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Effects of prestresses on mechanical properties of isotropic graphite materials

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Abstract

Graphite materials which are used for plasma facing components and other components are subjected to stresses due to the high heat flux from the fusion plasma. Some mechanical properties of graphite materials can change due to the prestresses. The property changes should be considered for the design of the plasma facing components. The purpose of this study is to examine the effects of prestresses on the mechanical properties of isotropic graphite materials. Compressive prestresses were applied to two kinds of isotropic fine-grained graphites (IG-430 and IG-11) at 298 K (both), 1873 K (IG-11), 2273 K (IG-11) and 2283 K (IG-430). As a result, the decrease in Young's modulus for IG-430 due to high-temperature prestressing was 56% which was much larger than the 6.4% that was due to prestressing at 298 K. The results for IG-11 were the same as those for IG-430 graphite. This finding was considered to be due primarily to a difference in degree of the preferred orientation of crystallites in the graphite on the basis of the Bacon anisotropy factor (BAF) obtained from X-ray diffraction measurement of the prestressed specimens. Furthermore, high-temperature compressive prestressing produced an increase in the strength of the isotropic graphite, although room temperature prestressing produced no such effect. The results obtained here suggest that the isotropic graphite which is subjected to high-temperature compressive stresses can become anisotropic in service.

1. Introduction

It is known [1–6] that different properties, such as Young's modulus and mechanical strength, change if compressive or tensile prestresses are applied to graphite materials at room temperature. When the graphite materials are used for reactor components, various stresses are produced in the structural components of the nuclear reactors [7]. The stresses are due to dead weight, inhomogeneous distribution of temperature and neutron flux in the anisotropic graphite block (leading irradiation-induced stresses), and external loads, such as an earthquake. Even in plasma facing components for fusion reactors, there are inhomogeneous distribution of temperature and neutron flux in the graphite tiles that are faced to the plasma since a joining structure should be adopted with copper on the opposite side of the graphite tiles [8]. The gradients of temperature and neutron flux generate strains and stresses even after shut down of the reactor. Such internal stresses and strains can break graphite tiles when the stresses attain to the fracture strength. Stresses generated in the graphite tiles should be calculated by using Young's modulus and other mechanical and physical properties. Consequently, in order to evaluate the correct stresses produced in the graphite tile it is important to know how the mechanical properties change when the reactor component graphite materials for fusion reactors are subjected to stresses.

This paper describes results of the influence of prestresses at 298 K, 1873 K, 2273 K and 2283 K on the Young's modulus and strength of the nuclear grade isotropic graphites and examines the reasons why the material properties change due to prestresses. The compressive stresses applied to the specimens at each temperature were about 90% (298 K) and over 95% (2273 K, 2283 K) of the compressive strength at room

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temperature. After removing stresses, changes in Young's modulus and strength at room temperature have been evaluated and changes in microstructures, such as crystal orientation, due to compressive prestresses have been examined.

2. Experimental

2.1. Materials tested

Two kinds of fine-grained isotropic nuclear grade graphites, IG-430 and IG-11 for fusion plasma components, made by Toyo Tanso were used as test materials. Table 1 shows typical properties of the tested materials. The specimen sizes were 10 mm in diameter and 20 mm in height for IG-430 and $5 \times 6 \times 10$ mm^3 for IG-11.

2.2. Compressive test

The compressive tests for IG-430 were conducted at room temperature before the prestress was applied. The compressive load was applied at a crosshead speed of 0.1 mm/min by using a universal testing machine with a maximum load capacity of 98 kN. Polyethylene sheet was used to reduce the frictional resistance between the edge surface of the specimen and the pressure block.

The compressive prestress of 90% of the average compressive strength (77.5 MPa) at room temperature was applied using results of the compressive tests at room temperature before prestressing. At 2283 K the compressive prestress of 95% of the average compressive strength at room temperature was applied to the specimen. In this case the actual prestress was deduced to be less than 95% of the high temperature fracture strength because the graphite material has a higher strength at high temperature.

