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Letter to the Editors

Effects of argon ion irradiation on the microstructures and physical properties of carbon fibers

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Abstract

Eight kinds of carbon fibers with different microstructures have been exposed to argon ion implantation at 175 MeV – 1 μA for 399 min using AVF cyclotron, Takasaki Radiation Chemistry Research Establishment, JAERI. After ion irradiation changes in diameters and cross-sectional areas of carbon fibers were determined by scanning electron microscopy. Tensile properties were measured before and after ion irradiation. As a result, the diameter generally tended to decrease after ion irradiation, except for the carbon fiber with the dual microstructure that has two directions (radial and circumferential) of basal planes in the cross-section of the fiber. Tensile strength decreased after ion irradiation. The decrease in tensile strength suggests that changes in axial microstructures due to ion irradiation give an influence on the mechanical properties of the fibers.

Carbon fiber reinforced carbon composites (C/C composites) are used as plasma facing components of most fusion devices in the world and it is probable for C/C composites to be used for the future plasma facing components of fusion reactors. It is desirable to develop radiation resistant C/C composite for nuclear fusion applications [1]. C/C composite materials are composed of carbon fibers and carbon matrices. It is generally known [2] that the structures and properties of carbon fibers with lower degree of graphitization are more greatly influenced by neutron irradiation. One of authors (M.I.) has intensively investigated microstructure and some properties of mesophase- and pitch-based carbon fibers, some of which were used in this experiment [3–5].

The purpose of this paper is to evaluate the effects of argon ion irradiation on the structures and properties in order to obtain useful information for the selection of radiation resistant carbon fibers. High-energy argon ions generated irradiated eight kinds of carbon fibers and a comparison of properties was made. The TRIM code was used to evaluate the damage rate in the carbon fibers [6].

The carbon fibers selected were six kinds of mesophase-pitch-based carbon fiber and two kinds of PAN-based carbon fiber. Table 1 shows the sample code and the cross-sectional feature of eight kinds of carbon fibers. Generally, mesophase-pitch-based carbon fibers indicate four kinds of microstructures that are radial (the direction of basal planes is circumferential) with notch, purely radial, dual (radial and circumferential), and concentric (the direction of basal planes is radial). Pitch-based carbon fibers showed dual structures as stated above. On the other hand, PAN-based carbon fibers indicated complex microstructures that include radial and concentric structures in the cross-section of the fiber. The diameter and cross-sectional area of carbon fibers were measured using a scanning electron microscope. Tensile tests of carbon fibers were conducted according to the Japanese Industrial Standard (JIS R 7601 1986) by using a universal testing machine. All tests were done on the specimens before and after ion

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Table 1
Carbon fibers tested

Sample code	Feature
I-1-28[a]	Mesophase-pitch-based carbon fibers, radial structure with notch
I-2-28[a]	Mesophase-pitch-based carbon fibers, radial structure
I-3-36[a]	Mesophase-pitch-based carbon fibers, dual structure
I-4-26[a]	Mesophase-pitch-based carbon fibers, concentric structure
Dialead (Mitsubishi)	Mesophase-pitch-based carbon fibers, dual structure
Dialead, heat treated (Mitsubishi)	Mesophase-pitch-based carbon fibers, dual structure
Shikishima Canvas	PAN-based carbon fibers, complex
Shikishima Canvas, heat treated	PAN-based carbon fibers, complex

[a] Manufactured by Kyushu National Industrial Institute.

irradiation. Ten to twenty specimens for one kind of carbon fibers were used for each test, and the average value was determined with its standard deviation.

Argon ion irradiation was conducted by using the AVF cyclotron in Takasaki Radiation Chemistry Research Establishment of JAERI. A 1 μA beam of $^{40}Ar^{8+}$ ions at 175 MeV was applied at a beam density of 2.6×10^8 ion/mm^2 s. Following irradiation carbon materials was simulated by the TRIM code.

