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Section 5.3. High Z (Mo, W, etc.)

# High energy neutron and charged particle irradiation effects on thermomechanical properties of carbon–carbon composites for divertor applications

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**Abstract**

To assess the degradation of carbon–carbon (C/C) composite materials for the divertor structure, 14 MeV neutron and charged particle simulation irradiations were performed on several grades of C/C composites. For the charged particle irradiation the damage to be caused was also estimated using EDEP-1 code. Materials used were several grades of C/C composites, all of which were used in the JT-60 as divertor armor tiles. The measurement of thermal diffusivity up to 1600 K, electrical resistivity during 14 MeV neutron irradiation and the micro-indentation test at room temperature were performed. The strength and Young's modulus were evaluated on the basis of the result of micro-indentation test. Main results are: (1) The maximum of the microhardness was found near the maximum projected range of the C/C composite for charged particles. (2) After the irradiation of 10 MeV protons ($<0.1$ dpa), 10–90% increases in tensile strength and Young's modulus were observed, depending on the grades of C/C composites. (3) Softening of C/C composites was observed in the micro-indentation test when they were irradiated by 14 MeV neutrons up to a fluence of $10^{19}$ n/m$^2$.

## 1. Introduction

Carbon materials have been used for the divertor plate in most tokamak-type fusion facilities [1–3]. Low atomic number and high heat resistance are advantages for their applications to plasma facing components. The carbon fiber-reinforced carbon material (C/C composite) is a candidate for the armor tile of the divertor of ITER. It is believed, however, that the effect of neutron irradiation, 14 MeV neutrons in particular, on thermal and mechanical properties of the material have not been extensively investigated so far, although it is apparent that the irradiation data on the material are essential for the screening, design and safety evaluation of the divertor structure.

It has been proposed that high energy proton and other ion irradiations are a good alternative for simulating the 14 MeV neutron irradiation, considering that the availability of facilities for the irradiation are scarce [4]. It is also known that high energy He as well as a neutron are produced in the fusion reaction, $D + T \rightarrow {}^4He(3.52\ Mev) + n(14.06\ MeV)$. From this fact it is suggested that the effect of He irradiation should also be investigated. One demerit of the ion irradiation is that the damaged region in a specimen is to be localized because of the limited range of ion. In this respect, it is considered that the self- ion irradiation would give more damage in the wider region. Taking the above situation into account, in this paper the irradiations of 14 MeV neutron, proton, helium and carbon ions are carried out on C/C composites and the produced property changes are compared between the neutron and ion irradiations by carrying out micro-indentation test and measurements of thermal conductivity and electrical resistivity.

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Table 1
Some of the typical properties of C/C composites examined in the present study

| Grade | MFc-1 | MCI-felt | CX-2002U | PCC-2S |
|---|---|---|---|---|
| Ash content (ppm) | <20 | <20 | <10 | 8 |
| Density (g/cm$^3$) | 1.96 | 1.92 | 1.65–1.70 | 1.81 |
| Heat capacity (J/gK) | 0.71 | na | na | 0.75 |
| CTE ($10^{-6}$/K) | P:−0.9/T:12 | P:0.12/T:10.2 | P:1.6/T:5.2 | P:na/T:1.7 |
| Thermal conductivity (W/mK) | P: 640 | P: 339 | P: 368 | P:318 |
| Young's modulus (GPa) | P:100/T:0.8 | na | na | P:18.1 |
| Tensile strength (MPa) | P:400/T:3 | P:83/T:3 | na | P:29–39 |
| Bending strength (Mpa) | P:480/T: >5 | P:152/T:10 | P:39 | P:38–52/T:14–16 |
| Compressive strength (MPa) | P:216/T: >16 | P:78/T:110 | P:48 | P:57–66/T:39–49 |

P, T: parallel to or transverse with the fiber orientation or felt plane. na: Not available.

