

## Radiation Resistance Studies of PIN Diode Detectors Irradiated with Heavy Ions

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The controlled destruction of the PIN diode detectors, SIEMENS SFH 870/F170 and SFH 871/F171, by the 35 MeV beam of the  $^{12}\text{C}$  and by 24 MeV of the  $^{14}\text{N}$ , respectively, was characterized using nuclear spectroscopy, the surface profile measurements, and the positron annihilation spectroscopy technique. The beam fluence was in the range of  $10^{12}$ - $10^{14}$  ions/cm<sup>2</sup>. It has been shown that the fluence of  $10^{12}$  ions/cm<sup>2</sup> of the  $^{12}\text{C}$  beam did not allow it to destroy the PIN diode detector. For this purpose, one needs the fluence of at least  $4 \times 10^{12}$  ions/cm<sup>2</sup> for the  $^{14}\text{N}$  ions beam and  $2.2 \times 10^{13}$  ions/cm<sup>2</sup> for the  $^{12}\text{C}$  ions one. The presence of divacancies in the irradiated sample was detected by the positron lifetimes measurements, with the fraction significantly higher for the  $^{12}\text{C}$  implanted sample. Furthermore, it was found that the surface roughness changed drastically following the implantation, i.e., the arithmetic average of profile height deviations from the mean surface of the  $^{14}\text{N}$  beam implanted sample is significantly higher than of that irradiated with the  $^{12}\text{C}$  ions and the reference one, and the surface average roughness was about 2–3 times higher.

topics: heavy ion irradiation, PIN diode detector, positron lifetime spectroscopy

### 1. Introduction

The PIN diode is a type of detector with a wide and undoped intrinsic semiconductor region of high resistivity placed between the strongly doped p-type and n-type layers. Such detectors are suitable for nuclear physics applications as they are capable of registering heavy ions (e.g., the charged particle detection systems in the Coulomb excitation experiments). Apart from that, PIN diodes are widely used in typical electronic elements, such as attenuators, fast switchers, photodetectors, and some other high-voltage power technologies. However, heavy-ion detectors based on silicon material, including PIN diodes, deteriorate quickly when exposed to ionizing radiation. So far, this process has not been thoroughly studied, leaving many open questions regarding the character of the damages as well as the resistance of the PIN diodes to such exposure. It has been shown that in the case of irradiation with the gamma quanta, PIN diodes get damaged due to exposure to the Compton electrons (of an energy of

about 1 MeV), which can produce impurity-related defects in the detector crystal lattice, as discussed in [1]. The radiation hardness of the PIN diodes has been a subject of the studies of B. Abi et al. [2]. In their paper, the authors claim that no sign of damage was found in the PIN diodes after the gamma irradiation with a total dose 10 Mrad [2]. Furthermore, no sensitivity to the gamma radiation with a total dose of 600 Mrad was observed for the oxidized silicon detectors as well [1]. Above such a dose, some single-volume defects of the silicon crystal lattice are formed [3].

The PIN diodes were also irradiated with a proton beam up to their complete damage, which was observed at the fluence of  $10^{15}$  particles/cm<sup>2</sup> [1], and recorded by the Open-air Optical Pathway method [2]. These measurements showed that the PIN diodes lose about 90% of their spectroscopic properties [1]. In the case of positively charged particles, due to the Coulomb interaction, some partially defective clusters and partially isolated single vacancies are created in the silicon crystal lattice.

Therefore, even with the same amount of non-ionizing energy loss in silicon, the defected structures caused by different radiation particles might be very different, as discussed in [3].

In [4], V. Sopko et al. study the PIN diode energy traps created by neutron radiation. The detectors were irradiated with the neutrons emitted from the  $^{252}\text{Cf}$  source up to the dose of 8 Gy. Depending on the energy levels, several types of damage caused by neutrons have been identified by deep-level transient spectroscopy [5]. The results show that vacancies, vacancy complexes, or complexes with the interstitial atoms, i.e., impurities were present in the irradiated PIN diodes.

