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Advanced SiC fiber strain behavior during ion beam irradiation

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The *in situ* strain behavior upon ion beam irradiation of a Tyranno SA3 SiC fiber was investigated in real time. For this purpose, a tensile test device suitable for micrometrical samples was developed to allow studies in various irradiation facilities. A 7.44 μm diameter SiC fiber was submitted to both low mechanical loading at 300 MPa and 92-MeV Xe ion beam irradiation at room temperature. The fiber exhibited a gradual increase of its longitudinal strain reaching a maximum value of 0.50% for a total fluence of 5×10^{14} ions cm^{-2} . Raman spectroscopy analyses performed on fibers submitted to the same irradiation conditions have shown significant local structure modification but no evidence of amorphization.

1. Introduction

Third generation SiC fibers have significantly improved the thermo-mechanical properties of SiC_f/SiC_m ceramic matrix composites which are considered as advanced materials for nuclear system applications [1–3]. These materials exhibit interesting features such as a low activation level and a higher operating temperature in comparison to ferritic steels and vanadium alloys. Such properties make them promising candidates for use as fuel cladding, structural components or flow channel inserts. However, their thermo-mechanical performances strongly depend on their complex microstructure which undergoes significant changes when they are irradiated [1,4–6]. Thus, the possible use of SiC_f/SiC_m composites points out the need to carefully study if their properties can fully comply with the aimed application requirements. In this context, a multi-scale approach is currently ongoing to provide a satisfying predictive modeling of the SiC_f/SiC_m composites behavior in a reactor. This is carried out by characterizing separately the different components of the composite, i.e. the fibers, the matrix and the interphase. However, only few data are available on the behavior of the last generation of SiC fibers [7–11]. As a consequence, it is necessary to improve this database especially under irradiation.

Numerous studies have been achieved on the irradiation effects in SiC. Most of them are related to its structural and microstruc-

tural evolution under irradiation [12–15]. Similarly, the effect of ion beam irradiation on the thermal and mechanical properties of SiC has also been investigated [16–19]. However, most of the studies are based on ion implantation with combined electronic and nuclear slowing down regimes leading to variable damage accumulation within the material. The purpose of this work was to specifically characterize the *in situ* swelling behavior of SiC fiber in real time for a 92-MeV Xe ion irradiation aiming a homogeneous damage profile in the material. This was conducted by measuring the longitudinal strain occurring in a thin diameter SiC fiber as a result of accumulation of ion-induced damage. To obtain accurate data, a miniaturized tensile test device was implemented on the IRRSUD irradiation line. This equipment was developed with the JANNUS Saclay irradiation platform [20,21] where the first *in situ* test was successfully performed [22]. A second experiment, which is the subject of this work, was achieved at the GANIL facility in Caen. The study was carried out by coupling both mechanical loading at 300 MPa and ion beam irradiation at room temperature (RT). The evolution of the irradiated fiber structure was studied using micro-Raman spectroscopy.

2. Experimental

2.1. Material

The test was performed on a Tyranno SA3 SiC fiber manufactured by UBE Industries Ltd, Japan (Fig. 1). This type of fibers

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exhibits a density over 97% of the theoretical density and consists in 3C polytype SiC crystals with sizes in the range from 100 to 300 nm [23]. These fibers are synthesized from graft polycarbosi-lane containing aluminum which acts as crystal growth inhibitor. The main characteristics of this type of fiber are given in Table 1.

2.2. Sample preparation

The first step of the experiment consisted in preparing a single fiber tensile test specimen according to the method described by Sauder et al. [8]. Accurate diameter measurements were regularly performed along the fiber using a laser diffraction technique resulting in an average diameter of 7.44 μm . The specimen was then introduced in the tensile test device (Fig. 2). Furthermore, additional fibers were irradiated in the same conditions without mechanical loading in order to perform a structural analysis by micro-Raman spectroscopy.

