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Effects of ion irradiation on the hardness properties of graphites and C/C composites by indentation tests

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A B S T R A C T

Small blocks of graphite and C/C composite materials were argon ion irradiated. The load–depth curves were obtained from the micro indentation tests before and after ion irradiation. For the C/C composite material, the indentation test was also performed perpendicular to the depth direction of ion irradiation and the distribution of the hardness properties was examined in the depth direction. As a result, hardness properties of the carbon materials were able to be expressed as a function of dpa values.

1. Introduction

Fine grained graphite and carbon–carbon (C/C) composite materials have been used for plasma facing components in many fusion devices and structural components or moderators in high temperature gas-cooled reactors. The property deterioration, such as decrease in thermal conductivity and changes in mechanical properties of carbon materials due to neutron irradiation damage, should be known in advance of the structural design of the reactor components. On the other hand, it is important to develop C/C composite materials in which radiation damage effects on various properties are small [1]. Accordingly, it is necessary to evaluate effects of radiation damage on mechanical properties of graphite and C/C composite materials. Some studies on mechanical properties of metals by indentation tests have been performed [2] and irradiation effects on the load vs. depth curve have been investigated [3]. In general, ion irradiation causes a distribution of radiation damage in the depth direction. According to the radiation damage, mechanical properties should change in the incident ion direction.

Indentation tests are powerful tools for evaluating mechanical properties, such as strength and modulus, of carbon materials. Combining micro indentation tests with ion irradiation gives us a simple and quick tool for selecting some candidate graphite or C/C materials from many grades. Four grades of graphite and two grades of C/C composite were used as test materials. Indentation tests for all materials were conducted in the same direction as the ion irradiation. One of the C/C composites was tested via micro indentation in order to examine hardness properties in the depth direction of ion irradiation. The dpa values were determined dependent upon the depth direction of ion irradiation.

The dpa dependence on the hardness properties was obtained from the above information. From the indentation test the parameter *B*, related to the strength, the parameter *D*, related to the Young's modulus, and the apparent hardness HA, which is closely related to the compressive strength, were determined. These hardness parameters were examined before and after ion irradiation. In order to examine mechanical properties of a small region in the graphite block, the load level was made as low as possible. The purpose of this paper is to examine the dpa dependence of hardness properties of carbon materials obtained from the indentation test for a single specimen.

2. Experimental

2.1. Tested materials

Four grades of graphite (IG-430U, IG-430UHP, ISO-88 made by Toyo Tanso Co., Ltd. and PD-330S made by Hitachi Chemical Co., Ltd.) and two grades of C/C composites (CX-2002U made by Toyo Tanso Co., Ltd. and PCC-2S made by Hitachi Chemical Co., Ltd.), were selected. Their properties are listed in Table 1. IG-430U graphite is a fine grained isotropic material and IG-430UHP is a higher purity version of grade IG-430U, having been heated treated to 3573 K. ISO-88 graphite is the material used for electrical discharge machining. PD-330S is almost the same as IG-430U.

CX-2002U and PCC-2S materials are the two dimensional C/C composites which have been used for fusion devices, such as the JT-60U (see Table 2).

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Table 1
Carbon materials tested

Material	Brand	Apparent density (kg/m^3)	Young's modulus (Gpa)	Bending strength (Mpa)	Compressive strength (Mpa)		Hardness (kg/mm^2)
Graphite	IG-430U	1820	10.8	53.9	83.3		26.25
	IG-430UHP	1820	6.7	43.6	82.9		27.37
	ISO-88	1915	12.7	92.8	181		43.45
	PD-330S	1820	9.6	41	82		21.42
C/C composite	CX-2002U(Ax)	1670	11	47	49.0	54.0	15.62
	CX-2002U(Per)		3.4	17			15.22
	PCC-2S(Ax)	1830	14	32	47.4	43.5	20.69
	PCC-2S(Per)		6	12			18.60

Table 2
Ion irradiation effects on hardness properties (*B*, HA) of carbon materials tested