The PIN diode detectors were used in the charged particle detection systems in the Coulomb excitation experiments, and it was shown that such detectors lose their excellent spectroscopic qualities after exposure to heavy ion beams. M. Matejska-Minda et al. [6] noticed a qualitative deterioration of spectroscopic properties of the PIN detectors after 5 days of irradiation with the 70 MeV  $^{32}\text{S}$  ion beam, impinging on the detector after scattering on the  $^{45}\text{Sc}$  target [6]. Following that observation and the studies reported in [7], three types of PIN diodes based on their spectroscopic properties were selected by us to explain why such an effect occurs. It was found that these effects were related to the change in the crystal structure of the silicon that followed exposure to the heavy ion flux. Thus, taking into account that the incomplete registration of the scattered ions could affect the collected physical data due to the decreased charge collection efficiency of the particle detector (and efficiency of particle detection by the radiation-damaged detectors), the information on the PIN-diode detector destruction point is vital for the Coulomb excitation experiments. In our recent studies [8], detectors with a thickness of  $380 \pm 15 \mu\text{m}$  were irradiated with a flux of  $^{12}\text{C}$  and  $^{14}\text{N}$  ions. The carbon beam bombarded a gold foil of  $9 \text{ mg/cm}^2$  thickness. After scattering, the 35 MeV  $^{12}\text{C}$  projectiles were implanted in a PIN diode placed in the laboratory frame, at an angle of 30.2 degrees with respect to the beam direction. Experimental details were presented in [5]. In the other experiment, the nitrogen beam was scattered on the  $^{110}\text{Cd}$  target foil of a thickness of  $2 \text{ mg/cm}^2$  and registered in the PIN diode placed in the laboratory frame, at an angle of 113 degrees with respect to the beam direction. The mean energy of the implanted  $^{14}\text{N}$  ions was equal to 22 MeV. The spectroscopic properties of the irradiated detectors were monitored using the alpha source of  $^{241}\text{Am}$ .

In the present paper, further experimental studies of PIN diodes irradiated with  $^{12}\text{C}$  and  $^{14}\text{N}$  ions are presented. The impact of the forementioned ions on the changes generated on the surface and in the structure of the detection material is investigated using the optical profilometry method and positron annihilation spectroscopy, respectively. The first

method allows for precise material surface characterization, while the second one is a sensitive technique for the detection of open-volume defects, such as vacancies and their clusters. Many studies proved that this is an effective tool for detecting irradiation-induced defects [9, 10].

## 2. Heavy ion irradiation results

The PIN diode detector of  $380 \pm 15 \mu\text{m}$  thickness (SIEMENS SFH 870/F170) with an active area of  $1 \text{ cm}^2$  was irradiated with a  $^{12}\text{C}$  beam delivered by the U-200P cyclotron at the Heavy Ion Laboratory, University of Warsaw, Poland (HIL). The beam was scattered on a  $9 \text{ mg/cm}^2$  thick gold target. After scattering, the beam of 35 MeV energy fully irradiated the PIN diode material with the fluence at  $2.2 \times 10^{13} \text{ ions/cm}^2$ . The number of ions impinged on the PIN diode detector was monitored using a collimated silicon detector placed at 22.5 degrees with respect to the beam axis. Saturation of the dark current<sup>†1</sup> was observed at the fluence value of  $2 \times 10^{13} \text{ ions/cm}^2$ . In the experiment, the spectroscopic properties, i.e., full width at high maximum (FWHM), peak position, and dark current of the PIN diode detector irradiated with a heavy ion beam, were frequently monitored by measuring the energy spectrum collected off-beam using the  $\alpha$  particles emitted from the  $^{241}\text{Am}$  radioactive source, as presented in Fig. 1.

The spectrum presented in panel (b) of Fig. 1 corresponds to the undamaged detector with the less than  $0.05 \mu\text{A}$  dark current. The  $\alpha$  particle peak from the  $^{241}\text{Am}$  source is clear and high, and FWHM is narrow. The peak centroid (the mid-position of the peak) is measured at the energy of  $\approx 5.31 \text{ MeV}$ . A smaller peak of lower energy is visible next to the main one, resulting from the registration of the radiation near the undamaged detector edge/frame region, where the charge collection is changed due to the presence of the thin strip of the material responsible for the electrical contact. This detector is considered a reference one, characterized by the best spectroscopic qualities. The spectrum presented in panel (c) shows the moment when the PIN diode got slightly damaged after receiving the radiation with the fluence of about  $6 \times 10^{12} \text{ ions/cm}^2$ , and the dark current reached the value of  $2 \mu\text{A}$ . The peak height became significantly lower than the one registered in the reference detector (b). The small peak, previously present on the left to the main one, is no longer distinguishable. However, the FWHM is compared to the peak presented in panel (b), and its centroid is measured in the same place as in the reference detector. The spectrum shown in panel (d) presents the detector significantly damaged after receiving the  $^{12}\text{C}$  radiation of  $1.4 \times 10^{13} \text{ ions/cm}^2$ .