The parameters used in the calculation by computer simulation were assumed that the density of the carbon fiber is 1.7 g/cm^3, the displacement energy for carbon atom is 37 eV and the binding energy of the carbon atom is 7 eV. The range of argon ion with 175 MeV in the carbon material was about 50 μm. Since the argon ion should penetrate into the carbon fiber with the diameter of less than 20 μm, radiation damages are expected to be formed uniformly. It is considered that the carbon fibers irradiated here had homogeneous damage through diameter of the fiber, since the damage distribution up to 10 μm in the carbon was flat. If we assume the carbon fibers to have a diameter of 10 μm and a length of 30 mm, we have the damage fluence of about 0.7×10^{-3} dpa. Irradiation temperature was not measured but was considered not to be over 100 °C.

An angle of the notch for the notched mesophase-pitch-based carbon fiber (I-1-28) decreased due to ion irradiation and by neutron irradiation [7,8]. The carbon fibers with dual structure (I-3-26 and two Dialead fibers) showed no significant changes in the cross-sectional microstructures. Fig. 1 indicates that the cross-sectional area of most carbon fibers, except two dual-structured fibers (I-3-26 and Dialead heat-treated), decrease due to argon ion irradiation. Changes in diameter and cross-sectional area of the fibers are due to radiation-induced defects that were produced during ion irradiation. The defects are reasonably supposed to be mainly carbon interstitial atoms, vacancies and their loops that are between hexagonal layers. These defects were known to result in the expansion along *c*-axis direction and, as a consequence, bring about shrinkage in the parallel direction to the hexagonal layer plane. These should reflect the changes in diameter and cross-sectional area of carbon fibers irradiated by argon ion. In the carbon fibers, the basal planes are oriented either in tangential or radial orientations to fiber axis. Since interplane distance expands due to interstitial atoms as defects, the angle of the notch in the cross-section of the I-1-28 fiber decreases after ion irradiation.

Young's modulus of carbon fibers was estimated from the slope at the origin of the stress–strain curve obtained by tensile tests. Young's modulus values

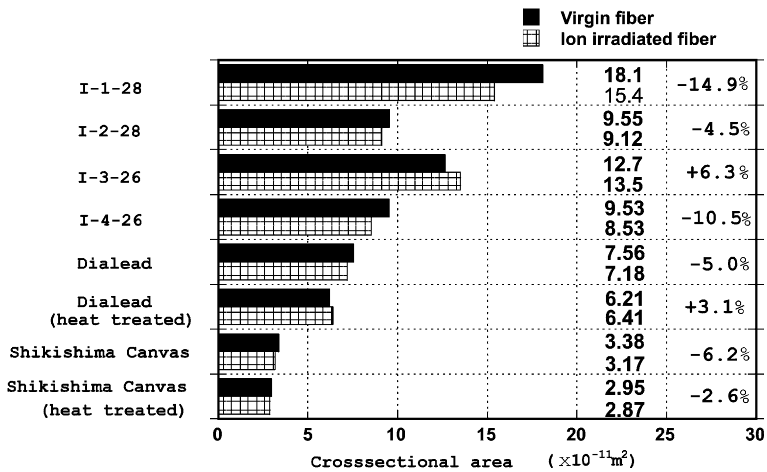

Fig. 1. Cross-sectional areas for unirradiated and argon ion irradiated carbon fibers.

