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Radiation effects in carbides: TiC and ZrC *versus* SiC

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ABSTRACT

The aim of this paper is to provide, in the framework of the use of carbides for nuclear fuel applications, a comparison of the effects of irradiation observed in TiC and ZrC with those already widely investigated in SiC. To achieve this goal, the three carbides (SiC, TiC, ZrC) were simultaneously irradiated at room temperature with 1.2 MeV Au ions at several fluences. The damage created by irradiation was evaluated by RBS-C, XRD and Raman experiments. The results indicate a drastic difference between TiC and ZrC *versus* SiC. Whereas amorphization is readily achieved for SiC at low Au fluence (between 5×10^{13} and 1.7×10^{14} cm^{-2}), TiC and ZrC maintain their crystalline structure at the highest Au fluence used (7×10^{15} cm^{-2}). Ionicity character of bondings as well as topological criteria in TiC and ZrC, as compared to SiC, could play a major role for preventing amorphization in the former two carbides.

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1. Introduction

While the most studied of covalent carbides, SiC, presents very few unknown areas about its characteristics under irradiation [1–5], almost no data is available on its TiC and ZrC counterparts. Nevertheless, these materials present a high interest since they cumulate the properties of both metals (high electrical and thermal conductivities) and covalent ceramics (refractory, corrosion resistance, fission product retention ability and good mechanical properties). For these reasons they are considered as potential coating and oxygen gettering materials or inert matrices for advanced high temperature reactor fuels [6,7]. For this latter application, the knowledge of their behavior under irradiation is a mandatory issue.

In this paper, we compare the radiation tolerance of SiC, taken as a reference material, with that of TiC and ZrC. It is well known that irradiation of SiC with neutrons or low-energy heavy ions leads to amorphization by nuclear elastic collisions [1–5] that induces deleterious modifications such as swelling or crack formation [8]. Yet, data on the effects of irradiation of TiC and ZrC are extremely scarce in the literature. Furthermore, the majority of researches on these carbides was done on polycrystalline materials, either hot-pressed, explosion-pressed or slip cast. In this study, to avoid grain boundary effects, we used TiC and ZrC single crystals. Unfortunately, due to the difficulty to synthesize good crystals (contrarily to SiC), we have studied only one stoichiometry and one microstructure for each carbide, despite the fact that the ratio C/Metal has a strong influence on the irradiation response. Earlier studies on the ion beam modifications of TiC and/or ZrC may be found in Refs. [9–15].

The experiments reported in this article constitute the first step of a more general study devoted to the comparison of the damage induced in TiC, ZrC and SiC (used as a reference) single crystals irradiated with MeV heavy ions in order to explore the nuclear collision regime. The damage was evaluated by combining Rutherford backscattering and channeling spectrometry (RBS-C), X-ray diffraction (XRD) and Raman spectroscopy; the complementarity of these techniques has been demonstrated in several works [16–18].

2. Experimental

The samples used are (0001)-oriented 6H-SiC single crystals supplied by CREE Research Inc, and (100)-oriented TiC and ZrC single crystals (purity 99.99%) supplied by MaTecK GmbH. The oxygen contamination was checked, prior to ion irradiation, by using the $^{16}O(d,p_1)O^{17}$ nuclear reaction analysis (NRA) with a deuteron energy E_d = 900 keV, a detection angle of 150° and a 12 μm thick mylar screen in front of the detector. The fact that no signal appears at

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the expected energy of protons $E_p \sim 1490$ keV indicates that the samples contain less than 10^{15} O atoms $\times$ cm^{-2}, which is the nuclear reaction analysis detection limit.

All ion irradiations were performed at room temperature in a random direction with 1.2 MeV Au^+ ions at the JANNUS facility (CSNSM in Orsay and SRMP in Saclay). The projected ranges of Au ions obtained from SRIM [19] are ~200 nm for SiC, ~150 nm for TiC and ~140 nm for ZrC. The ion fluences used range from 3×10^{13} cm^{-2} to 7×10^{15} cm^{-2}. NRA and RBS analyses indicated that the stoichiometry of the compounds was not (or very slightly) modified by irradiation.

The RBS-C experiments were carried out with the ARAMIS accelerator at CSNSM Orsay using a 1.4 MeV $^4He^+$ ion beam. The detector is positioned at 165° with respect to the incident beam direction. The energy resolution of the experimental setup is about 14 keV. The depth distribution of accumulated damage (f_D) in the Si, Ti and Zr sublattices was extracted from the analysis of RBS-C spectra with the McChasy Monte-Carlo simulation code [20]. For the simulations we used the basic assumption that a fraction f_D of (Si, Ti, Zr) atoms were randomly displaced from their original lattice site during irradiation.

The XRD experiments were carried out at IEF in Orsay (for a complete description of the setup, see e.g. [21]). The wavelength of the X-ray beam was 0.15406 nm (Cu Kα1 radiation) and the beam divergence was 18′ arc-second. θ–2θ scans were recorded on the (00012) and on the (400) reflections of the hexagonal SiC and cubic TiC and ZrC crystals, respectively.