The compressive tests for IG-11 were performed at a crosshead speed of 0.5 mm/min using a universal mechanical testing machine in an atmosphere of purified nitrogen in an ultra-high temperature furnace. The specimens were kept at 293 K, 1873 K and 2273 K for about 15 min and the compressive tests were conducted. The specimens were kept for 1 min at the predetermined stress levels. All the measurements were done at room temperature after unloading.

Table 1
Typical properties of tested materials

Material	IG-430 Graphite	IG-11 Graphite
Apparent density (kg/m^3)	1820	1770
Young's modulus E_0 (GPa)	11.7	9.8
Electrical resistivity (μΩ m)	9.0	11
Specimen size (mm^3)	ϕ 10 × 20	5 × 6 × 10

2.3. Young's modulus measurement

Young's modulus, E, was determined from the ultrasonic wave propagation velocity, v, using the ultrasonic detector (FD-1800, Mitsubishi) with 2 MHz ultrasonic vibrator and receiver before and after applying prestresses, and the following equation:

$$E = \rho v^2,$$

where ρ is the apparent density of the material.

2.4. Diametral compressive test

The sample for this test was made by cutting from the IG-430 graphite specimen after measuring Young's modulus. The specimen size was 10 mm in diameter and 6 mm in length. An arc type indentor with inner diameter of 11.5 mm was used. Machine grease was applied to the specimen as a lubricant to reduce the frictional resistance between the indentor and the pressure block. The test was performed at a crosshead speed of 0.5 mm/min.

2.5. X-ray diffraction experiment

In order to investigate changes in the microstructure due to compressive prestressing the crystal orientation before and after compressive prestressing for IG-430 graphite was analyzed using a X-ray diffractometer. The X-ray diffractometer was a rotating anode type Rartaflex RU-300 (Rigaku). In the diffractometer, copper was used as an anode and the amp voltage was 40 kV and the amp current was 80 mA. The diffraction intensity was measured by a scintillation counter and recorded automatically. In the measurement of a (0 0 2) diffraction pattern the slits for diffusing, scattering and receiving beams were 1°, 1° and 0.3 mm, respectively. The scanning speed was 2°/min and sampling time was 0.06 s.

Bacon has measured the distribution of the (0 0 2) diffraction intensity for polycrystalline graphites by using an X-ray camera and expressed as $I(\phi)$ the intensity of the direction making an angle ϕ to the symmetrical axis [9]. $I(\phi)$ was called the orientation function and has been used as a parameter which shows the crystal orientation in the bulk material. In this experiment the orientation function $I(\phi)$ of the specimens was measured by using the X-ray diffractometer. The specimen was a thin rod, about 1 mm in dia. × 8 mm, cut from the direction perpendicular to the applied stress, as shown in Fig. 1. At first, the specimen was set parallel to the central axis of goniometer and the angular dependence of (0 0 2) diffraction pattern was examined. Secondly, the detector was fixed at the peak position of the (0 0 2) diffraction line and the (0 0 2) diffraction patterns were continuously measured from 0° to 360° as shown in