2.3. Tensile test device

The tensile test device (Fig. 2) operates in secondary vacuum (10^{-5} Pa) which makes it fully compatible with the required vacuum of the IRRSUD irradiation line. It allows tensile tests to be carried out up to 5 N from 298 up to 2073 K. The device consists in a Newport MFA CCV6 motorized linear stage for accurate fiber strain determination combined to a HBM U1A load cell. A dedicated LABVIEW program was developed to apply a constant loading of 300 MPa to the fiber during all the experiment in order to determine its strain variation in real time.

Fiber heating can also be performed using a Keithley 2410 High-Voltage SourceMeter under secondary vacuum. Besides, due to the very small diameter of the fiber in comparison to its length (25 mm), heat transfer through the grips could be neglected. As a result, the whole supplied electrical power P_{elec} is converted into radiative power which depends on fiber temperature according to Eq. (1):

$$P_{elec} = \sigma \varepsilon FS(T^{*4} - T_R^4) \quad (1)$$

where σ is the Stefan's constant, ε is the fiber emissivity, F is the shape factor, S is the surface of the specimen, T^* is the estimated temperature in K, and T_R is the RT in K.

The fiber temperature can only be measured above 1473 K with an IRCON Modline 3R bichromatic pyrometer due to the small diameter of the fiber. Below 1473 K, the fiber temperature is estimated from the supplied electrical power.

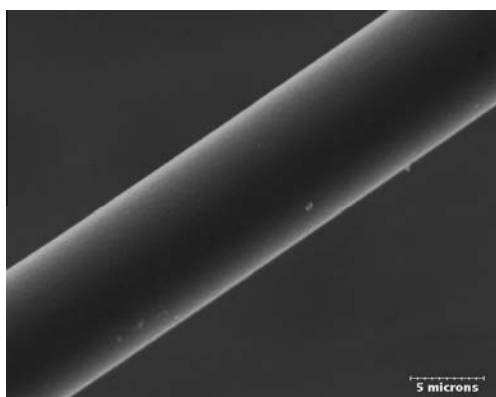


Fig. 1. SEM image of Tyranno SA3 SiC fiber.

Table 1
Main characteristics of Tyranno SA3 SiC fiber.

Fiber type	Tyranno SA3
Average diameter (μm)	7.5
Oxygent (wt.%)	<0.5
Young modulus (GPa)	390
Tensils strength (GPa) at RT (as received)	2.7

2.4. Irradiation conditions

The SRIM 2010 code was used to assess the damage induced in the material [24]. The displacement threshold energies for C and Si sublattices were, respectively equal to 20 and 35 eV [25]. Calculations show that 92-MeV Xe^{23+} ions have a projected range of about 9.02 μm in SiC which is higher than the fiber diameter. The irradiation was carried with an average flux of 3.3×10^9 ions $\text{cm}^{-2} \text{s}^{-1}$ and lead to a total fluence of 5×10^{14} ions cm^{-2} . At this fluence, the displacement per atom (dpa) level in the fiber varied in the range from 0.05 to 0.3 dpa for the most damaged side. For the latter, the value is close to the threshold amorphization dose for single crystalline SiC at RT [26,27]. However, it should be mentioned that this dpa value is overestimated due to healing in the electronic stopping power regime which is the major contribution in these irradiation conditions [28–30]. Indeed, the 92-MeV Xe^{23+} electronic stopping power is about 17 keV nm^{-1} in the SiC whereas the nuclear stopping power is about 0.13 keV nm^{-1} .

2.5. Raman spectroscopy characterization

Micro-Raman investigations were performed at RT in a back-scattering geometry using a Jobin-Yvon T64000 spectrometer coupled with an Olympus microscope which contains an X-Y-Z stage. The 514.5 nm line of an argon ion laser was focused on a $1 \times 1 \mu\text{m}^2$ spot and collected through a $100\times$ objective with numerical aperture value of 0.9. The laser output intensity was kept around 30 mW. Preliminary tests were carried to avoid any heating of the sample which could result in defect annealing. To analyze the different Si-Si, Si-C, and C-C related modes, the spectra were recorded in the spectral range between 600 and 1800 cm^{-1} . The fitting module of the Labspec Raman spectroscopy software was used for simulations of spectra [31].

