Mechanical properties of SiC fibers irradiated by swift heavy ions

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Abstract. Mechanical properties and surface features of SiC fibers irradiated with 246.8-MeV Ar ions at different fluences (1.5x10¹⁴, 9.0x10¹⁴, 1.8x10¹⁵, and 2.7x10¹⁵ ions/cm²) were investigated using a specific single filament tensile test and a field emission scanning electron microscopy (FE-SEM). Results reveal that carbon concentration on the fiber surface decreases while silicon concentration increases, with increasing ion fluence, accompanied by an adsorption of oxygen. Meanwhile, the diameter of fibers first reduces and then increases, with increasing ion fluence. Moreover, at 2.7x10¹⁵ Ar ions/cm² irradiation, some fibers were fractured. As a result, the mean tensile strength and the average elastic modulus of fibers decrease generally with respect to the ion fluence. The degradation mechanisms of mechanical properties of SiC fibers under irradiation were discussed in detail.

1. Introduction

Due to low neutron capture cross-section, low induced radioactivity, low afterheat and quick decay of activity, high thermal stability and excellent high-temperature mechanical properties, continuous silicon carbide fiber reinforced silicon carbide (SiCf/SiC) composites have been recognized as the promising structure materials of harsh environments, such as aerospace, aviation, fission and future fusion reactors [1-4]. SiC/SiC composites are composed of near-stoichiometric SiC fibers, stoichiometric and fully crystalline SiC matrices, and the pyrocarbon (PyC) or BN interphase between the fiber and the matrix [5]. The performance of SiC/SiC composites, especially its toughness and high ultimate strength, depends on the effectiveness of the interface component and the high strength of the fiber component [6]. In fission reactors, SiC fibers are inevitably subjected to various fission fragments (heavy ions with up to hundreds of MeV) irradiation, which are produced via chain fission of the uranium. While in fusion reactors, the average energy of recoils produced by neutron irradiation...
in materials can reach several hundred kV. Most of the energy of fission fragments/recoils is deposited in the materials mainly through electronic processes (excitation and ionization of atoms along the trajectory range). Therefore, it is indispensable to acquire a comprehensive knowledge of the mechanical behavior of SiC fibers under energetic-heavy-ion irradiation. In this work, in order to understand the mechanical stability and the failure mechanisms of SiC fibers induced by energetic heavy ions and forecast the performance of SiC/SiC<sub>n</sub> compositions in irradiation environments, we investigate changes of surface characteristics and tensile properties of SiC fibers irradiated with 246.8-MeV Ar ions at different fluences, by using a FE-SEM and a specific single filament tensile test.

2. Experiment
The SiC fiber specimens with diameter of about 10 μm were cut into a length of 50 mm and mounted on aluminum sheets with little overlap for irradiation. The irradiation was performed with high-energy Ar ions at a terminal chamber of the sector-focused cyclotron (SFC) at HIRFL (Heavy Ion Research Facility in Lanzhou) located in Institute of Modern Physics. The beam current was monitored with an Al foil assembly placed in the beam line before the chamber, which is composed of an Al foil of 2 μm in thickness to collect electric charge, a Φ18 mm aperture with a bias voltage of -300 V to suppress the secondary electron emission and a Φ15 mm aperture to limit the irradiation area. 40Ar<sup>16+</sup> ions passed through the assembly carry a kinetic energy of 246.8 MeV. The irradiation chamber consists of an energy degrader, a cooling stage (liquid nitrogen) and a heating stage (up to 600°C) of specimens for irradiation (Fig. 1). A description in detail of the irradiation terminal was given in our previous paper [7].