Material	Unirr B/GPa-15 g	Unirr HA/kgmm^{-2}	Irr B/GPa-15 g	Irr HA/kgmm^{-2}	BI/BU	HAI/HAU	dpa
IG-430U	3.97	26.25	4.96	31.33	1.25	1.19	5.15E−04
IG-430UHP	4.34	27.37	5.16	22.58	1.19	0.82	5.42E−04
ISO-88	7.2	43.45	10.8	45.77	1.50	1.05	5.15E−04
PD-330S	3.75	21.42	4.61	18.74	1.23	0.87	5.61E−04
CX-2002U-A	3.08	15.62	5	28.69	1.62	1.84	5.20E−04
CX-2002U-P	3.5	15.22	4.37	23.14	1.25	1.52	5.42E−04
PCC-2S-A	4.28	20.69	4.36	32.79	1.02	1.58	5.15E−04
PCC-2S-P	4.87	18.6	6.6	21.45	1.36	1.15	5.50E−04

2.2. Micro indentation tests

The micro indentation test was performed by using the dynamic hardness tester (DUH-201) made by Shimadzu Corp. In the indentation test two load levels (19.6 mN, 147 mN) were mainly used. The load level of 19.6 mN (2 g) gives us about 2 μm depth. The load level of 147 mN (15 g), which makes about a 5 μm deep indent, was used for the normal measurements. Its standard deviation was comparatively low and was considered to be representative over a narrow range. However, the properties in a small region were evaluated using a 19.6 mN load because the indented depth was not so deep, which was especially beneficial for the measurements in the depth direction. The indentation tests were done on the ion irradiated surface in case of graphites and one C/C composite (CX-2002U). As for a C/C composite (PCC-2S), the indentation tests were conducted on the ion irradiated surface and the perpendicular to it.

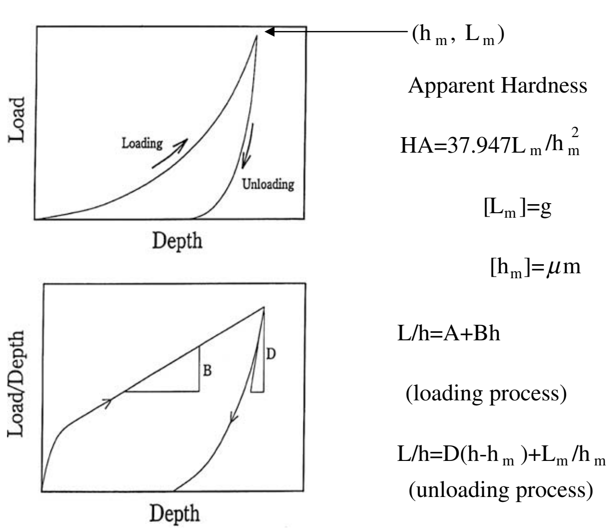


Fig. 1. Indentation tests consist of loading and unloading processes.

2.3. The parameters in the indentation test

The indentation test consists of a loading and unloading process. The upper part of Fig. 1 indicates load vs. indented depth relation. From the maximum loading point the apparent hardness value is defined. The load vs. depth curve can be redrawn as a load/depth vs. depth curve, as shown in the lower part of Fig. 1. The parameter *B* which is related to the strength is defined as the slope at the maximum loading point in the loading process of the *L*/*h* vs. *h* curve. On the other hand, the parameter *D* (which is closely related to the Young's modulus) is defined as the slope at the maximum point in the unloading process of the *L*/*h* vs. *h* curve.

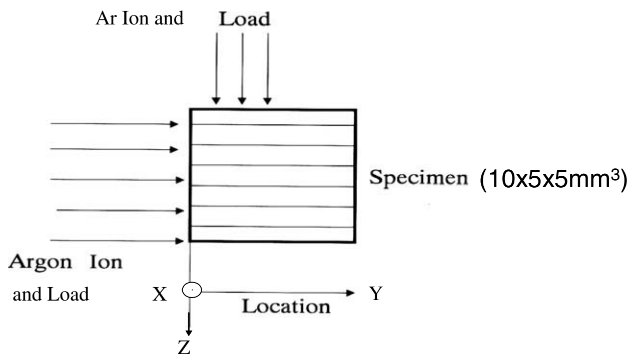
2.4. Ion irradiation

Argon ions ($^{40}Ar^{8+}$) with 175 MeV and 1 μA were used for irradiation of the carbon materials. The beam flux was 2.6×10^8 ion/mm^2 s. Ion irradiation was performed by using AVF cyclotron, TIARA, Takasaki, JAEA. Fig. 2 shows the directions of ion irradiation and loading to the carbon materials tested. In the case of the graphites and a C/C composite (CX-2002U), the direction of ion irradiation and loading were the same. However, in the case of PCC-2S (C/C composite), there were two cases, *Z* and *Y*. The measurements of PCC-2S in the *Y*-direction gave us the distribution of hardness properties in a small region of the carbon block.