3. Results and discussion

The SiC fiber strain arising during the test is presented (Fig. 3). By using the same vacuum and loading conditions, a significant difference exists between irradiated and non-irradiated fiber curves. The strain origin was set to 0% after loading at 300 MPa (1). As a consequence of the irradiation starting, a first strain of 0.04% is observed (2). Then, a gradual increase is measured reaching a maximum value of 0.50% at the end of the experiment (4). This value is consistent with those already found using similar materials and irradiation conditions [32,16]. The fiber temperature was calculated by taking into account the measured ion flux and the deposited energy, and found not to exceed 318 K during the experiment. As it can be seen, the thermal contribution due to the ion beam can be neglected since the shrinkage occurring in (3) corresponds to a temporary beam shutdown for 25 min. In addition, the strain curve exhibits numerous slope changes without any correlation with ion beam intensity fluctuations.

SiC seems to be suitable for high-temperature nuclear applications. However, the irradiation induced swelling phenomenon in this material can result in significant dimensional changes of structural components. Although the effect of irradiation on the

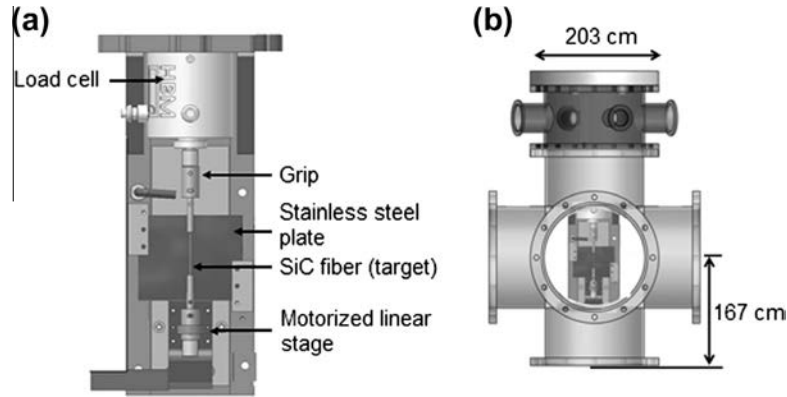


Fig. 2. Detailed view of MiniMecaSiC tensile test (a), implemented in the vacuum chamber (b).

microstructural evolution of pure SiC has been carefully studied [33], the defects contribution to the SiC swelling has still to be investigated. Furthermore, it has been shown that the physical-chemical characteristics of the material appear to be a key issue since the crystallinity and the presence of significant additional phases drastically affect the material stability under irradiation [4,34–37].

SiC irradiations at relatively low temperature lead to microstructures exhibiting ‘black spots’ mainly related to self-interstitial atoms clusters. For irradiation temperatures lower than 423 K which is considered as the critical amorphization temperature in the ballistic regime, the irradiation-produced defects can lead to the complete crystal amorphization. In addition, the swelling was found to vary logarithmically according to the dose up to the amorphization threshold [38–41]. Above 423 K, the swelling increases with the dose up to a saturation value which significantly decreases with the irradiation temperature. This temperature regime is called the point-defect swelling regime and extends from 423 to 1273 K. In this regime, ‘black spot’ defects tend to get larger with increasing temperature. The lower swelling is mainly explained by enhanced recombination of Frenkel defects at higher temperatures.

For irradiations carried out at temperatures ranging from 1173 to 1673 K, interstitial faulted Frank loops are considered as the most represented defects [42,43]. For temperatures where the vacancies mobility is high enough, vacancy clusters are formed. This leads to void formation which is reported to start at 1273 K in the case of Si ion irradiation [43,44]. Over this temperature range, a huge swelling increase is observed as described by Price et al. [45].