In order to increase the electron energy loss of Ar ion in 10-μm SiC fibers, we used the energy degrader, which is composed of 10-Al foils with thickness ranging from 4.899 to 45.009 μm. Fig. 2 shows estimates of the electron energy deposition density (in eV/μm<sup>3</sup>) and the displacement damage level (in dpa, induced by nuclear energy loss) of energy-degraded Ar ions in 10-μm SiC fiber specimen, corresponding to a fluence of 1.8x10<sup>15</sup>Ar-ions/cm<sup>2</sup>. The displacement damage level was estimated by using the “quick calculation” mode of SRIM 2012 code [8]. The displacement threshold
energies for C and Si sublattices were 20 and 35 eV, respectively [9]. The 10-Al foils with different thickness in the energy degrader result in a successive increasing electron energy deposition density in the SiC fibers with an average diameter about 10 µm. Ar ions which passed through the two foils with thickness of 40.699 and 45.009 µm could produce a considerable displacement damage via nuclear collision in 10-µm SiC fibers. The irradiation fluence was from 1.5x10^14 to 2.7x10^15 Ar ions/cm^2. The temperature of specimens was kept around 40°C during irradiation in a vacuum of 10^-5 Pa range.

Fig 2. Electron energy deposition density and dpa distribution with respect to depth in SiC fibers under irradiation with 1.8x10^15 Ar ions/cm^2 through the energy degrader. The horizontal column shows the thickness of 10 Al foils used in the energy degrader. The dpa values corresponding to Ar ions passed the 40.699 and 45.009 µm Al foils were plotted.

After Ar ions irradiation, tensile properties and surface features of SiC fibers were characterized by means of a specific single filament tensile testing machine and a field emission scanning electron microscopy (FE-SEM). The model of the electronic single filament tensile machine is Textechno FAVIMAT®. The two ends of a fiber segment were fixed to two dog-bone shaped grips carefully during the tensile test. The span is 25 mm and the tensile rate is 1 mm/min. The model of the FE-SEM is Nano-SEM 450 with the maximal acceleration voltage of 35 kV. Electron image scanning and energy dispersive spectra (EDS) area scanning with electron energy of 15 keV were carried out at a high-vacuum mode.

3. Results and discussion

3.1. Surface characteristics and diameter analysis
The surface morphology, compositions and the diameter of SiC fibers after Ar ions irradiation were studied by a FE-SEM. The FE-SEM measurement was carried out along the fiber length within a fiber tow. Figs. 3 and 4 show SEM images, energy dispersion spectra and the mean diameter of SiC fibers irradiated with Ar ions at different fluences. For un-irradiated SiC fibers specimen, its average diameter is about 9.7 µm, the surface of fibers is smooth and the atom ratio between silicon and carbon atoms is about 2:3.

After irradiation at a low fluence (9.0x10^14 Ar ions/cm^2), the average diameter of the SiC fibers decreases to 8.9 µm and the atom ratio between silicon and carbon atoms is above 2:3. That is, the
concentration of carbon content reduces at the surface, possibly due to the lower displacement threshold energy of carbon atoms compared with silicon atoms [9]. On the other hand, oxygen atoms also appear at the fibers surface, because the damaged SiC fibers absorb oxygen atoms easily. After irradiation at a high fluence (2.7x10^{15}Ar ions/cm^2), SiC fibers were fractured and at the fracture site, an enrichment of the silicon and a lack of the carbon were found. Meanwhile, big grains/bubbles appear on the fiber surface and the surface roughness of the SiC fibers also sharply increases. Moreover, the average diameter of fibers increases to about 10 µm.

![Image](image-url)

