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## Effects of electronic and nuclear interactions in SiC

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### ABSTRACT

In this study, we performed irradiation experiments on nanostructured 3C-SiC samples, with 95 MeV Xe ions at room temperature. This energy permits the observation of the combined electronic and nuclear interactions with matter. The grazing incidence X ray diffraction results do not reveal a complete amorphization, despite value of displacement per atom overcoming the total amorphization threshold. This may be attributed to competing effects between nuclear and electronic energy loss in this material since a total amorphization induced by nuclear interactions was found after low energy ion irradiation (4 MeV Au).

Moreover, electronic interactions created by high energy ion irradiations induce no disorder in single crystalline 6H-SiC. But in samples previously disordered by low energy ion implantation (700 keV I), the electronic interactions generate a strong defects recovery.

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Keywords: irradiation, SiC, nanostructure, single crystalline, grazing incidence X ray diffraction, Rutherford Backscattering Spectrometry.

## INTRODUCTION

The aim of this paper is to study the structural evolution of silicon carbide under ion irradiation. Numerous studies have been already performed with low energy ions in order to observe the nuclear interaction effects on single 6H-SiC crystals. They have shown a rapid amorphization during irradiation at room temperature (RT) [1]. On the other hand, only few studies concern the electronic interaction effects. They indicate a very low disordering of virgin samples under high energy ion irradiation at RT and a defects recovery of predamaged samples under 827 MeV Pb irradiations [2].

Beside the peculiar properties of SiC, its elaboration as a nanostructured ceramic (NSC) is also worth studying because of the high grain boundaries density that is expected to provide an efficient source of traps for irradiation induced defects [3], but the enhancement of the behaviour under irradiation has still to be proven.

The present work is focussed on the structural evolution of NSC-SiC samples under irradiation at RT with 95 MeV Xe delivered by the IRRSUD line of the GANIL facility. These ions are similar to fission products and allow the observation of the combined electronic and nuclear interactions with matter. To study separately the nuclear and the

electronic interactions effects, the samples were irradiated with 4 MeV Au ions on the ARAMIS accelerator of the CSNSM and 910 MeV Xe ions delivered by the GANIL.

## EXPERIMENTAL

3C-SiC nanopowders with an average grain size of 10 nm were synthesized by the laser pyrolysis technique [4,5], which allows an accurate control of the granulometry and stoichiometry. Using these nanopowders, two ceramic samples were subsequently prepared by hot isostatic pressing (HIP) without sintering additives. The densification of the final NSC-SiC pellet is close to 92 % with a grain size after sintering around 36 nm.

The first pellet was irradiated at RT with 95 MeV Xe ions and a fluence of  $5 \times 10^{15} \text{ cm}^{-2}$  on the IRRSUD line of the GANIL facility. The sample was fixed on a water-cooled plate in order to minimize the heating of the sample under the ion beam. The second pellet was implanted at RT with 4 MeV Au ions and a fluence of  $5 \times 10^{14} \text{ cm}^{-2}$  on the ARAMIS accelerator of the CSNSM laboratory. The pellets were subsequently characterized by X-ray diffraction (XRD) grazing incidence X-ray diffraction (GIXRD) in order to probe the irradiated zone while avoiding the signal of the non irradiated part.

Three samples with dimensions of  $10 \times 10 \text{ mm}^2$  were cut from commercial single crystalline (0001)-oriented 6H-SiC wafers. Two of them were implanted at RT with 700 keV I ions at the CSNSM laboratory and two fluences were used ( $8 \times 10^{13} \text{ cm}^{-2}$  and  $1 \times 10^{15} \text{ cm}^{-2}$ ) in order to obtain two different damage levels. One sample was not implanted to preserve a virgin reference. The three samples were afterwards irradiated with 910 MeV Xe ions and a fluence of  $2 \times 10^{13} \text{ cm}^{-2}$  at RT on the SME line of the GANIL facility. Before and after each irradiation, the samples were analyzed by Rutherford backscattering (RBS) and RBS-C with 1.4 MeV  $\text{He}^+$  beam detected at a  $160^\circ$  scattering angle on the ARAMIS accelerator.