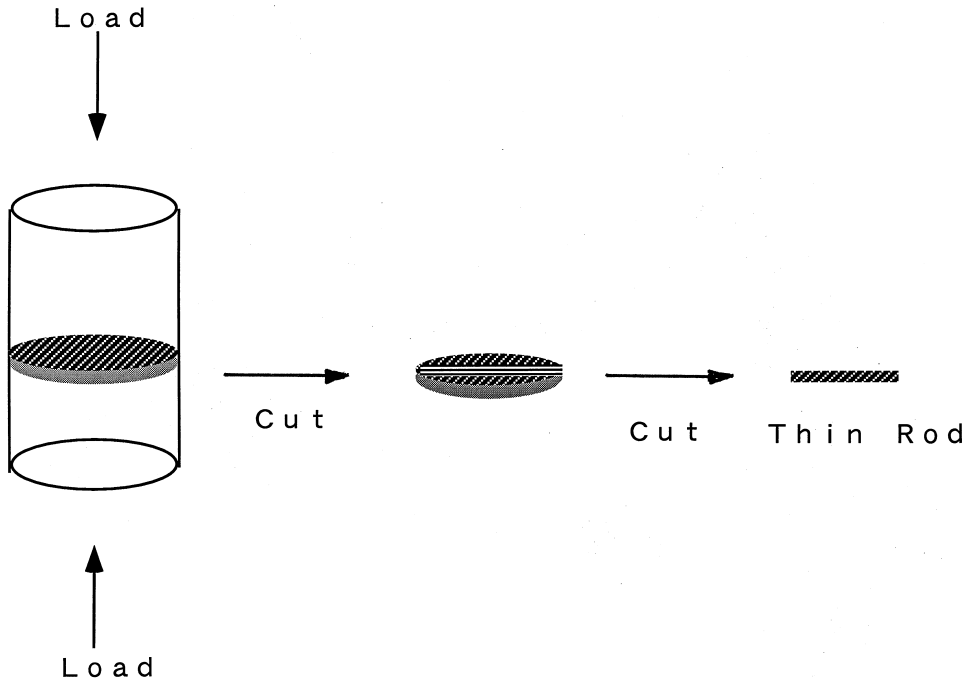

Fig. 1. Cutting plan of specimens for X-ray analysis after compressive test.

Fig. 2. The measurement conditions were 40 kV–80 mA, 45°/min for scanning speed and 0.2° for sampling width. When the specimen was rotated from 0° to 360°, four pairs of $I(\phi)$–ϕ curves from 0° to 90° were obtained with corrected background. The average of the four curves were determined and the value of ϕ for which $I(\phi)$ is the maximum was defined as 0°. The use of the average curve reduces any effect of deviation from the gonio-axis in setting up the specimen and eliminates deviation from the molding axis and the symmetry axis.

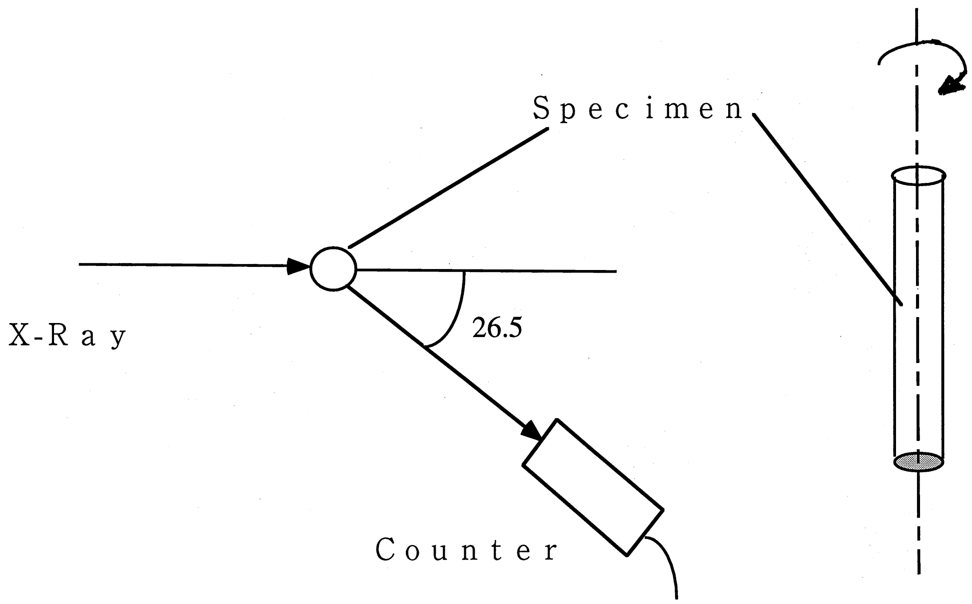

Fig. 2. Method of measurement for orientation function by thin rod specimen.