<sup>†1</sup>Dark current is an unwanted current in a photodetector

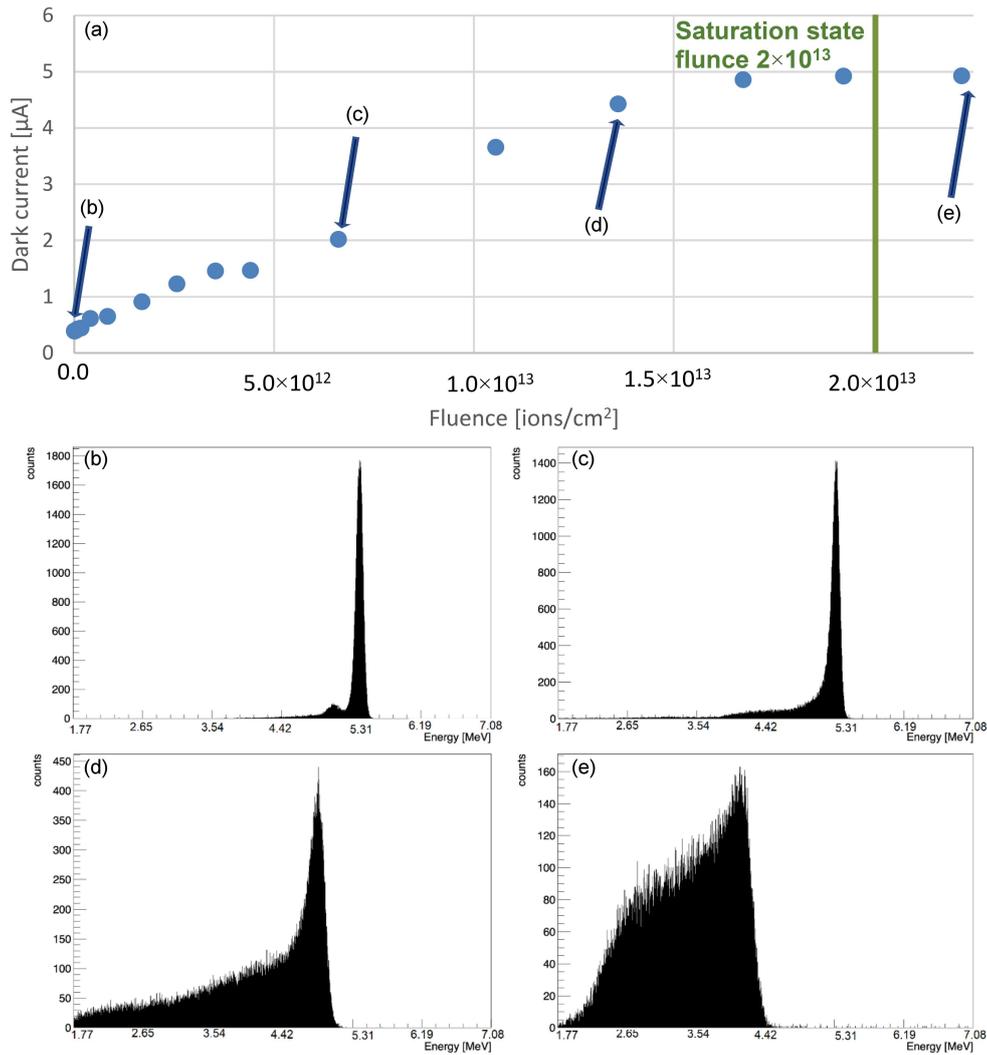


Fig. 1. Fluence-dependent evolution of PIN diode dark current measured in the PIN diode detector irradiated with the  $^{12}\text{C}$  beam (a). The  $^{241}\text{Am}$  alpha particle spectra obtained in different stages of the detector destruction process (b–d), collected immediately after cutting the beam flux off, compared to the spectrum collected for the final dark current saturation level (e).