**Fig 3.** SEM images, EDS and the diameter of SiC fibers irradiated with Ar ions at different fluences.

**Fig 4.** The average diameter of SiC fibers as a function of the Ar-ion fluence.

### 3.2. Single filament tensile testing

In order to evaluate the influence of the irradiation on the mechanical stability of fibers, we carried out the single filament tensile test. The single filament tensile test can provide a mean tensile strength and an average elastic modulus. Mean tensile strength and elastic modulus were calculated from the load \( F \), gauge length \( L \) and the diameter \( d \) measurement. The tensile strength (failure strength) was characterized by \( 4F_{\text{max}}/\pi d^2 \), where, \( F_{\text{max}} \) is the load at failure. The average diameter \( d \) was determined from above FE-SEM views (Figs. 3 and 4). The elastic modulus was characterized by \( 4LF_{\text{max}}/\pi d^2/(L_{\text{max}}\cdot L) \), where, \( L_{\text{max}} \) is the fiber length at failure. The mean failure load is taken as the arithmetic mean for the same irradiation condition. For the same irradiation condition, twenty or more specimens were tested to ensure statistics. The mean diameter is also taken as the arithmetic mean.
Fibers that failed at the grip fiber interface were discarded from the analysis. Typical single filament stress-strain curves of SiC fibers after they were irradiated with Sn ions at a fluence of $1.8 \times 10^{15}$ Ar ions/cm$^2$ were shown in Fig. 5. The scattering of the ultimate tensile strength and the strain is due to the fluctuation of diameters of SiC fibers. All SiC fiber specimens were tested in the same way.

Fig 5. Typical single filament stress-strain curves of SiC fibers after they were irradiated with Ar ions at a fluence of $1.8 \times 10^{15}$ Ar ions/cm$^2$.

Figs. 6 and 7 show the mean tensile strength and the elastic modulus of SiC fibers as functions of the ion fluence, after they were irradiated with Ar ions at different fluences. The elastic modulus value of fibers exhibit an obvious decrease as the ion fluence increases up to $1.8 \times 10^{15}$ ions/cm$^2$. Above this fluence, the elastic modulus slightly increases with the ion fluence (Fig. 7). And the tensile strength always displays a reduced trend with increasing ion fluence (Fig. 6). However, no significant change was observed in tensile-strength values. The mechanical strength of the fibers is determined mainly by the bonds between the particles rather than by the constituent particles themselves [10]. After irradiation, disordered structures were produced because the intergranular amorphous SiC$_x$O$_y$ phase was decomposed into four fragments (SiC, C, SiO, CO), meanwhile the rearrangement and coalescence of SiC and C phases occurred under the heat effects of electron energy loss of Ar ions [11]. This rearrangement and coarsening of the crystal grains reduce its mechanical retention through the increase in the density of grain boundaries, because bonds between the grains are much weaker than those between atoms in crystalline or amorphous states [10, 12]. Simultaneously, during the decomposition-agglomeration process, the stress between the grains may be produced. The generation of these stresses is also responsible for the failure in fiber mechanical stabilities. On the other hand,

Fig 6. Average tensile strength of SiC fibers versus the irradiation fluence, after SiC fibers were irradiated with Ar ions at different fluences.

Fig 7. Mean elastic modulus of SiC fibers versus the irradiation fluence, after SiC fibers were irradiated with Ar ions at different fluences.

the formation of surface defects accelerates the attack of oxygen to fibers, resulting in an obvious reduction in fracture toughness (Fig. 3). However, at a fluence of $2.7 \times 10^{15}$ Ar ions/cm$^2$ irradiation, the
elastic modulus of fibers slightly increases. This is due to the material experiences a recovery from an amorphous phase to crystalline state under the high electronic energy deposition of Ar ions, revealed by TEM analysis in our previous work [13]. Summarily, the strength degradation of SiC fibers induced by irradiation can be ascribed to the decomposition of amorphous phase and the surface damage as well as the generation of stresses.

4. Conclusions
Surface morphology, compositions, and the mechanical properties of SiC fibers irradiated with 246.8-MeV Ar ions at different fluences were investigated using FE-SEM and a special single filament tensile test. Results show the irradiated SiC fibers first radically shrink and then expand, and at the surface, carbon concentration decreases while silicon concentration increases gradually, with increasing ion fluence. At the highest fluence irradiation (2.7\times10^{15} \text{Ar ions/cm}^2), the SiC fibers were fractured. As a consequence, the fiber mechanical stability generally degrades with respect to the ion fluence due to severe stoichiometric imbalance between Si and C atoms and the addition of oxygen. Meanwhile, this degradation in mechanical properties also can be ascribed to that the decomposition and the coalescence of the amorphous phase lead to the grain agglomeration and coarsening, and flaws/stresses generation under the heat effects of electron energy loss of Ar ions.

5. References
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Acknowledgements
This work has been supported by the National Natural Science Foundation of China (Grant Nos. 11675231, 91426304).