3. Results and discussion

3.1. Residual strain after compressive prestressing

The average compressive fracture stress was 77.5 MPa from the result of compressive tests (nine specimens) conducted at room temperature. The compressive prestress which corresponds to about 90% of the average fracture stress at room temperature was applied to the specimens. At about 2283 K the compressive prestress which corresponds to about 95% of the average fracture stress at room temperature was applied to the specimen. The dimensional changes before and after compressive prestressing are shown in Tables 2 and 3. Even if an applied prestress is small when it is applied to the polycrystalline graphite, after removing stress the dimension of the specimen does not completely recover and a residual strain is produced. In this case the residual strain is compressive and the specimen elongation is perpendicular to the applied stress. The residual strain of the material prestressed up to 69.8 MPa ($0.9\sigma_f$) at room temperature was only 0.16%. In contrast to this, the residual strain of 8.1% was observed for the material prestressed up to 73.6 MPa ($0.95\sigma_f$) at high temperature. Although an increase in apparent density was hardly seen for the material prestressed at room temperature, the density increase of 0.3% was observed for the material prestressed at high temperature. It is considered that the residual strain of compressive prestressed materials consists of deformation of crystal grains, formation of cracks and so on. On the other hand, for the materials prestressed at high temperature a transverse residual strain was observed together with large longitudinal one. Although there is some uncertainty on grain deformation in the materials prestressed at high temperature, this deformation could be large. It is considered that deformation of pores by movement or rotation of grains can contribute to the residual strain.

3.2. Change in Young's modulus

Tables 2 and 3 also indicate changes in Young's modulus before and after compressive prestressing. It is generally well known [1] that the Young's modulus of graphite decreases with increasing applied compressive stress levels. The Young's modulus (E_1) of the specimen prestressed up to 69.8 MPa ($=0.9\sigma_f$) at room temperature decreased 6.4% in the direction of applied stress. In contrast with this a decrease of 56% was seen for the Young's modulus of the specimen prestressed up to 73.6 MPa ($=0.95\sigma_f$) at high temperature. The decrease in Young's modulus of the specimen prestressed at high temperature is extremely large compared with that of the specimen prestressed at room temperature, as well as the case of residual strain. It has been reported [4] that there is a correlation between Young's modulus and residual strain of the graphite prestressed at room temperature, that is, Young's modulus ratio (E/E_0) before and after compressive prestressing decreases with increasing longitudinal residual strain. It has also been reported [5] that there is little dependence of the grade of graphite and cutting direction on the Young's modulus ratio. This suggests that Young's modulus decreases due to the same factors. There is a large difference in residual strain of the specimens prestressed at room temperature and high temperature. The reason for the decrease in Young's modulus might be due to some different factors. Therefore, it cannot be determined if there is a proportional relation between Young's modulus and residual strain or not. However, it was shown that Young's modulus decreases with increase of residual strain. On the other hand, the Young's modulus of the specimen was also measured in the direction perpendicular to the applied stress. As shown in Tables 2 and 3, the Young's modulus (E_2) of the specimen decreased in any case as compared with before prestressing and it indicates that the Young's modulus in the perpendicular

Table 2
Effects of compressive prestress on the Young's modulus and length for IG-430 graphite

Temp. (K)	σ_f (MPa)	σ_a (MPa)	E_1/E_0	E_2/E_0	$\Delta L/L$ (%)	BAF
293	77.5	0	1	1	0	1.06
293	77.5	69.8	0.94	0.98	−0.16	1.45
2283	–	73.6	0.44	0.77	−8.1	2.52

Table 3
Effects of compressive prestress on the Young's modulus and length for IG-11 graphite

Temp. (K)	σ_f (MPa)	σ_a (MPa)	E_1/E_0	E_2/E_0	$\Delta L/L$ (%)
293	75	67.5	0.95	1.08	−0.41
1873	93	83.3	0.87	0.99	−0.27
2273	104	107	0.32	0.68	−10.1

Table 4
Effects of compressive prestresses on diametral fracture strength

Material	Diametral fracture strength σ_p (MPa)
Virgin	29.8 ± 1.2 (8)
Prestressed to 0.6 σ_f at 293 K	30.1 ± 0.7 (9)
Prestressed to 0.7 σ_f at 293 K	29.1 ± 0.7 (6)
Prestressed to 0.9 σ_f at 293 K	28.2 ± 2.2 (4)
Prestressed to 0.9 σ_f at 2283 K	31.0 ± 0.5 (2)

direction becomes larger than that in the applied stress direction.