## RESULTS AND DISCUSSION

The results obtained from XRD on the nanostructured samples before irradiation for a probed depth of 2.5  $\mu\text{m}$  (fig.1) clearly show the cubic phase of SiC. The slight contribution at  $2\theta = 34.2^\circ$  could be attributed to the hexagonal phase. This contribution is in fact the consequence of stacking faults in some crystallites of the starting nanopowders [6]. Fig.1 also exhibits the typical peak broadening coming from the nanometric size of the crystallites [7,8].

The samples were irradiated with 95 MeV Xe ions at RT. The SRIM 2003 simulation (fig.2) shows the number of displacements per atom (dpa) in function of depth for a Xe fluence of  $5 \times 10^{15} \text{ cm}^{-2}$ . At this fluence, the number of dpa is higher than the amorphization threshold established for the single-crystalline SiC and found in the 0.2-0.5 dpa range at RT [1]. After irradiation, the diffraction peaks ascribed to the 3C-SiC can still be observed on the XRD diagrams (fig.1). No amorphization could be detected, as the diagrams appear very similar to those recorded before irradiation. According to SRIM 2003 simulation, the Xe fluence used is ten times higher than necessary for the amorphization, so if one takes into account the damaging mechanisms observed with low energy ions (i.e. mainly with nuclear interactions) the material should be amorphous on the whole ion pathway. The XRD diagrams indicate that the irradiated SiC is at least partially crystalline. Then, a question arose about the reason for this absence of amorphization: is it due to a nanostructuring effect or an electronic interactions effect?

In order to avoid the electronic interactions, the second sample was implanted at RT with low energy ions (4 MeV Au) and a fluence of  $5 \times 10^{14} \text{ cm}^{-2}$ . At this energy, the nuclear stopping power is higher than the electronic one, according to SRIM 2003 simulation. The GIXRD diagrams (fig.3) recorded before and after irradiation show a clear peak broadening, an intensity decrease and an increase of the background which reveal an amorphization of the

sample. It means that the absence of amorphization observed after 95 MeV Xe irradiation is not due to a nanostructuring effect, but may be caused by electronic excitations effect.

In order to study the electronic excitations effect on SiC, single crystalline 6H-SiC samples were irradiated at RT with high energy ions (910 MeV Xe) delivered by the SME line of the GANIL. According to SRIM 2003 simulation (fig.4), the nuclear stopping power is negligible compared to electronic stopping power at this energy in the region analyzed by RBS. The RBS spectra (fig.5) recorded in channeling geometry do not exhibit significant difference before and after irradiation with a fluence of  $2 \times 10^{13} \text{ cm}^{-2}$ . This high energy ion irradiation appears not to induce disorder in SiC.

In order to understand the electronic excitations effect on disordered samples, two SiC samples were first disordered by low energy ion implantation (700 keV I) at RT with  $8 \times 10^{13} \text{ cm}^{-2}$  fluence. The RBS spectra recorded before and after iodine irradiation in random and channeling geometry are reported on the figure 6. The broad peak on the RBS-C spectra after irradiation is due to the disorder induced by the iodine irradiation. The spectra analysis (not shown here) gives a maximum disorder rate around 60%. Afterwards, the disordered sample was irradiated with high energy ions (910 MeV Xe) at RT. The RBS-C spectra recorded after Xe irradiation (fig. 6) exhibits a strong decrease of the disorder peak. After irradiation with a Xe fluence of  $2 \times 10^{13} \text{ cm}^{-2}$ , the maximum disorder rate obtained by the RBS-C spectra analysis (not shown here) is around 12% only. This means that the electronic interactions induce a strong disorder recovering at RT. This disorder recovering effect induced by electronic interactions explains the absence of amorphization under the 95 MeV Xe ions irradiation. At this latter energy, there is a combination of electronic and nuclear interactions. The electronic interactions seem to recover the defects induced by the nuclear interactions and prevent the disorder accumulation in the sample.