3. Results and discussion

3.1. DPA calculation

Calculation of dpa was done by using the TRIM-98 code [4]. The following assumptions were made in the dpa calculation: the density of carbon was 1.9 g/cm^3, the displacement threshold energy was 37 eV, and the binding energy was 15 eV [5].



Case	Argon Ion	Load	Material
1	X(Y,Z)	X(Y,Z)	G
2	Y	Y	C/C-1
3	Z	Z	C/C-1
4	Z	Z, Y	C/C-2

G=Graphites, C/C-1=CX-2002U, C/C-2=PCC-2S

Fig. 2. Specimen geometry and directions of ion irradiation and loading. (Y = fiber axis = perpendicular).

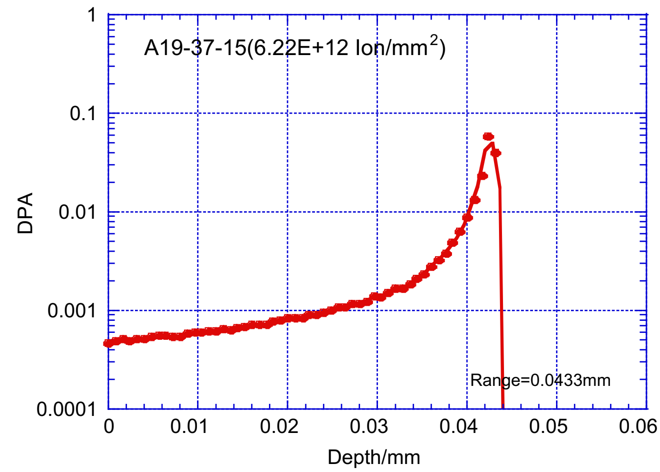


Fig. 4. DPA value as a function of depth(2):0–0.06 mm.

Fig. 3 shows dpa values as a function of the indented depth expressed in a linear scale. If dpa is expressed by a log scale, Fig. 4 is obtained. The range the argon ion can penetrate was calculated to be 0.0433 mm. Fig. 4 gives dpa distribution in the depth direction. From this curve the hardness properties (HA, *B*, *D*) can be evaluated for each depth level up to the range over which the dpa was zero. The dpa values for each hardness property were determined as the value at the depth of the indenter for its loading (19.6 mN).

3.2. Indentation hardness properties

Fig. 5 indicates an example of argon ion irradiation effect on the load vs. depth relation for CX-2002U C/C composite. Clearly, the apparent maximum depth and the residual depth decrease due to argon ion irradiation. The hardness parameters HA, *B* and *D* were obtained from the curves in Fig. 5 through the analysis of Fig. 1.

The dpa values for the carbon materials tested were determined by the dpa vs. depth curve at the deepest position of the indenter. It is known that the parameter *B* is related to strengths and HA, and the parameter *D* is related to the Young's modulus of carbon materials [6–7]. Parameters HA, *B* and *D* increase due to ion irradiation, except for HA values of IG-430UHP and PD-330S. It has proved that there exists a close relationship between the parameter *B* and strengths through the apparent hardness HA. It turned out that a similar relationship between the parameter *D* and Young's modulus was found. Therefore, it was thought that the parameters *B* and *D* can be used as indices of the corresponding mechanical property, even after ion irradiation. However, the relationship of the parameter to a mechanical property must be taken into consideration before and after ion irradiation. If the load used is 19.6 mN, the indented depth is known to be below 3 μm. In order to check the distribution of *B* and *D* values in the depth direction due to ion irradiation the load of 19.6 mN was used; it is believed to represent a mechanical property in a comparatively small region of carbon materials. Fig. 6(1) and (2) show the relationship between the compressive strength and HA, and the relationship between HA and the parameter *B*, which give roughly linear data plots. According to Fig. 7, the correlation of the parameter *B* to strengths, the correlation with compressive strength is better than that with bending strength.