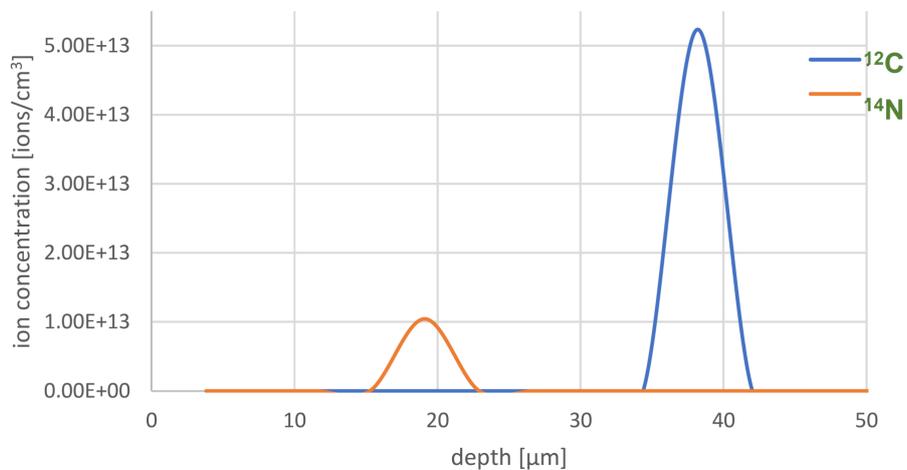


Fig. 2. Depth and the ion concentration for the  $^{12}\text{C}$  and  $^{14}\text{N}$  ions with the energy of 35 MeV and 24 MeV, respectively, implanted into the PIN diode material, calculated using the SRIM code [11].

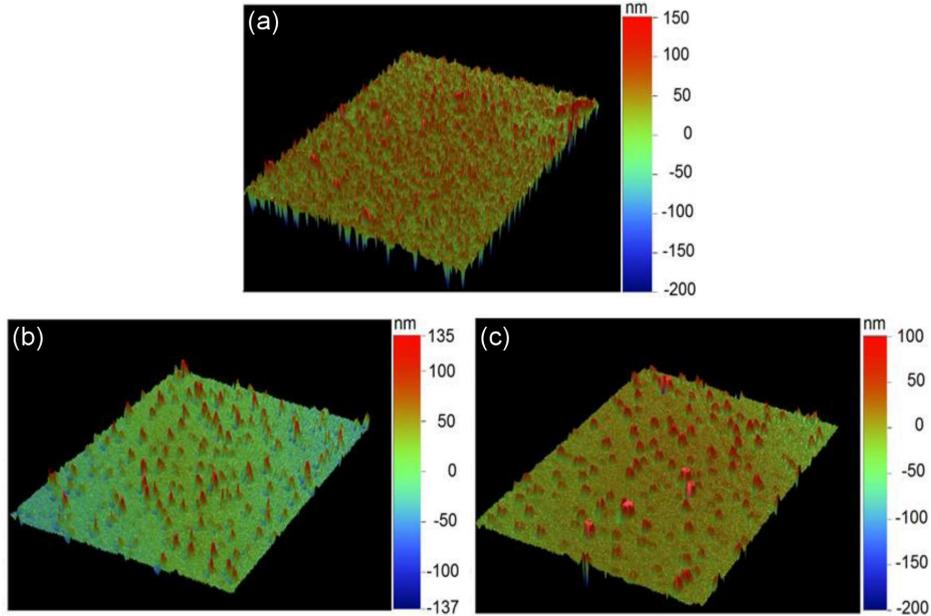


Fig. 3. Typical surface profiles of the PIN diodes with the surface size of  $118.3 \times 157.7 \mu\text{m}^2$ : (a) PIN diode after implantation with nitrogen ions, (b) reference PIN diode, (c) PIN diode after implantation with carbon ions.

The peak height is drastically lower than in reference detector (b), and the dark current reached  $4.4 \mu\text{A}$ . The low-energy peak tail is now visible. The FWHM is visibly broadened compared to the previously registered spectra. The centroid has moved to the energy  $\approx 4.78 \text{ MeV}$ . The spectrum presented in panel (e) shows a completely destroyed PIN diode. The peak height is much lower than that presented in spectra (b)–(d), and the quality of the spectrum makes it impossible to read the peak energy and FWHM. The dark current reached  $5 \mu\text{A}$  at the fluence of  $2.2 \times 10^{13} \text{ ions/cm}^2$ . The centroid of the wide structure was shifted to the energy  $4.25 \text{ MeV}$ .