Table 2
Irradiation conditions for ions and neutrons

| Particle | Proton | Helium | Carbon | Neutron |
|---|---|---|---|---|
| Energy (MeV) | 10 | 50 | 90 | 14 |
| Damage (dpa) | 0.003 | 0.07 | 9.6 | $8 \times 10^{19}/m^2$ |
| Temperature | RT | RT | RT | RT |
| Accelerater | Cyclotron | Cyclotron | Tandem | D + T |
| Facility | TIARA | TIARA | Tokai | FNS |

## 2. Experimental procedure

Four kinds of C/C composites whose typical properties are shown in Table 1 were used for the experiment. Since the tendency of property changes is similar to each other, the results of MFC-1, one-dimensional composite are mainly shown in this paper. Specimens 6 × 6 mm in cross-section and 12 mm in length and those 10 mm in diameter and 1–3 mm in thickness were machined from a block of each material. Specimens 10 mm in diameter and 60 mm in length were machined for the measurement of electrical resistivity. For ion irradiations, 10 MeV proton, and 50 MeV helium were employed using TIARA at JAERI-Takasaki and 90 MeV carbon using TANDEM accelerator at JAERI-Tokai. 14 MeV neutron irradiation was carried out at Fusion Neutronics Facility (FNS) at JAERI-Tokai. The irradiation conditions are summarized in Table 2. For ion irradiations an oblique wedge made of the same material as that irradiated was placed in front of a specimen to be irradiated so that the damaged area is searched as a function of depth, i.e., in a manner that the depth can be regarded as the distance from the edge of the specimen surface. The set-up is shown in Fig. 1.

To estimate the changes in the strength and Young's modulus of irradiated composites, micro-indentation test was carried out before and after irradiation using a Shimadzu dynamic hardness tester [5,6]. The maximum applied load was 5 gf (49 mN) in most tests. Dynamic hardness ($D_h$) was calculated using the following equation:

$$D_h = 37.84(L_{max}/d_{max^2}). \quad (1)$$

Here, $L_{max}$ is the maximum applied load (gf) and $d_{max}$, the maximum indentation depth (μm). In the case of ion irradiations, tests were carried out on the points with 10–50 μm intervals on the irradiated surface. As for the neutron-irradiated specimens the test was done on the points selected randomly bacause the defects were believed to be produced homogeneously in this case. Thermal conductivity was measured in vacuo for neutron-irradiated specimens by the laser flush method up to temperature of about 1600 K. Holding time was automatically set at 5–10 min at lower temperatures and up to about 20 min at higher temperatures. Since it is well known that the specific heat $C_p$ of graphite materials

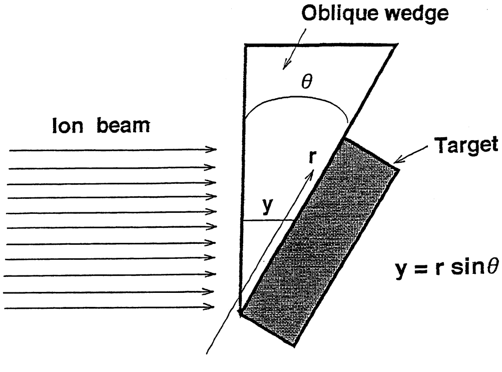

Fig. 1. Set-up for a wedge and a specimen for ion irradiation.

show almost no dependence on kinds of materials, values of $C_p$ (J/g K) were estimated here according to the equation which had been proposed by Butland and Maddison [7] for the temperature range from 250 to 3000 K.

$$C_p = 2.268 - 1.015 \times 10^{-5}T - 3.777 \times 10^{2}T^{-1} - 1.818 \times 10^{5}T^{-2} + 6.665 \times 10^{7}T^{-3} - 6.011 \times 10^{9}T^{-4}. \quad (2)$$

Electrical resistivity of neutron-irradiated specimens was measured in situ at room temperature by 4-point contact method.