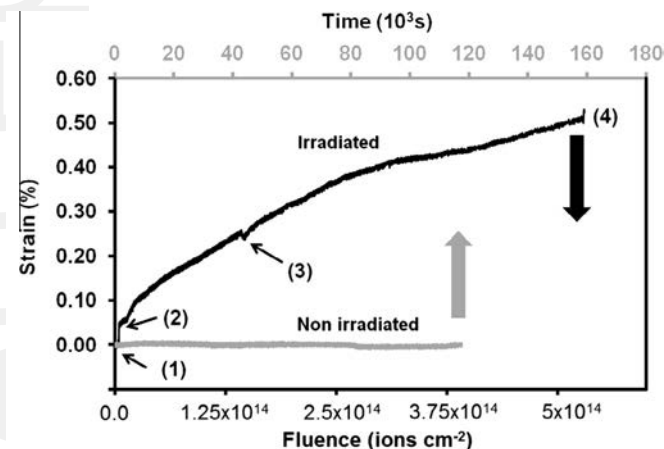


Fig. 3. SiC fiber strain as a function of time or fluence: irradiated fiber (black line) and non-irradiated fiber (grey line).

SiC exhibits a peculiar behavior to ballistic collisions as compared to electronic excitations since amorphization can occur for the first process while it is not the case for the second one. The contribution of electronic excitations to the ion stopping power is inefficient to produce defects and results in target heating. In addition, there is no evidence of swelling in SiC in this latter regime [46]. However, it should be mentioned that the SiC fibers could not be considered as pure SiC single crystal. Indeed, the Tyranno SA3 SiC-based fibers investigated in this work exhibit a dense microstructure consisting in nanometric equiaxed β SiC grains. This type of fiber contains less than 0.2% of oxygen and has a heterogeneous concentration in C and Si with a larger amount of free carbon in the core of the fiber. It also contains aluminum located at grain boundaries [8,47]. As a consequence, the effect of electronic energy loss on the fiber swelling cannot be clearly identified since behavior of each phase is specific.

In this study, we assumed that the irradiation conditions have led to point-defect accumulation distributed into small cascades. Point defects are produced by elastic interaction along the ion path with a range greater than the fiber diameter. As can be seen (Fig. 4), a relatively homogeneous nuclear-collision damage profile was obtained for a total fluence of 5×10^{14} ions cm⁻². However, it should be noticed that the side of the fiber opposite to the ion beam incidence exhibits a slightly higher damage level, in particular in the 6–7.5 μ m region.

The Raman spectra presented in (Fig. 5) give information related to the structural parameters of irradiated and non-irradiated fibers. In the case of SiC, various polytypes exist and consist in alternating Si and C layers. For the non-irradiated fiber, the Raman

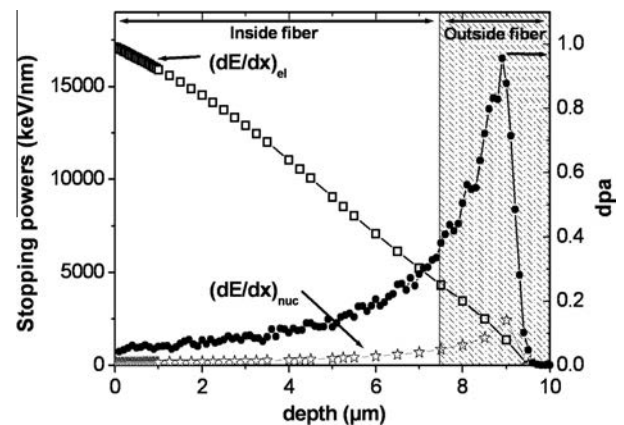


Fig. 4. SRIM calculations of the electronic and nuclear stopping power, and displacement damage level of 92-MeV Xe ions in SiC in the fiber for a fluence of 5×10^{14} ions cm⁻².

