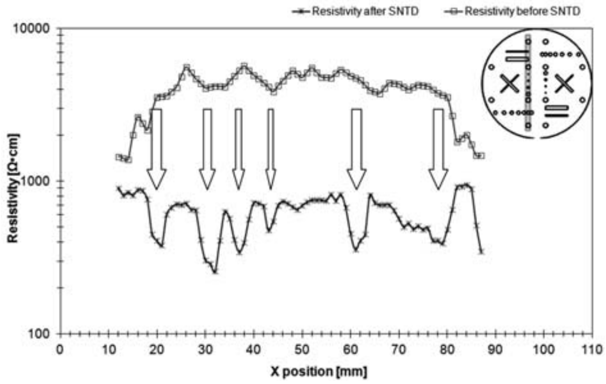

Fig. 7. Resistivity graph of silicon wafer irradiated through neutron mask with 4, 3, 2, 1, 0.5 mm holes in Cd-mask (line with crosses). Data for the analysis were taken along vertical gray line marked on the Cd-mask design, (see the right upper corner of the picture). The second minimum from the left is produced by a pair of coupled 3 mm holes in the mask. Distances (in mm) between middle of the holes are 6 mm. Arrows illustrate position and thickness of holes in the mask (only for hole diameters: 4, 3, 2 mm). Holes with diameters 1 and 0.5 mm do not produce any noticeable changes for resistivity of silicon. Line with open squares is the wafer resistivity before neutron irradiation.

and 3 mm wide was created in the Cd-mask, (Fig. 4). Unfortunately, we have not observed any meaningful dependence of silicon resistivity on Cd-thickness. The next two figures (Figs. 8 and 9) present the resistivity modification by the neutron irradiation through crossed rectangular openings of 1 and 2 mm width, respectively. The FWHM of resistivity distributions for both slots is about 3 mm. After neutron irradiation of the silicon through 1 mm thick Cd-sheet, the average resistivity was reduced from about 4000 to about 700 Ω·cm.

Discussion and conclusion

The preliminary test of systematic modification of silicon resistivity by the SNTD method has been presented.

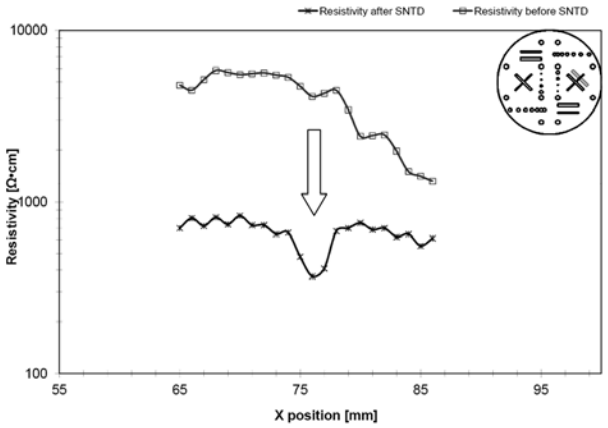

Fig. 8. Resistivity graph of silicon wafer irradiated through neutron mask with a 1 mm gap localized perpendicular to the gray line marked on the Cd-mask design, (see the right upper corner of the picture).

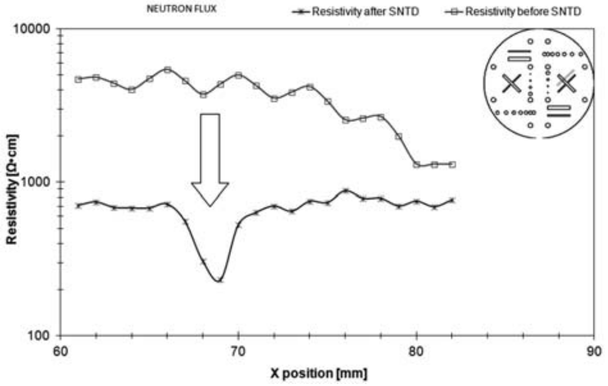

Fig. 9. Resistivity graph of silicon wafer section irradiated through neutron mask with a 2 mm gap localized perpendicular to the gray line marked on the Cd-mask design, (see the right upper corner of the picture).

Selective (local) reduction of the silicon wafer resistivity from about 700 to about 300 Ω·cm by neutron irradiation through the 3 and 4 mm diameter holes and 1 and 2 mm slots in 1 mm thick Cd-mask was demonstrated. At the present stage of investigation, the resistivity distribution FWHM connected with 3 mm, and 4 mm diameter holes was about 4 mm. Thermal neutron irradiation of silicon through small diameter holes (0.5, 1, 2 mm) comparable with Cd-mask thickness does has no meaningful impact on the silicon resistivity. For this reason, the value (FWHM = 4 mm) can be treated as the spatial resolution parameter of this experiment. For 2 and 1 mm wide gaps in Cd-mask, the FWHM is about 3 mm. Our test of influence of the thickness of Cd-filter with the Cd wedge 20 mm long and 3 mm wide on modification of the silicon wafer resistivity was inconclusive, like we had with the neutron irradiation of silicon through the low diameter holes. To have reliable dependence of the electrical resistivity change on the Cd-layer thickness, one should use longer and broader wedge.

Next task is to explain the observed resistivity reduction of silicon wafer covered by an "undrilled" 1 mm thick Cd-sheet. This can by understood if we assume the impact of scattered neutrons entering the silicon wafer from the back. Another explanation of this phenomenon could be the activation of silicon by higher-energy (non-thermal) neutrons, such that the

Cd-sheet is unable to capture and protect silicon against neutron activation.

Our future task is the testing of other materials for the masks containing boron, which has "broader" cross section for the neutron capture.

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