During one of the Coulomb excitation experiments performed at HIL, the PIN diode detector of the  $380 \pm 15 \mu\text{m}$  thickness (SIEMENS SFH 871/F171) with an active area of  $0.5 \times 0.5 \text{ cm}^2$  was exposed to the  $^{14}\text{N}$  beam of  $24 \text{ MeV}$  energy measured after scattering on the  $2 \text{ mg/cm}^2$  thick  $^{110}\text{Cd}$  target. The detector was positioned at  $113$  degrees with respect to the beam axis and exposed to the radiation for 4 days. The saturation state of the dark current corresponding to the complete damage of the detector was estimated at the fluence greater than  $4 \times 10^{12} \text{ ions/cm}^2$ . The ambiguity in the fluence determination is caused by the fact that this PIN diode was already used in the previous experiments, and therefore we cannot tell exactly what the total radiation dose and the kind of radiation that could impact the spectroscopic quality of this detector are. However, the described PIN diode exhibits clear signs of radiation damage after being used in the aforementioned specific Coulomb excitation physical experiment.

TABLE I

The surface average roughness ( $S_a$ ) of the reference sample and of samples implanted with nitrogen and the carbon beam.

PIN diodes	$S_a$ [nm]
Reference	20.48
Implanted with $^{12}\text{C}$	14.51
Implanted with $^{14}\text{N}$	49.04

Since both  $^{12}\text{C}$  and  $^{14}\text{N}$  ions had different incoming energies, the penetration depth in the PIN diode material was different for each of them. Based on the calculations performed using the Stopping and Range of Ions in the Matter (SRIM) code [11] for both beams, we conclude that the carbon ions reached a depth of  $\approx 38 \mu\text{m}$  while the nitrogen ions only  $\approx 18 \mu\text{m}$  (Fig. 2). The energy of nitrogen ions beam is about 30% lower than that of the carbon one. This corresponds to half the penetration depth and about one order of magnitude lower saturation flux compared to the case of the carbon ion beam, completely destroying the detector.

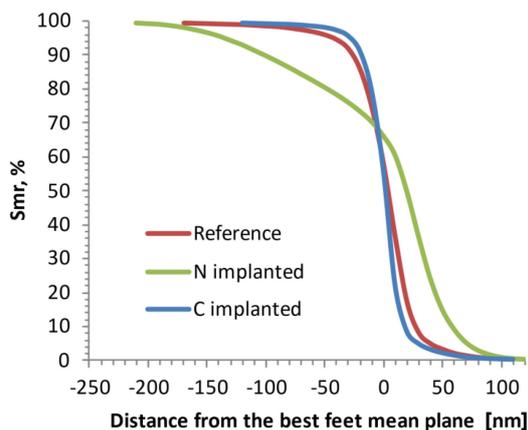
### 3. The surface profile

The Wyko NT9300 optical profiler by Veeco, New York, NY, USA, was used for the surface roughness measurement. The three-dimensional images of the surface, characteristic of the tested materials, are shown in Fig. 3. The surface roughness was characterized by the average height of the surface

TABLE II

The results from the positron annihilation lifetime spectroscopy.

Ions	$\tau_1$ [ps]	Intensity <sub>1</sub> [%]	$\tau_2$ [ps]	Intensity <sub>2</sub> [%]	$\tau_3$ [ps]	Intensity [%]	Fluence [ions/cm <sup>2</sup> ]
<sup>12</sup> C	127 ± 2	11	310 ± 2	9	220	80	2 × 10 <sup>13</sup>
<sup>14</sup> N	108 ± 2	7	340 ± 2	4	220	89	> 4 × 10 <sup>12</sup>
Unimplanted diode	–	–	–	–	220	100	–
pure Si	–	–	–	–	220	100	–

Fig. 4. Areal material ratio of the scale limited surface ( $S_{mr}$ ) values for the investigated samples.

profile ( $S_a$ ) and the ratio of the area surface material at a specified height to the evaluation area ( $S_{mr}$ ). The average height of the surface profile can be defined as the arithmetic average of profile height deviations from the mean surface. The results are shown in Table I and Fig. 3.