3.3. Change in strength

It is generally known [1] that the tensile strength of graphite decreases with increasing compressive prestress at room temperature. In this study the diametral compressive strength was determined by using disk specimens. Since the contact area between the specimen and an arc type indentor could not be measured in this experiment, the tensile strength could not be deduced from the diametral compressive test. If the concentrated load, P, is applied to the disk of radius, R, and thickness, t, the tensile stress σ_p $(= P/\pi Rt)$ on the load line was defined as the diametral compressive strength and examined at the different prestress levels as shown in Table 4. In this table the column, σ_p shows the average ± standard deviation (number of specimen). The strength of the specimen prestressed at room temperature changed little in the perpendicular direction to the applied stress. However, the strength of the specimen prestressed at high temperature indicated a slight increase.

3.4. Change in crystallite orientation

In order to investigate changes in the orientation of the crystallite due to prestresses, an X-ray diffraction experiment has been carried out [9]. The diffraction patterns of (0 0 2) for IG-430 specimens have been obtained as a function of rotating angle of the specimens which were not loaded and were prestressed at room temperature and high temperature, respectively. The orientation function $I(\phi)$ was obtained based on the above diffraction curves. After correction of the background of the figures the four curves of $I(\phi)$–ϕ from 0° to 90° were averaged. The value of ϕ for which $I(\phi)$ is a maximum was taken as 0 and three $I(\phi)$ curves are shown in Fig. 3 for value of ϕ from 0° to 90°. The orientation function was expressed as a relative strength and the maximum of $I(\phi)$ taken as unity.

The curves in Fig. 3 were approximated by the fourth order equation:

$$I(\phi) = d_0 + d_1\phi + d_2\phi^2 + d_3\phi^3 + d_4\phi^4,$$

where d_0–d_4 are constant. As a result, the following equations are obtained: $I(\phi) = 0.999 + 0.013\phi - 0.186\phi^2 + 0.093\phi^3 - 0.008\phi^4$ for original material, $I(\phi) = 0.998 + 0.004\phi - 1.406\phi^2 + 1.188\phi^3 - 0.287\phi^4$ for

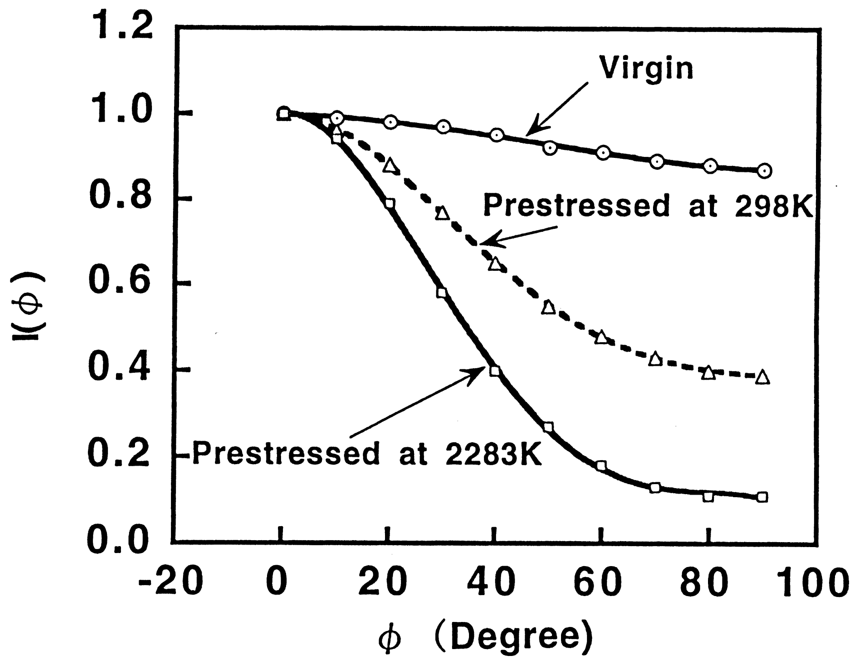

Fig. 3. Orientation function $I(\phi)$ as a function of rotating degree for the axis of thin rod specimen.