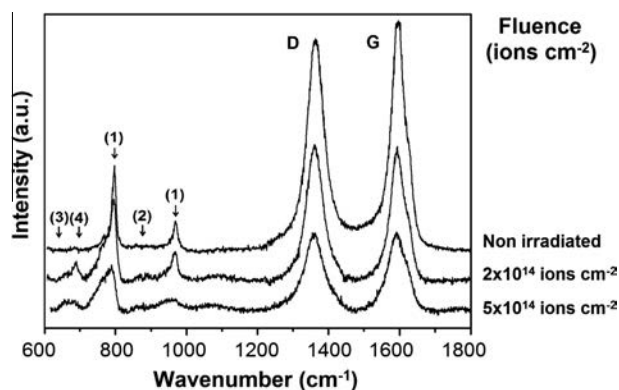


Fig. 5. Raman spectra of irradiated and non-irradiated SiC fibers.

bands can be attributed similarly to Ref. [48]. Actually, the β -SiC cubic symmetry exhibits two main bands at 796 and 972 cm^{-1} which, respectively correspond to the TO (transverse) and LO (longitudinal) optical modes. According to Colomban et al. [49], the two bands at 1331 and 1581 cm^{-1} are related to disordered carbon and correspond to the well-known D and G band constituents. The Raman spectra of irradiated fibers performed on the most damaged sides show a significant evolution in comparison to non-irradiated one. These differences concern the local structure modification. The intensities of the main SiC phonon bands (1) tend to decrease with dose indicating an increase in disorder. This is accompanied by band broadening and increase in intensity of the band at 896 cm^{-1} (2) with respect to the D and G bands. Appearance of highly disordered SiC bands at 659 (3) and 688 cm^{-1} (4) are also observed. Although a significant decrease in Raman line intensities related to crystalline lattice phonons was observed, no evidence of amorphization was found from this analysis [31]. This result is mainly explained by both above mentioned simultaneous electronic healing and nuclear damaging processes occurring in SiC which induces defects recombination or migration especially in nanostructured materials [28,30].

Thus, we assume that the strain variation was mainly due to the fiber swelling in these irradiation conditions for a very low mechanical loading level. In the range 1.25×10^{14} – 5×10^{14} ions cm^{-2} , the strain was found to increase in proportion to the 0.60 power of the fluence without reaching a saturation value. This dependence towards the fluence is in the range of the usually found value [50] but slightly lower than that in the Ref. [32]. This is mostly explained by the difference between both experiments, especially in terms of ion species and irradiation conditions. This type of results could be suitable to assess long term swelling rate at RT but also at high temperature with clearly known irradiation parameters. This could lead to a better comprehension of the physical meaning of the fluence exponent since it implies very complex defect production and recombination processes.

4. Conclusion

The purpose of this work was to investigate the fiber strain variation in real time upon ion irradiation. This objective was achieved using a miniaturized tensile test device dedicated to study SiC fiber properties. The irradiation was carried out with 92-MeV Xe ions for a total fluence of 5×10^{14} ions cm^{-2} leading to an estimated average irradiation dose of 0.1 dpa in the fiber, with a relatively homogeneous nuclear-collision damage profile.

Raman spectroscopy data have shown some local structure modifications of the irradiated fiber. Although appearance of

highly disordered SiC coupled with a decrease in Raman line intensities was observed, no evidence of amorphization was found. The fiber submitted to a low mechanical loading level of 300 MPa during irradiation did exhibit a gradual increase of its longitudinal strain reaching a maximum value of 0.50% at the end of the experiment indicating that radiation induced swelling phenomenon occurred.

This work will be taken further using complementary range of ions and energies in order to study various damage processes and their impact on the fiber properties. The next step will consist in coupling fiber heating and ion irradiation since creep tests can be performed with the above-mentioned equipment. Consequently, these experimental conditions will be as close as possible to the extreme conditions which could be found in nuclear reactor core. They will provide the opportunity to determine damage mechanisms involved in material during an irradiation at high temperature.

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