The parameter *D* has a good correlation to Young's modulus, as shown in Fig. 8, though the correlation of graphites is different from that of C/C composites.

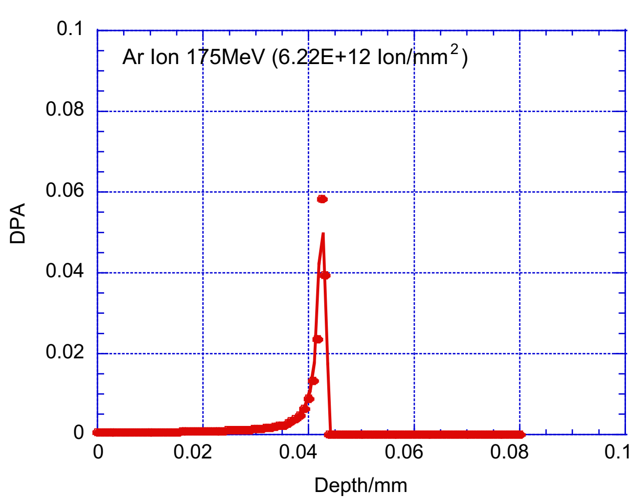


Fig. 3. DPA value as a function of depth(1):0–0.1 mm.

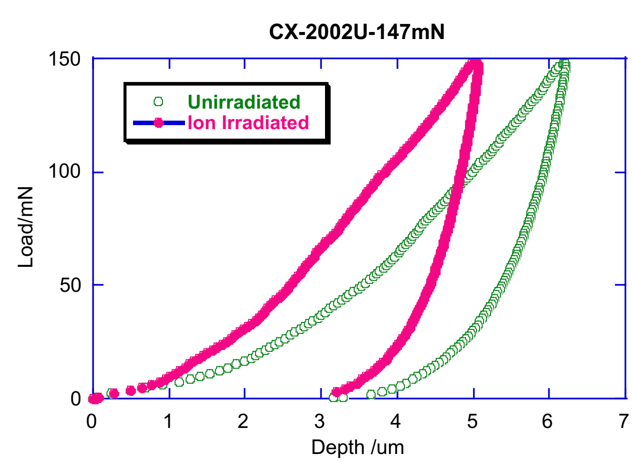


Fig. 5. An example of load vs. depth curve for CX-2002U (L = 147 mN).

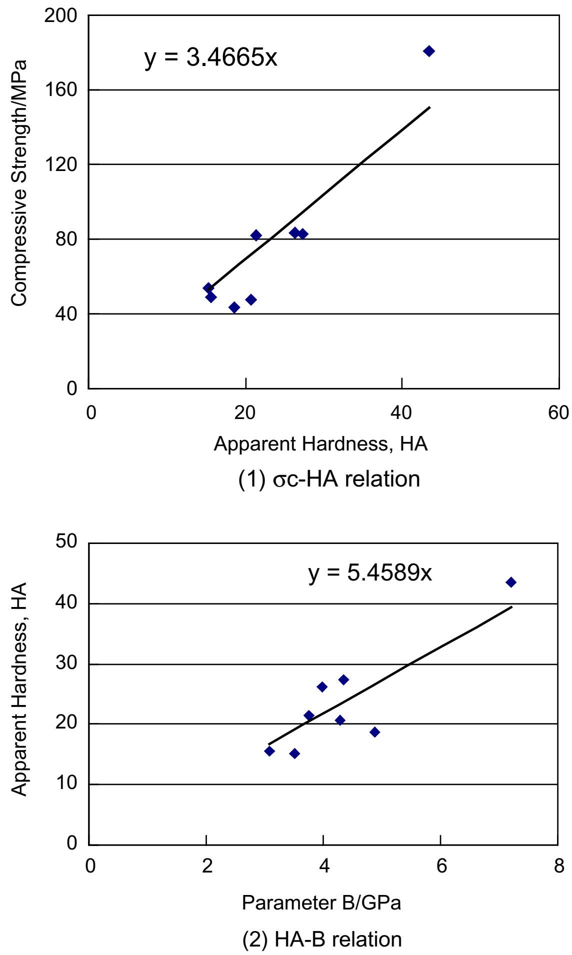


Fig. 6. Compressive strength vs. apparent hardness and apparent hardness vs. parameter *B* relations for carbon materials tested.

