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Effect of Neutron Irradiation on the Microstructures and Tensile Properties of Different Carbon Fibers

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Since carbon fiber reinforced carbon composite (C/C composite) materials have high thermal conductivity and good mechanical properties, they have been used as the plasma facing components in fusion facilities. As the plasma facing components are subjected to neutron irradiation in the fusion reactors, it is necessary to use irradiation damage resistant C/C composite materials as plasma facing components. Properties of C/C composite materials after neutron irradiation are generally influenced by irradiation behavior of carbon fiber and carbon matrix. In particular, the effect of irradiation on carbon fiber is important, because it is less crystalline than carbon matrix. The purpose of this study is to evaluate neutron irradiation effects on the microstructures and tensile properties of nine kinds of carbon fibers and to find out the necessary knowledge to identify the radiation resistant carbon fiber.

KEYWORDS: carbon fiber, microstructure, tensile property, neutron irradiation,

1. Introduction

Recently, fine-grained isotropic graphites and C/C composite materials for nuclear reactors are used for plasma facing components of fusion devices in the world. This is due to that carbon materials have excellent properties of high specific strength, high specific elastic modulus and high thermal shock resistance. When such carbon materials are used for fusion reactors, it is desirable to use carbon materials in which radiation effects are permissibly small. In particular, decrease in thermal conductivity due to neutron irradiation is considered to deteriorate remarkably the function as plasma facing materials. Consequently, to develop carbon materials in which the radiation damage effect is small is an important problem to solve in order to use them for fusion reactors. As C/C composite materials are composed of carbon fiber and carbon matrix, the radiation damage effects are considered to determine the behavior of C/C composite after neutron irradiation.[1)2)]

According to results obtained so far, it made clear that highly crystalline carbon materials give smaller effects of neutron irradiation damage. Since carbon fibers are less crystalline than carbon matrix, irradiation effects are considered to be controlled by effects of neutron irradiation on properties of carbon fibers.

In this study nine kinds of carbon fibers which have different kinds of microstructures were irradiated by using JMTR. After neutron irradiation examinations of microstructures by the scanning electron microscope, measurements of diameter of carbon fiber and tensile tests were performed and compared with results obtained before irradiation.

2. Experimental

2.1 Materials

The nine kinds of carbon fibers are used as test materials : four kinds of mesophase pitch carbon fibers, made by National Institute for Industrial Research, heat treatment temperature of 2800°C (I-1-28, I-2-28), two kinds of pitch carbon fibers called Dialead, made by Mitsubishi Chemical Co., and three kinds of PAN carbon fibers, made by Shikishima Canvas Co.and Showa Denko Co..

The neutron irradiation was conducted by JMTR, G-11 hole, to the fluence of $(2.0\text{-}2.7)\text{x}10^{24}\text{n/m}^2$ at 150-180°C. The carbon fibers tested are listed in Table 1.

Table 1 Specimen and feature.

Specimen	Feature
I−1−28	mesophase pitch fiber (on notch radial)
I−2−28	mesophase pitch fiber (radial)
I−3−26	mesophase pitch fiber (dual structure)
I−4−26	mesophase pitch fiber (concentric)
Dialead	pitch fiber
Dialead (heat treated)	pitch fiber
Shikishima Canvas	PAN fiber
Shikishima Canvas (heat treated)	PAN fiber
Used for CC−312	PAN fiber

2.2 Microstructure Observation

The microstructures of carbon fibers were observed at the magnification of 1000-7500 by the high resolution scanning electron microscope with EDS, JEM-5400, made by Nippon Electronics Co. The diameter and cross sectional area of carbon fibers were measured by using SEM photos.

2.3 Tensile Test

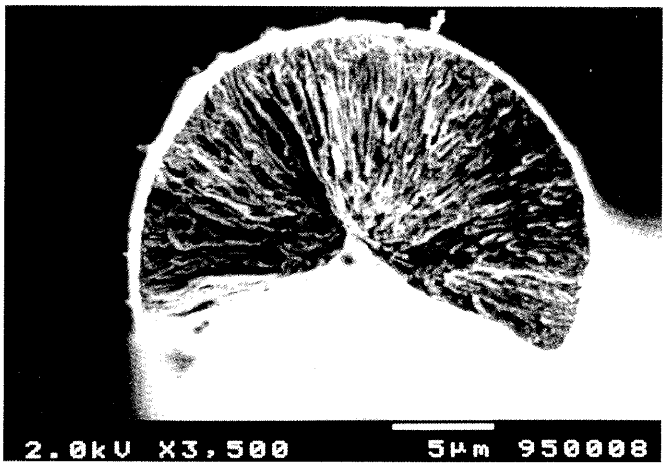
The tensile test of carbon fibers was conducted on the basis of JIS Standards. Both edges of the fiber was fixed on the center of the paper made a square hole with the epoxy resin glue. The test was done by pulling the clipped paper and cutting the paper before test. The gage length of the specimen was 10 mm for other than I-1-28 and CC-312, 5 mm for I-1-28 and 3 mm for CC-312.