## 3. Results and discussion

Fig. 2 shows Weibull plots for the dynamic hardness of 14 MeV neutron-irradiated MFC-1. Fluence ranged from 7 to 66 × $10^{18}$ n/m$^2$. The neutron beam was perpendicular to the fiber orientation. It is to be noted that the hardness decrease, i.e., strength decrease is caused by the high energy neutron irradiation for both indentation directions at least when the fluence is low as in the case of the present experiment. Such a tendency that is contrary to the general trend for the neutron-irradiated C/C composites [8] was also observed for the other composites examined here. It has been reported that on the basis of hardness measurement a decrease in the yield strength was observed for 316 stainless steel irradiated to a low fluence [9]. It was suggested in the literature that the small radiation-produced defects were cut by the movement of dislocations during the indentation process. A mechanism similar to this may be considered for C/C composites examined in the present experiment.

Fig. 3 shows the distribution of the hardness of MFC-1 irradiated by protons as a function of depth from the surface. Here, L1, L4, etc. indicate the lines along which the hardness measurements were carried out. It is also seen in the figure that the range calculated using EDEP-1 code [10] is in fairly good agreement with the point where the hardness is maximum. The variation of the hardness distribution would probably result from the inhomogeneous micro-structure of the composite (pores, boundary between the fiber and matrix, etc.) An agreement between the point of maximum hardness and the calculated range was also found in the case of He-ion irradiation. In contrast with the neutron irradiation, the strength and Young's modulus of the composites increased after the irradiation by 10 MeV protons up to 0.003 dpa. Here, the strength and the modulus were estimated from the slopes of the loading and unloading curves obtained during the indentation test, respectively, i.e., *B* and *D* in Fig. 4. Ratios of these properties after the proton irradiation to those before irradiation are summarized in Table 3. It is to be noted that all the materials showed pronounced increases in these properties, even though the fluence was very low.

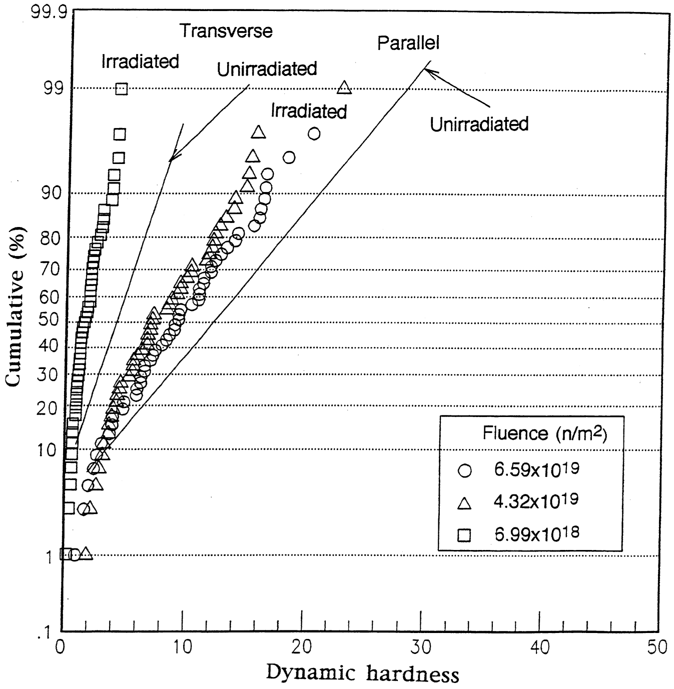

Fig. 2. Weibull plots for the dynamic hardness of neutron-irradiated MFC-1.

