Channeling STIM analysis of radiation damage in single crystal diamond membrane

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Channeling STIM analysis of radiation damage in single crystal diamond

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ABSTRACT

The use of focused ion beam transmission channeling patterns to monitor the damage creation process in thin diamond singe crystal membrane is described. A 0.8 MeV proton beam from the RBI nuclear microprobe was used to perform Channeling Scanning Transmission Ion Microscopy (CSTIM) measurements. Damage was introduced in selected areas by 15 MeV carbon beam in range of fluences $3 \cdot 10^{15}$ – $2 \cdot 10^{17}$ cm^{-2}. Contrary to IBIC (Ion Beam Induced Charge), CSTIM is shown to be sensitive to the large fluences of ion beam radiation. Complementary studies of both IBIC and CSTIM are presented to show that very high fluence range can be covered by these two microprobe techniques, providing much wider information about the diamond radiation hardness. In addition micro Raman measurements were performed and the hegith of the GR 1 peak was correlated to the ion beam fluence.

Introduction:

Important advantage of the microprobe irradiation of materials to study processes of defect creation is its capability to damage only a small fragment of irradiated material or device. This is in particular important for valuable materials, such as diamond, that are difficult to study by bulk irradiation. In the case of diamond, influence of radiation damage has been studied previously using microprobe technique ion beam induced charge (IBIC) [1] that provides data about degradation of charge transport properties. Recently it has been have shown that thin diamond membranes (less than 10 microns thick) are much more suitable for such studies as effects of space charge (polarisation) are minimized [2, 3]. However, due to the almost complete degradation of electronic properties when fluence reaches certain value, other characterization techniques should be used. In attempt to monitor effects of diamond membrane irradiated by high ion beam fluence, we have explored the use of ion channeling that seems to be more applicable since it is more sensitive to significant degradation of the crystal lattice structure. Furthermore a channeling rate that corresponds to the radiation damage can be correlated quantitatively to the density of created defects. In order to minimize effects of irradiation during the measurements, Channeling Scanning Transmission Ion Microscopy (CSTIM) is used instead of the more conventional RBS channeling. CSTIM technique has been frequently used to study defects in crystals, starting with silicon [4, 5, 6, 7], and then other materials like SiGe [8] or HgCdTe [9] and others.

This technique is supposed to be sensitive to high damage does, whereas IBIC studies are sensitive to significantly lower damage fluences. Hence, complementary studies with both IBIC and CSTIM can cover rather large fluence range, providing more comprehensive information about the diamond radiation hardness. Since both are low current techniques, additional damage to the sample during the testing is minimal.

Experimental setup and results:

A new Channeling Scanning Transmission Ion Microscopy (CSTIM) system has been implemented at the microbeam scattering chamber of the Ruđer Bošković Institute (RBI) accelerator facility in Zagreb [10]. The microbeam setup of the RBI consists of a quadrupole focusing lenses used mostly as a high demagnification triplet, a sample manipulation stage and several different detectors with accompanying electronics. Sample manipulation stage has been reconstructed by addition of 2 rotation axes that were added on the existing linear axes (x,y,z). Rotation is achieved by using a closed-loop piezo positioners with controllers produced by SmarAct [11]. The specified angular resolution of the rotation movement is only 10 μ deg. However, as the final setup has no temperature stabilization this resolution is most likely not achieved. This is not essential since even the 1000 times lower angular resolution is still sufficient for the channeling measurements. Divergence of the focused beam should play more important role, as the width of the channeling peak is of the order of 1 deg. On the basis of object and collimator slits opening as well as depending on the system demagnification, the divergence of the focused beam used in measurements is estimated to be better than 0.1 deg.

In the CSTIM measurement, individual ions pass through the sample and their energy is measured by a silicon surface barrier detector or by a gas ionization chamber (only for heavier ions). Distance from the sample detector is fixed to 8 cm in the case of the standard setup with silicon detector. Since there was no additional collimation of the ions after the sample, resulting maximum scattering angle is dependent on the detector active area. Geometrical calculation yields maximum scattering angle of 25 mrad.

Signals from surface barrier detector were collected and amplified with a charge sensitive preamplifier Ortec 142A, followed by a shaping amplifier Ortec 570. Signal was than digitalized with Canberra 8075 ADC which was controlled by custom designed data acquisition software SPECTOR [12].