The average of the cross sectional area of carbon fiber observed by SEM was used as that of the specimens.

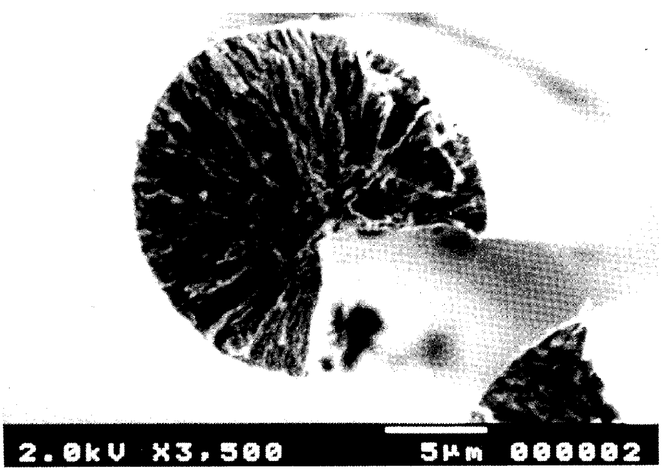
3. Results and Discussion

3.1 SEM Observation

Results of observations by SEM are shown as an example in Fig.1. The notch angle of I-1-28 specimen decreased due to neutron irradiation. The diameter and the cross sectional area changed according to the kind of fiber.



(a)Unirradiated



(b)Irradiated

Fig. 1. Photos of the cross section of the fiber with notch-radial structure before and after neutron irradiation.

3.2 Dimensional Change

The dimensional change in diameter of carbon fibers and change in cross sectional area are shown in Table 2 and 3, respectively. The diameter of pitch and PAN fiber showed comparatively small change, compared with that of mesophase fibers. The fiber with notch-radial structure and a kind of PAN fiber (CC-312) indicated decrease in diameter, while that of the other fibers increased.

Table 2 Changes in diameter of carbon fibers after neutron irradiation.

Specimen	Changes in diameter (%)
I-1-28	-1.9
I-2-28	+5.4
I-3-26	+3.2
I-4-26	+2.7
Dialead	+0.5
Dialead (heat treated)	+1.6
Shikishima Canvas	+1.7
Shikishima Canvas (heat treated)	+0.6
Used for CC-312	-3.2

Table 3 Changes in cross-sectional area of carbon fibers after neutron irradiation.

Specimen	Changes in crosssectional area (%)
I-1-28	+6.2
I-2-28	+7.9
I-3-26	+7.5
I-4-26	+7.0
Dialead	+1.2
Dialead (heat treated)	+4.6
Shikishima Canvas	+6.9
Shikishima Canvas (heat treated)	+1.4
Used for CC-312	-7.0

The cross-sectional area of all the fibers except CC-312 increased and that of CC-312 decreased 7%.

The diameter of the radial with notch fiber (I-1-28) decreased, although the cross-sectional area increased. This is due to decrease in the notch angle.

It seemed to be easy to understand from the viewpoint of irradiation damage of graphite crystals that the diameter and cross-sectional area of the fibers showing concentric structure increased. This is due to increase in d-spacing by defects produced by neutron irradiation. However, it seemed to be difficult to understand that the diameter and cross-sectional area of the fibers showing radial structure without notch increased. Because the dimension of the lattice parameter in the a-direction is considered to decrease due to neutron irradiation. It might be explained that the fiber showing radial structure contains not only radial structure but also concentric one which contributed to the increase in diameter and cross-sectional area.

3.3 Tensile Strength

Changes in tensile strength were shown in Table 4. Tensile strength of all the fibers increased and the amount of increase was different according to the kind of fiber.

Increase in tensile strength was mainly due to increase in the frictional strength by radiation damage defects.

Table 4 Changes in tensile strength of carbon fibers after neutron irradiation.

Specimen	Changes in tensile strength (%)
I-1-28	+20.5
I-2-28	+1.3
I-3-26	+0.1
I-4-26	+3.1
Dialead	+25.4
Dialead (heat treated)	+3.7
Shikishima Canvas	+7.2
Shikishima Canvas (heat treated)	+13.0
Used for CC-312	+4.5

4. Conclusion

Neutron irradiation of nine kinds of carbon fibers has been performed by using JMTR up to (2.0-2.7) x 10^{24} n/m^2 at 150-180°C. As a result, the diameters of the mesophase pitch fiber with notch-radial structure and a kind of PAN fiber (CC-312) decreased, while those of other fibers increased. The cross-sectional area of all the fibers except CC-312 increased. The tensile strength of all the fibers increased. In particular, it is noted that the tensile strengths of the fibers with notch-radial structure and a kind pitch fiber structure (Dialead) indicated larger increase.

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