increased by ion irradiation, except for the dual-structured carbon fibers [I-3-26 and the two Dialead carbon fibers (non-heat-treated and heat-treated)], as shown in Fig. 2. On the other hand, tensile strength of the carbon fibers, except for two non-heat-treated fibers (Dialead and Shikishima Canvas), decreased by ion irradiation, as shown in Fig. 3. Tensile strength of the heat-treated Shikishima Canvas fiber was the same as the unirradiated one. It is considered that non-heat-treated carbon fibers are composed of poorly crystallized regions, which are significantly influenced due to ion irradiation as compared with well crystallized ones of heat-treated carbon fibers. It is generally understood that Young's modulus and strength of graphite materials increase by ion irradiation. However, decreases in Young's modulus and tensile strength that were seen on most of present carbon fibers must be interpreted by other mechanisms than the pinning of dislocation due to ion irradiation damage. One of the possibilities for this is that the axial microstructure of the carbon fibers might have been destroyed by ion irradiation. The mesophase-pitch-based carbon fibers have a microstructure like chained-beads in the axial direction [9]. Since the bond between carbon beads is considered to be more easily destroyed than carbon beads themselves that are more crystalline than the bond, the above idea may be understood.

The eight kinds of carbon fibers, mesophase-pitch-based and PAN-based type fibers, were irradiated using high-energy argon ions. As a result, the following conclusions were drawn:

1. The diameter and cross-sectional area of carbon fibers decreased due to argon ion irradiation, except for the dual-structured fibers.

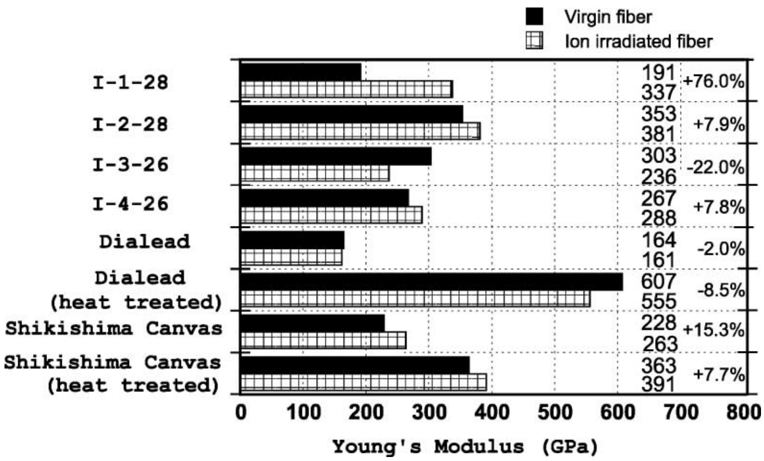

Fig. 2. Young's moduli for unirradiated and argon ion irradiated carbon fibers.

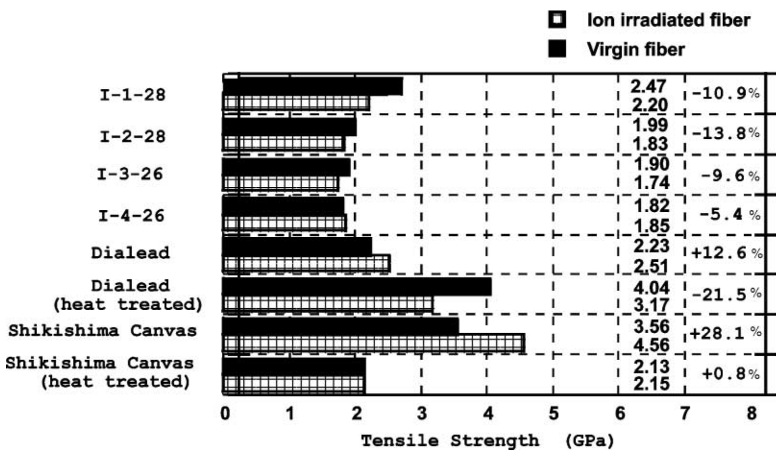

Fig. 3. Tensile strengths for unirradiated and argon ion irradiated carbon fibers.

2. The tensile strength of carbon fibers also decreased due to ion irradiation. The mechanism for this was considered that the axial microstructure of the carbon fiber may control fracture of the fiber.
3. Young's modulus of most carbon fibers increased after ion irradiation. However, Young's modulus of some fibers decreased as well as tensile strength. The same mechanism as tensile strength was considered.

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