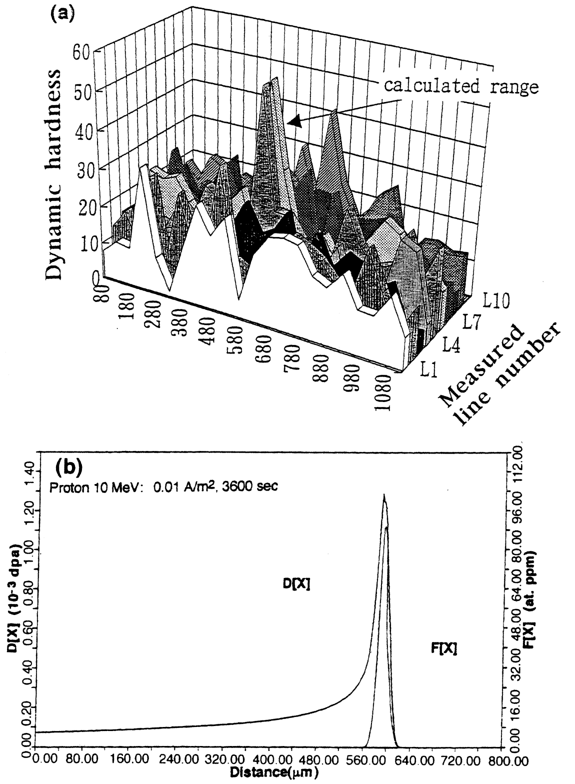

Fig. 3. Distribution of hardness as a function of depth (a) and the range calculated using EDEP-1 code (b) for proton-irradiated MFC-1.

Thermal conductivity of neutron-irradiated MCI-felt is shown as a function of temperature in Fig. 5 where one can see that the conductivity decreased after irradiation but not monotonically with the fluence. This trend was also found for the other composites. Although it is still uncertain at the moment what is the cause for this phenomenon, it may well be said that one should take into account the possibility that the high energy neutrons would result in changes in microstructure different from those caused by fast neutrons in the fission reactor. In fact, a composite, CX-2000U irradiated in a fission reactor only up to 0.01 dpa showed more than 95% decrease in the thermal conductivity [11]. Fig. 6 shows the change in the electrical resistivity in situ as a function of neutron fluence. The resistivity of CFCs as well as a nuclear graphite, which is designated as IG-110 in the figure, increase with increasing neutron fluence up to $5 \times 10^{18}$ n/m$^2$, levels off, then starts to increase at $1 \times 10^{19}$ n/m$^2$. The microscopic interpretation of this behavior is yet to be investigated.

Table 3
Ratio of the value at the damage peak of proton-irradiated C/C composites

| | MFC-1 | MCI-felt | CX-2002U | PCC-2S |
|---|---|---|---|---|
| Tensile strength | 1.7 | 1.3 | 1.8 | 1.9 |
| Young's modulus | 1.3 | 1.1 | 1.5 | 1.4 |

## 4. Conclusions

Irradiations by 14 MeV neutrons and high energy ions including protons, He and C, were performed on several grades of C/C composites for divertor applications. Micro-indentation tests were carried out on these materials together with the measurement of thermal conductivity of neutron-irradiated ones. Conclusions are: (1) The maximum of the microhardness was found near the range of ions for the carbon, which indicated that the calculation and the experiment were in agreement with each other. (2) After the irradiation by 10 MeV protons (<0.1 dpa), 10–90% increases in tensile strength and Young's modulus were observed. (3) Softening of the composites was observed in the micro-indentation test when they were irradiated by 14 MeV

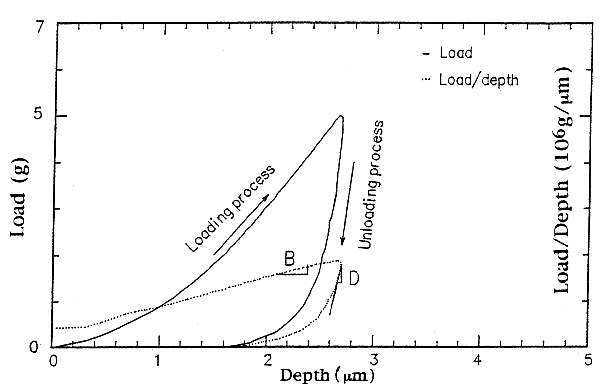

Fig. 4. Typical load–depth curves for the micro-indentation test.