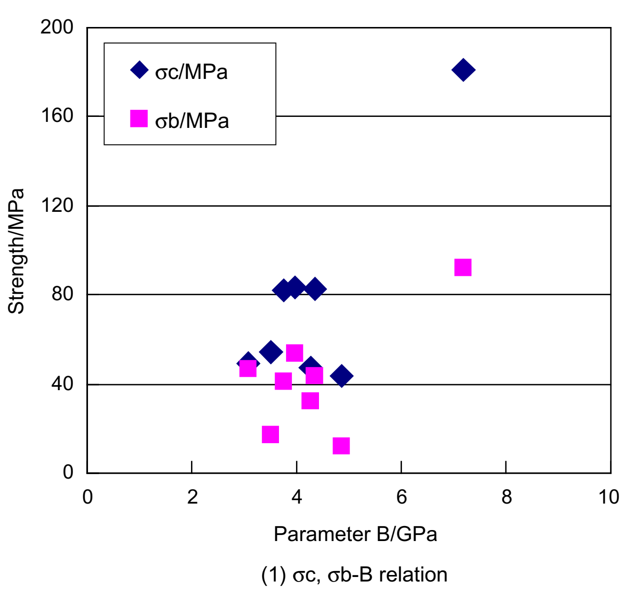


Fig. 7. Compressive and bending strength vs. parameter *B* relation for carbon materials. (σ_C = compressive strength, σ_b = 4-point bending strength).

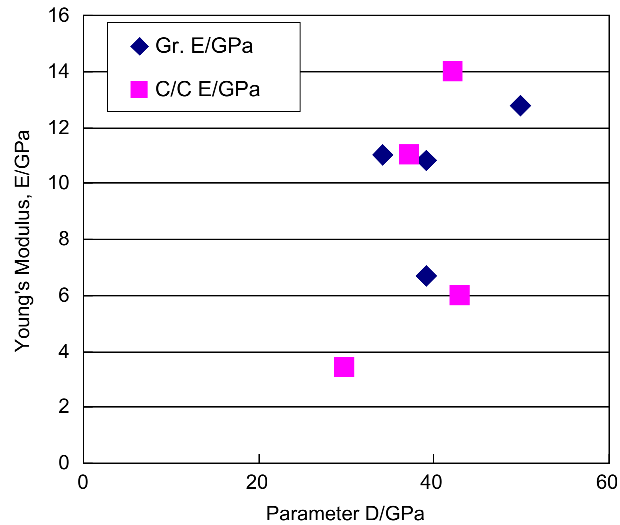


Fig. 8. Young's modulus vs. parameter *D* relation for carbon materials.

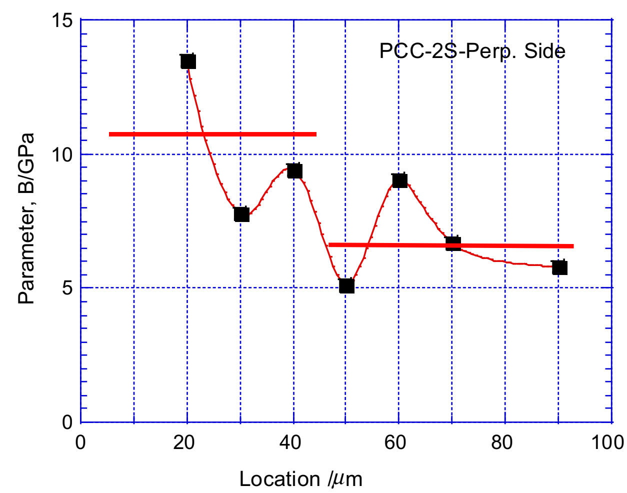


Fig. 9. Parameter *B* as a function of location.