Raman experiments were carried out on an Invia Reflex Renishaw® spectrometer implemented at the Jannus Saclay facility. The spot size under the ×100 objective was 1 μm^2. The laser power was filtered down to 1.65 mW to avoid any transformation of the matter by heating. Spectra were recorded with a resolution of 1.5 cm^{-1}. Samples were studied with an excitation wavelength of 532 nm. A spectral window between 200 and 1800 cm^{-1} was used.

3. Results on SiC

3.1. Rutherford backscattering spectrometry-channeling

Random and aligned RBS-C spectra recorded on SiC crystals are displayed in Fig. 1a. The random spectrum exhibits a plateau starting at 800 keV, which corresponds to the backscattering of analyzing particles from Si atoms. A second plateau appears below 350 keV, which is due to the backscattering of analyzing particles from C atoms. On virgin crystals a strong decrease of the backscattering yield is observed in the main axis direction, due to the channeling effect. Fig. 1b shows the variation of f_D as a function of depth in SiC. As expected from the spectra in Fig. 1a, $f_D = 1$ at a depth of 200 nm for a fluence of 1.7×10^{14} cm^{-2}, indicating the creation of total disorder (amorphization). The amorphous layer broadens towards both the sample surface and greater depth with increasing fluence up to 3.3×10^{15} cm^{-2}.

3.2. X-ray diffraction

θ–2θ scans recorded on SiC are displayed in Fig. 1c. In this figure, the scattered intensity is plotted as a function of the scattering angle, 2θ (bottom axis) and of the elastic strain, ε_N (top axis). The signal detected at high-angle corresponds to the scattering of the X-ray beam by the virgin part of crystals (the thickness probed

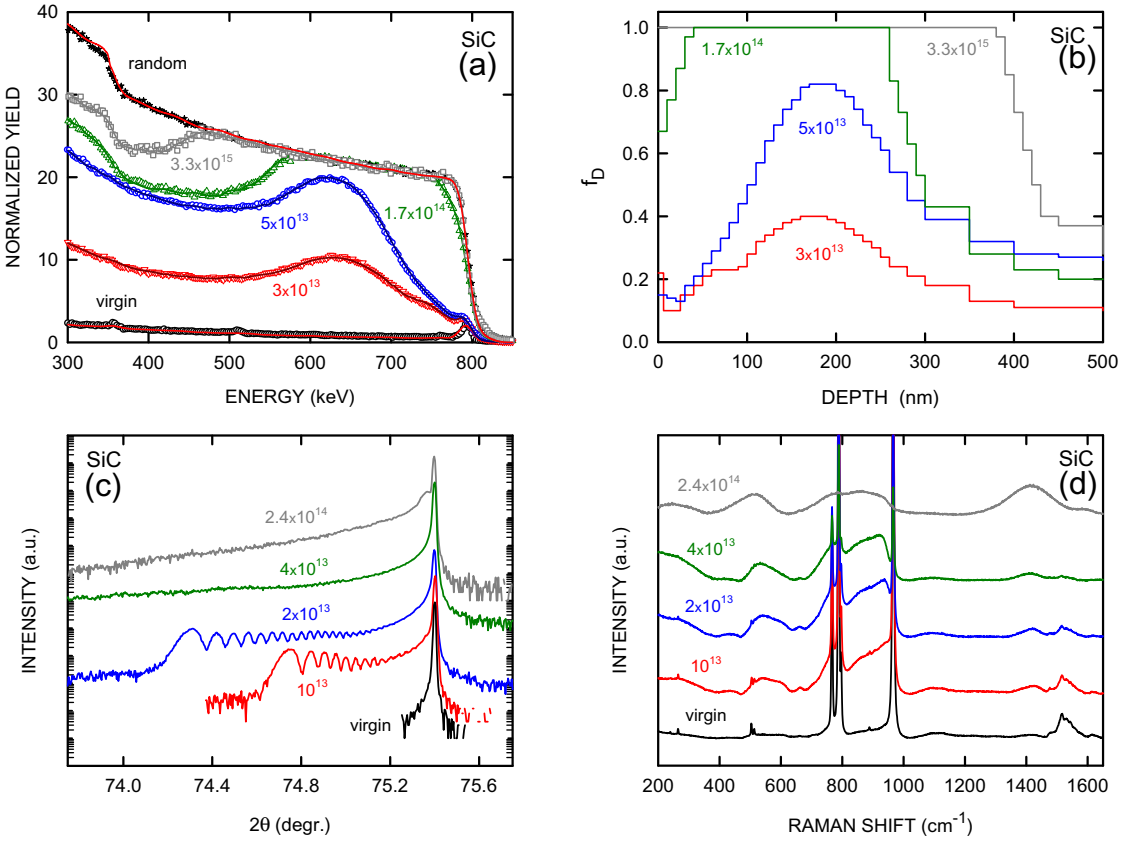

Fig. 1. (a) RBS-C spectra recorded in random (filled circles) and axial (open symbols) directions on SiC crystals irradiated with 1.2 MeV Au ions. The energy of the analyzing beam is 1.4 MeV. (b) Variation of f_D as a function of depth extracted from the analysis of RBS-C spectra with the McChasy code (lines superimposed to the spectra). (c) θ–2θ scans recorded in the vicinity of the (00012) reflection of hexagonal SiC crystals irradiated with 1.2 MeV Au ions. Curves are shifted vertically for visualization easiness. (d) Raman spectra recorded on SiC crystals irradiated with 1.2 MeV Au ions.

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