This experiment was repeated with an higher iodine fluence ( $1 \times 10^{15} \text{ cm}^{-2}$ ) in order to create an extended total amorphous layer in the SiC sample. The RBS spectra recorded in random and channeling geometry are reported on the figure 7. The disorder peak on the RBS-C spectra recorded after iodine implantation reaches the random spectra. As the RBS-C technique can not distinguish an amorphous from a polycrystalline structure, we can not conclude on the real structure of this layer. But according to the XTEM micrographs of 6H-SiC implanted in similar conditions (low energy ions implantation at RT) found in the literature [9], this disordered layer should be amorphous. The subsequent Xe irradiation with a fluence of  $2 \times 10^{13} \text{ cm}^{-2}$  induces a small narrowing of the disorder peak but no decrease of this peak is observed. This is due to a recrystallization of the partially disordered layer between the complete amorphous part of the sample and the crystalline part. The absence of decrease of the peak does not imply an absence of recrystallization of the amorphous structure. This part of the disordered layer could have recrystallized in a polycrystalline structure, as it was observed after a thermal annealing of amorphous SiC [9].

## CONCLUSION

Nanostructured 3C-SiC samples were irradiated at RT with 95 MeV Xe ions and no large scale amorphization could be detected in spite of a number of dpa exceeding the amorphization threshold found in literature with low energy ions. However, a complete sample amorphization was noticed after irradiation at RT with low energy ions (4 MeV Au), where the nuclear interactions are dominant. The absence of amorphisation under 95 MeV Xe ions irradiation is assumed to be the consequence of the competing simultaneous effects of the electronic (healing) and nuclear (damaging) energy losses in SiC.

In order to study only the electronic interactions, single crystalline 6H-SiC were irradiated with 910 MeV Xe ions. In the virgin sample, no disorder was created by electronic

interactions. However, in the predamaged sample with a disorder rate around 60 %, the electronic interactions induce a strong disorder decrease. In the sample comprising a total amorphous layer, further analysis are needed to know the exact structure of the sample after high energy irradiation.

#### ACKNOWLEDGEMENTS

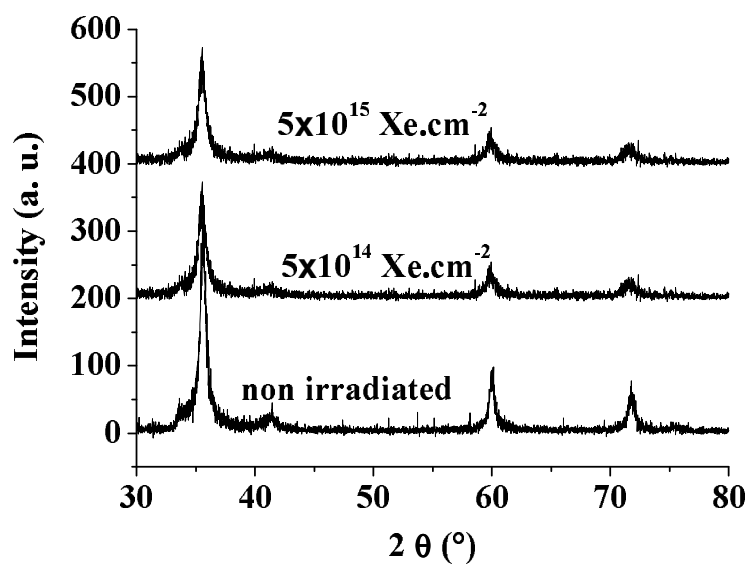
The authors thank the DEN/DMN/SRMA/LTMEX laboratory for the pellets sintering.