Ion that are channeled loose less energy in the membrane than ions in random orientation. As a result higher energy is recorded in the detector for the channeled ions. Channeling angle is found by making 2 axes angular scans and taking the median of the obtained energy spectra in each angle pair in order to obtain image as the one presented in Figure 1. Control of 2 motors for rotation was integrated in the SPECTOR software, making it much easier and faster to setup the experiment, to find the channeling angle, and to analyze the data after experiment.
A diamond membrane used in this experiment was fabricated from the optical grade single crystal (less than 1 ppm nitrogen concentration), <1 0 0> oriented CVD diamond produced by Element Six [13]. The sample was mechanically polished to a 30 μm thickness by AlmaxEasyLab [14]. Further thinning of the central portion of the sample to 3 μm was performed using an Ar/O plasma etching technique described in [15].
Diamond membrane was damaged using a 15 MeV carbon ion beam with the beam spot size of approximately 1 μm. 6 different square regions with dimensions 10 x 10 μm^2 were selected and irradiated in random orientation with different fluences. Since the range of 15 MeV carbon ions is higher than the membrane thickness, the variation of vacancy profile by depth is relatively homogeneous. The difference in number of vacancies, calculated using SRIM software [16], is less than 50% between the front and the back part of the irradiated membrane.
After the damaging process, the channeling experiment was performed using 0.8 MeV protons. By changing both angles in incremental steps and by calculating median of the recorded energy spectra, "channeling star" image (Figure 1.) has been obtained. From this image the channeling angle and position of the sample in the channeling direction can be determined. After the sample is oriented in the channeling direction, the beam is scanned in x,y directions and CSTIM areal image was obtained. These images can be used to map distribution of defects within the scanned area. Since all the data is saved, post processing can be used to extract spectra from the specific areas within the map. Thus, we can obtain the spectra for virgin part of the membrane as well as for all the different damaged areas of the diamond membrane. Examples of the extracted channeling and random spectra are presented in Figure 2.
Total energy loss of 0.8 MeV protons in this particular sample is 360 keV in random orientation and 300 keV in channeling orientation. When protons are passing through the partly damaged zone in the channeling direction, their total energy loss will be between these two values and will present an estimate of disorder of the crystal lattice. For the totally damaged sample, it should be the same as the measurement in the random orientation (to observe that, the damage should be higher than in this experiment). Therefore, we attribute the value 1 to represent the virgin crystal in channeled orientation and the value 0 to represent the random orientation. All partly damaged areas will have intermediate values. The normalization is performed by comparison of medians of energy shifts, damaged to random and virgin to random, respectively. Results of the measurements are displayed in this way and are shown in Fig 3. Previous measurements using the IBIC that were done on 5.6 μm thick diamond membrane using 1.3 MeV protons are presented for comparison on the same graph as well [3]. In order to be able to better compare these two measurements, the

number of created vacancies per volume is shown instead of the fluence. Number of vacancies was calculated using the SRIM software [16]. We can observe that the difference between damaged and undamaged spectra in this particular experiment shown on Figure 2. is rather small, but fitting spectra to Gaussian functions or taking the median yield reduce the uncertainty. In the case of results shown on Fig 3, the position of the centroid from the Gaussian fit has been used.
Additionally, the same sample has been tested using micro Raman measurements. Raman experiments were performed using HORIBA micro Raman device with 532 nm laser and grating with 600 nm/mm. Resolution of the laser focus was lower than 2 μm. The focus of the laser was aimed at the middle of the 10 x 10 μm^2 area. In the obtained spectra the height of the GR 1 peak has been observed [17]. The GR 1 peak is attributed to the diamond vacancy defect. The results are shown in the Fig 4. alongside with the CSTIM measurements. It can be seen that the peak increases with the fluence up to graphitization threshold when structure becomes too damaged and when there is no longer enough diamond structure to produce a diamond-vacancy peak.

Conclusions and future work

The existing RBI microprobe scattering chamber has been upgraded to include a CSTIM experimental setup. Our experimental results show that CSTIM technique can be used in wide range of applications and among others to evaluate quantitatively high radiation damage processes in single crystal diamond detectors where IBIC measurements can no longer give usable data. We conclude that IBIC and CSTIM, the two low current microprobe techniques are complementary. They can be used to study the high range of fluences and corresponding radiation damage that will influence properties of the single crystal diamond that may be important for a range of its applications. Since the detector collimation was not used, the technique sensitivity could be further improved in future work. This would come at the cost of having less ions arrive at the detector, so higher currents or longer measurement times would be needed, which would damage the sample more.