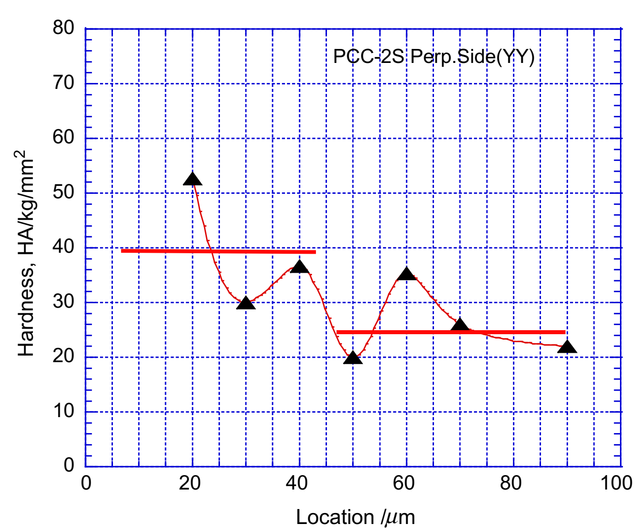


Fig. 10. Apparent hardness value as a function of location.

3.3. Changes in distribution of hardness properties after ion irradiation

Fig. 9 indicates changes in the parameter *B* for PCC-2S material as a function of location in the depth direction (perpendicular to the fiber axis) after ion irradiation. In this case, the indentation test was conducted in the *Y-direction* of Fig. 2. It should be understood that the upper level of the *B* values in Fig. 9 go down to the lower level over the range of 0.0433 mm. Fig. 10 shows changes in the apparent hardness HA for PCC-2S material as a function of location in the depth direction. This is the same as Fig. 9.

On the other hand, changes in the parameter *D* for PCC-2S material are indicated as a function of location, as shown in Fig. 11. In this case the variation of the parameter *D* is rather small.

Apparent hardness values of various grades of carbon materials are shown in Fig. 12 as a function of dpa, including neutron irradiation effects on Vickers hardness of different grades of graphite from the literature [8]. Although the definition of the hardness value is different (Vickers vs. micro indentation), it would be possible to compare the results of various irradiations if the same method for determining hardness values was used. If the hardness ratio is plotted as shown in Fig. 13, data scatter in various grades of materials are comparatively small.

The parameters *B* and *D* are indicated as a function of dpa as shown in Figs. 14 and 15. It turns out that data scatter of the parameter *D* is smaller than that of the parameter *B*. The reason for this is not clear. However, it is believed that the microstructure correlates better to parameter *B*, as compared with parameter *D*.

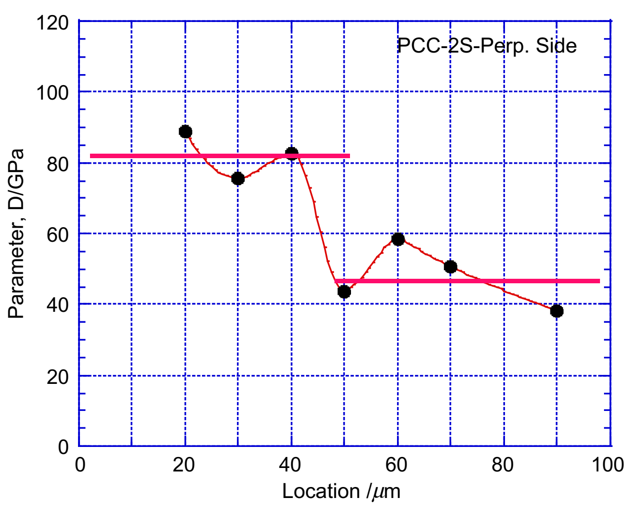


Fig. 11. Parameter *D* as a function of location.

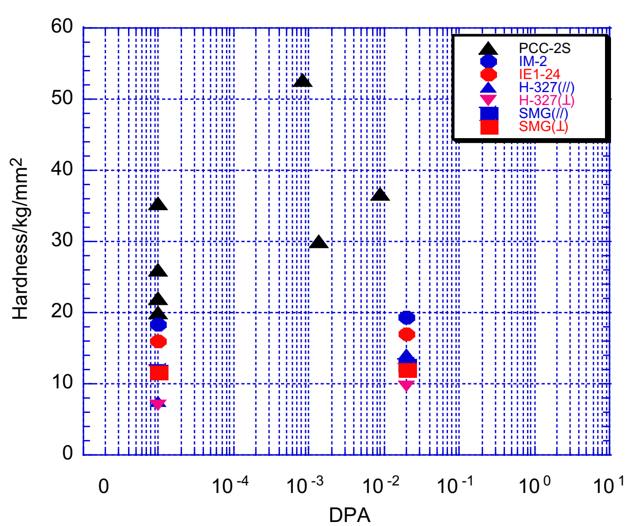


Fig. 12. Hardness values as a function of dpa:HA for PCC-2S and Vickers hardness for other graphites. (Neutron fluence for IM-2, IE1-24, H-327 and SMG graphites was 3×10^{19} n/cm^2 (1 MeV), which was estimated as about 0.02 dpa.)