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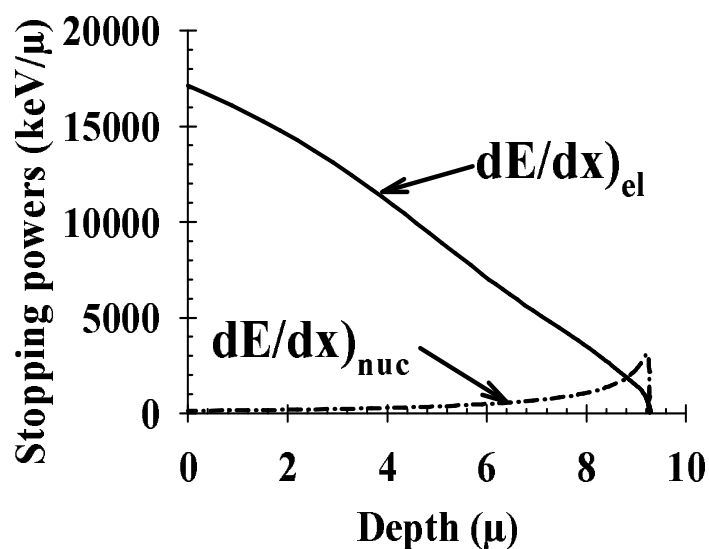
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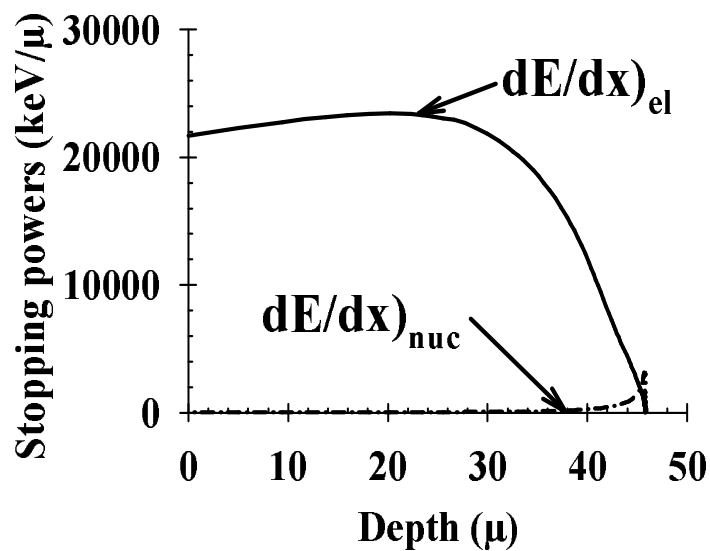
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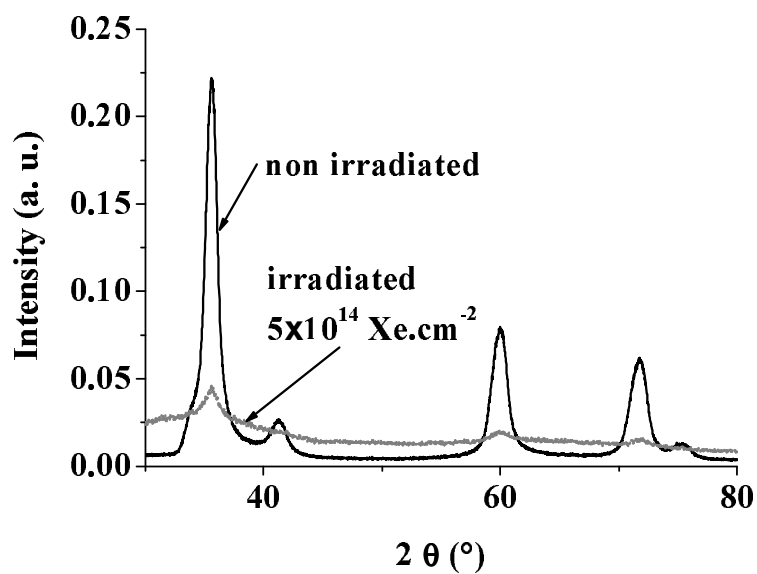
**Figure 1:** XRD diagrams recorded on the nanostructured sample before and after irradiation with 95 MeV Xe ions at  $5 \times 10^{14} \text{ cm}^{-2}$  and  $5 \times 10^{15} \text{ cm}^{-2}$  for a probed depth of 2.5  $\mu\text{m}$ .



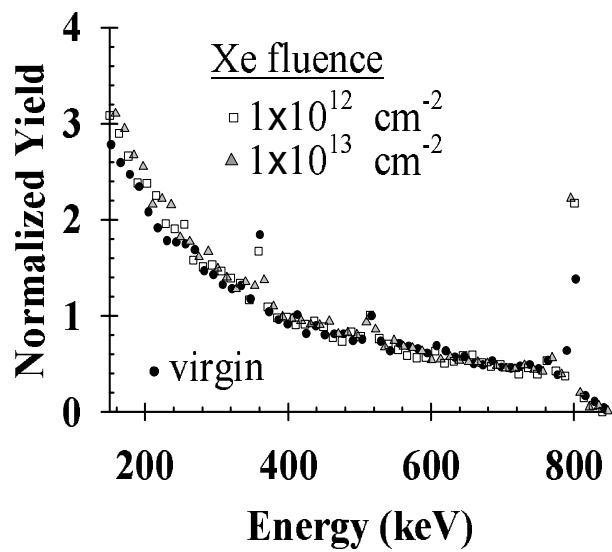
**Figure 2:** SRIM 2003 simulation of the ion pathway and corresponding nuclear and electronic stopping powers for 95 MeV Xe irradiations.