prestressed material at room temperature, $I(\phi) = 1.002 + 0.066\phi - 2.865\phi^2 + 2.736\phi^3 - 0.746\phi^4$ for prestressed material at high temperature.

Bacon expressed theoretically the coefficient of thermal expansion based on the orientation function and defined a factor that quantitizes anisotropy of a material, that is, the Bacon Anisotropy Factor (BAF) [10]. The equation which gives BAF values based on the orientation function is shown in the following:

$$\mathrm{BAF} = \frac{2\int_0^{\pi/2} I(\phi)\,\cos^2\,\phi\,\sin\,\phi\,\mathrm{d}\phi}{\int_0^{\pi/2} I(\phi)\,\sin^2\,\phi\,\mathrm{d}\phi}.$$

The value of BAF is unity if the material is completely isotropic and becomes large with increase of anisotropy. Since the measurement error of BAF becomes large when the material has strong preferred orientation, it is not desirable to use the BAF value. It is said that BAF is an effective factor as a measure that indicates a little anisotropy for isotropic nuclear graphites. The BAF values calculated from the above equation are shown for each material in the column at the right edge in Table 2. The BAF values obtained from X-ray diffraction method are 1.06 for the original material, 1.45 for the prestressed at room temperature, and 2.52 for the prestressed at high temperature. It means that grains in the graphite rotate due to compressive prestressing and the orientation of the basal plane of crystallites takes a preferred orientation in the direction perpendicular to the applied stress.

Consequently, changes in the orientation of the basal plane of crystallites decreases the Young's modulus in the direction of applied stress and increases the Young's modulus in the direction perpendicular to the applied stress. However, it is considered from the fact that Young's modulus decreases also in the perpendicular direction to the applied stress that there are not only changes in the orientation of crystallites but there are other mechanisms, e.g. crack formation that also contribute to a decrease in Young's modulus.

4. Conclusions

Compressive stresses have been applied to two kinds of isotropic fine-grained graphites (IG-430 and IG-11) at 298 K (both), 1873 K and 2273 K (IG-11), and 2283 K (IG-430). Changes in the Young's modulus and the orientation of crystallites have been investigated at room temperature before and after prestressing. As a result, the following conclusions were drawn.

1. When the compressive prestresses are applied at 298 K, residual strain is produced and the Young's modulus for both graphites decreases. The residual strain increases and the Young's modulus for both graphites decreases with increasing applied stress. On the other hand, when the compressive prestresses are applied at 2273 and 2283 K, the residual strain increases markedly and the Young's modulus largely decreases. The prestress applied to IG-11 graphite at 1873 K produced intermediate effects.
2. When the compressive prestresses are applied to IG-430 at room temperature, the strength in the direction perpendicular to the applied stress change little compared to that before prestressing. The strength increases when the prestress is applied at 2283 K.
3. Changes in the orientation of the basal plane of crystallites were investigated by the X-ray diffraction method for both materials prestressed at room temperature and at 2283 K. It turned out that the orientation of the basal plane of crystallites changes due to prestresses. The anisotropy factor, BAF was 1.06 for the original material, 1.57 for the material prestressed at room temperature, and 2.52 for the material prestressed at 2283 K.
4. Crystallites rotate due to compressive prestress and the basal planes take preferred orientations in the direction parallel to the applied stress. Consequently, it is considered that Young's modulus decreases in the applied stress direction and increases in the perpendicular direction. However, it is considered from the fact that Young's modulus decreases also in the perpendicular direction that there are not only changes in the orientation of crystallites but there are other mechanisms, e.g. crack formation that also contribute to a decrease in Young's modulus.

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