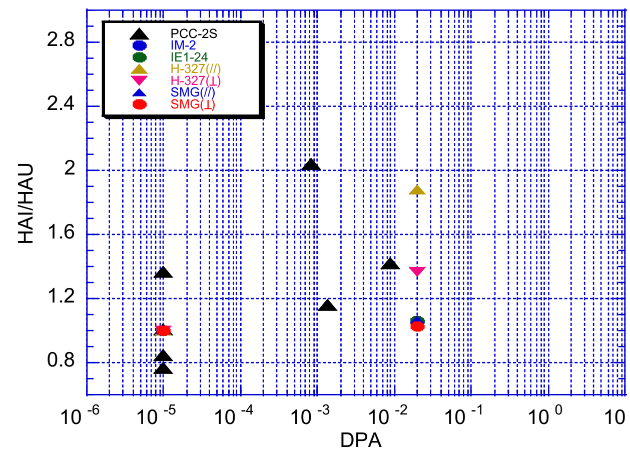


Fig. 13. Hardness ratio as a function of dpa.

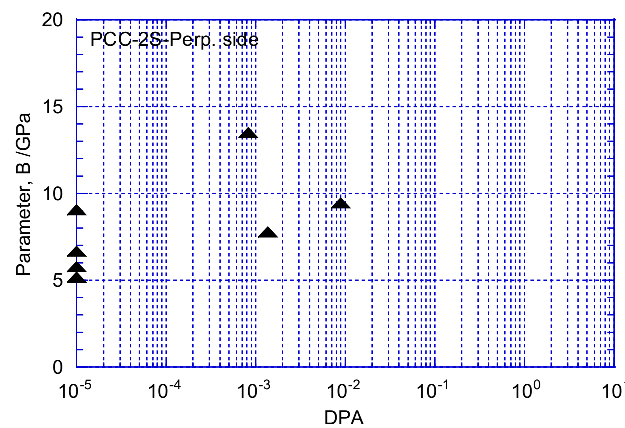


Fig. 14. Parameter *B* as a function of dpa.

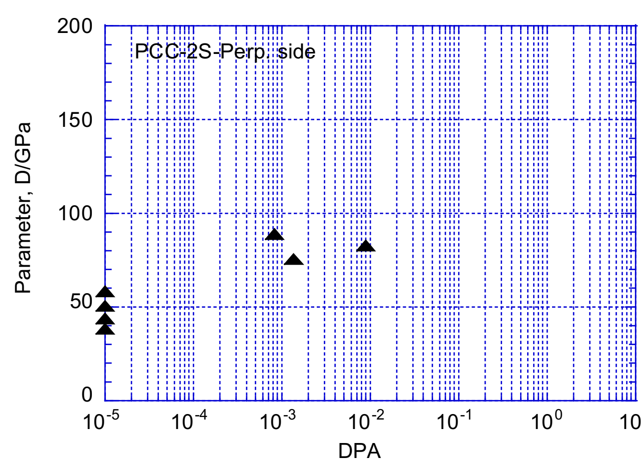


Fig. 15. Parameter *D* as a function of dpa.

4. Conclusions

The effect of argon ion irradiation on the mechanical properties of four grades of graphite materials and two grades of C/C composite materials were examined by micro indentation tests. The apparent hardness HA and the hardness property parameters *B* and *D* have been discerned from the loading and unloading processes in the indentation test before and after argon ion irradiation. The parameters *B* and *D* are believed to be good indices of mechanical properties, especially compressive strength and Young's modulus, respectively.

The parameters *B* and *D* increased due to argon ion irradiation. It has been proven that changes in mechanical properties as a result of radiation damage, as measured over a small region of a carbon block, may be detected and examined in detail by using micro indentation testing. The results obtained here indicate that the hardness properties can be expressed as a function of dpa, including neutron irradiation effects.

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