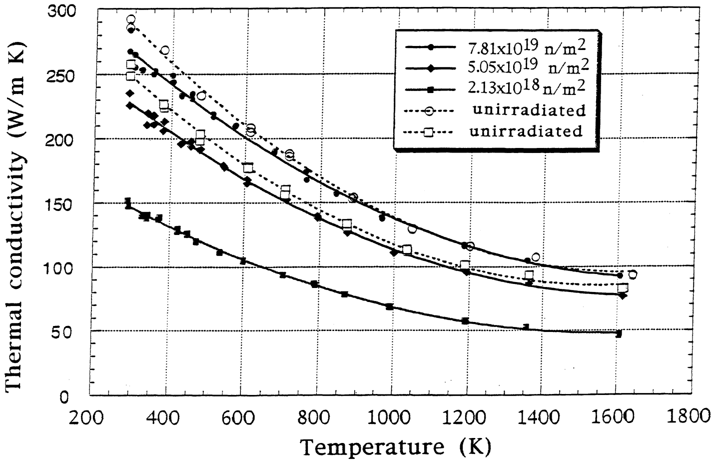

Fig. 5. Thermal conductivity of CX-2002U irradiated by neutrons as a function of temperature.

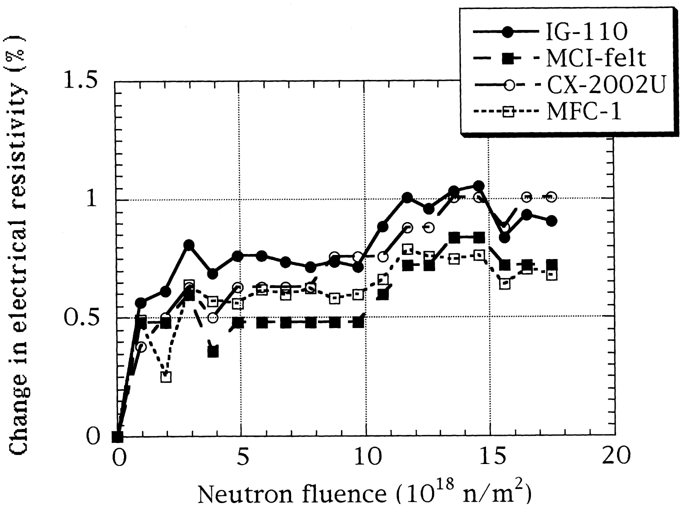

Fig. 6. Electrical resistivity change of carbon materials after 14 MeV neutron irradiations at room temperature.

neutrons up to a fluence of $10^{19}$ n/m$^2$. (4) Thermal conductivity of the materials was decreased by 14 MeV neutrons, though no monotonic decrease with regard to the fluence was observed in the present fluence range.

## References

[1] H. Takatsu et al., J. Nucl. Mater. 155–157 (1988) 27.
[2] JT-60 Team, J. Nucl. Mater. 162–164 (1989) 172.
[3] The NET Team, J. Nucl. Mater. 162–164 (1989) 14.
[4] C.M. Logan, J.D. Anderson, J. Nucl. Mater. 48 (1973) 223.
[5] K. Yasuda et al., J. Nucl. Mater. 187 (1992) 109.
[6] K. Tanaka et al., Int. J. Eng. Sci. 27 (1989) 11.
[7] A.T.D. Butland, R.J. Maddison, AEEW-R-815, 1972.
[8] M. Eto et al., J. Nucl. Mater. 212–215 (1994) 1223.
[9] N.F. Panayotou, J. Nucl. Mater. 108–109 (1982) 456.
[10] T. Aruga et al., Nucl. Instr. and Meth. B 33 (1988) 748.
[11] T. Maruyama, M. Harayama, J. Nucl. Mater. 195 (1992) 374.