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Figures:

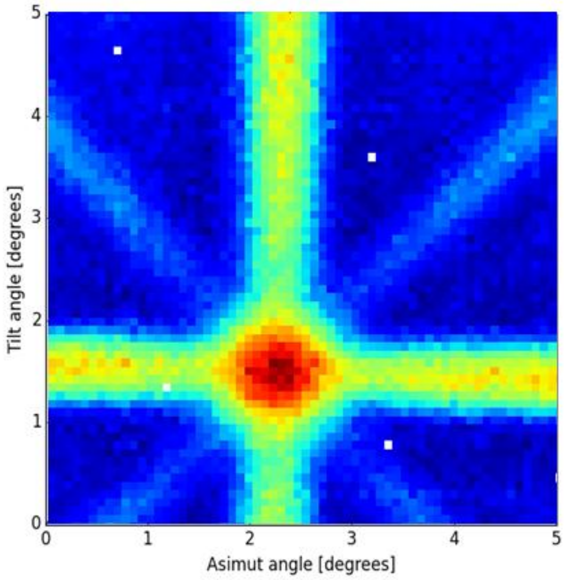

Figure 1. Channeling star image of the diamond. <1 0 0> orientation is seen in the middle. Planar channeling is also visible.

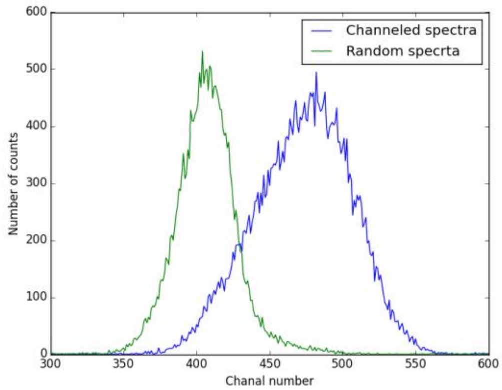

Figure 2. Spectra of random orientation and channeling orientation both damaged and virgin diamond membrane

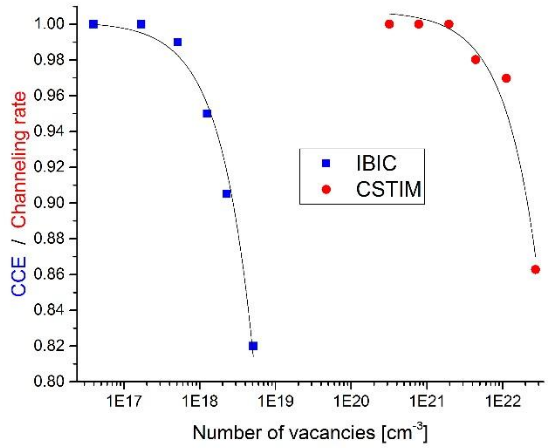

Figure 3. Charge collection efficiency and channeling rate as a function of number of created vacancies. Complementary techniques of IBIC vs. CSTIM is visible, large range of damages can be investigated using this two technique together.

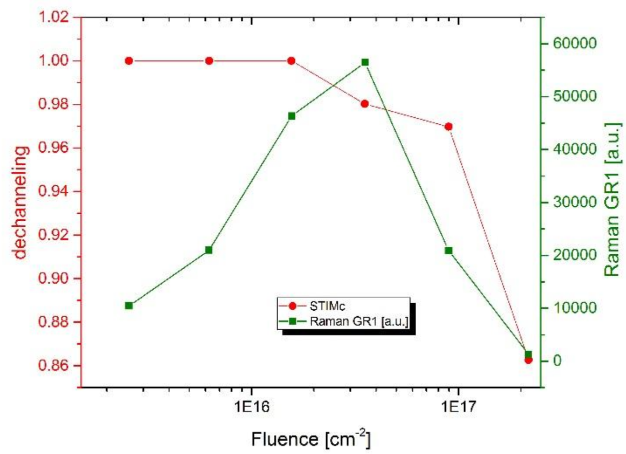

Figure 4. Channeling degradation and Raman GR1 peak of diamond membrane.