Significantly greater roughness (i.e., in  $S_a$  value) of the sample implanted with nitrogen, compared to the two others, is visible. The areal material ratio curves of the specified highest area to the evaluated area ( $S_{mr}$ ) are shown in Fig. 4. This curve represents the amount of area with the specific or greater distance from the mean plane. One can see that the highest peaks and the deepest valleys distributions are similar for the reference sample and the <sup>12</sup>C-implanted one. This corresponds to similar  $S_a$  values for these samples (i.e., the difference in the  $S_a$  value of 6 nm does not seem to be significant and may be related to the diode production technology). However, the distribution for the <sup>14</sup>N-implanted specimen is different. First, the valleys are much deeper. Second, the peaks are higher, and their proportion is greater. This corresponds to a significantly higher  $S_a$  value registered for this sample.

The advantage of the valleys over the peaks is visible. Thus, a valley-type structure of the surface with relatively high peaks and deep valleys is very distinct for the reference and the carbon-implanted samples and much less pronounced for the nitrogen-implanted one.

#### 4. Positron annihilation spectroscopy results

The positron lifetime measurements were performed using the digital spectrometer consisting of an APU8702 unit and two BaF<sub>2</sub> detectors. The <sup>22</sup>Na positron source with the activity of 1 MBq, enveloped into a 5 μm titanium foil and placed between two samples was used. The first sample was always pure, non-defected silicon with a positron lifetime of 220 ps [12]. The second one was either the unirradiated PIN diode, the diode irradiated with nitrogen ions, or one irradiated with carbon ions. The positron lifetime spectra consisted of 3 × 10<sup>6</sup> counts. The spectra were deconvoluted using the LT program [13]. The subtraction of the background and the source components of the annihilation, as well as 50% positron annihilation in the nonirradiated Si sample, were taken into account in the analysis. The fraction of positrons annihilated in the irradiated areas (estimated on the basis of SRIM simulations) was calculated using the LYS-1 code [14]. It was 20% for the <sup>12</sup>C and about 11% for the <sup>14</sup>N implanted PIN diode. This was also taken into consideration in the spectrum analysis by fixing the intensity of positron lifetime component  $\tau_3$  for the nonirradiated part of the Si sample. In the case of an unimplanted diode, only one positron lifetime component exactly like for Si (i.e., 220 ps) was found. In turn, two positron lifetime components ( $\tau_1$  and  $\tau_2$ ) were registered in the irradiated areas (Table II). A lifetime greater than 300 ps indicates the presence of divacancies in the irradiated samples, as discussed in [15]. The shorter positron lifetime component, called the reduced bulk lifetime, reflects the existence of undamaged regions in the irradiated areas. The percentage fraction of positrons annihilated in the divacancies for the <sup>12</sup>C implanted sample is 45%, much higher than for the <sup>14</sup>N implanted sample, i.e., 36%.

#### 5. Conclusions

The experiment aiming at the controlled destruction of the PIN diode detector was performed at the Heavy Ion Laboratory, University of Warsaw, Poland, using the <sup>12</sup>C beam until the leakage current saturation limit was reached for the fluence of about 10<sup>12</sup> ions/cm<sup>2</sup>. The radiation damage of the detector was manifested in the observed alpha particle spectrum, collected off-beam using the <sup>241</sup>Am

source. The increase of the dark current was monitored constantly during the experiment. The full deterioration of the energy spectrum was observed at the saturation level of about  $5 \mu\text{A}$ ; however, the investigated detector was still able to measure the spectrum. In other words, the fluence of less than  $10^{12}$  ions/cm<sup>2</sup> did not allow it to destroy the PIN diode detector completely.

In this paper, two samples were investigated, one, never used before, irradiated with the <sup>12</sup>C beam in the dedicated controlled destruction measurement, as explained above, and the other, irradiated with the <sup>14</sup>N in the Coulomb excitation experiment. The damage characteristics of the irradiated PIN detector were investigated by positron annihilation spectroscopy. The divacancies were detected in the ion irradiated samples. The percentage fraction of the divacancies for the <sup>12</sup>C implanted sample is much higher than for the <sup>14</sup>N implanted one (i.e., about 45% and 36% of the positrons annihilated in the divacancies were detected in these samples, respectively).

The surface roughness of the implanted samples was measured using the optical profile method, and it was found to be significantly higher for the <sup>14</sup>N beam irradiated sample than for the <sup>12</sup>C beam implanted sample and the reference ones.

Further studies to allow monitoring the structural defects induced in the detector by the heavy ion irradiation are underway.

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