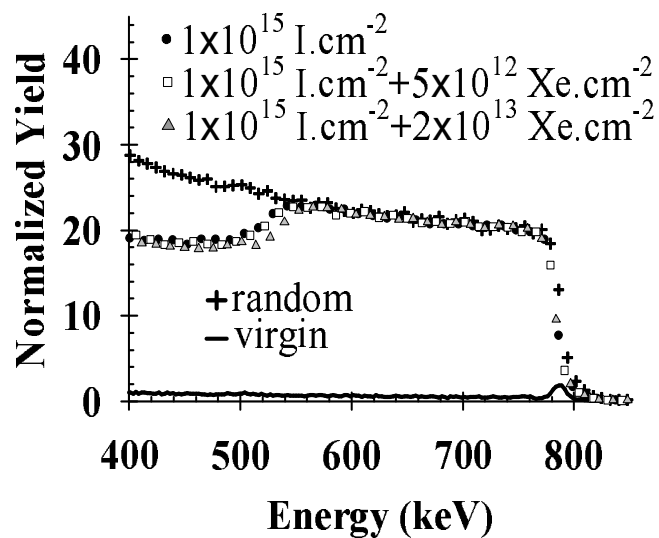
**Figure 3:** SRIM2003 simulation of the ion pathway and corresponding nuclear and electronic stopping powers for 910 MeV Xe irradiations.



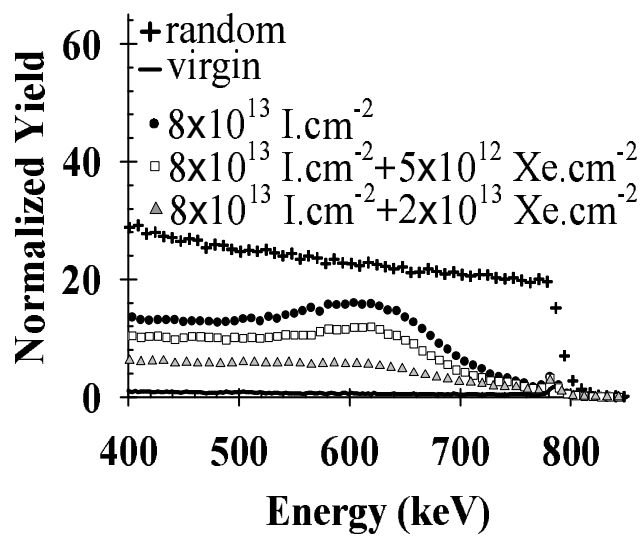
**Figure 4:** GIXRD diagrams recorded on the nanostructured sample before and after irradiation with 4 MeV Au ions at  $5 \times 10^{14} \text{ cm}^{-2}$  for a probed depth  $d$  of  $2.5 \mu\text{m}$ .



**Figure 5:** RBS-C spectra recorded before and after irradiation at RT with 910 MeV Xe ion at  $1 \times 10^{12} \text{ cm}^{-2}$  and  $1 \times 10^{13} \text{ cm}^{-2}$ .



**Figure 6:** RBS spectra recorded in random and channelling geometry on SiC single crystals irradiated at RT with 700 keV I ions ( $8 \times 10^{13} \text{ cm}^{-2}$ ) and subsequently irradiated at RT with 910 MeV Xe ions.



**Figure 7:** RBS spectra recorded in random and channelling geometry on SiC single crystals irradiated at RT with 700 keV I ions ( $1 \times 10^{15} \text{ cm}^{-2}$ ) and subsequently irradiated at RT with 910 MeV Xe ions.