Journal of Ceramic Processing Research. Vol. 14, No. 2, pp. 251~254 (2013)

J O U R N A L O F
Ceramic Processing Research

Fracture behavior of nuclear grade graphite under mixed mode I-II

Eung-Seon Kim*, Sung-Deok Hong and Yong-Wan Kim
Nuclear Hydrogen Reactor Technology Development Division, Korea Atomic Energy Research Institute, Daedeok-daero 989-111, Yuseong-gu, Daejeon 305-353, Republic of Korea

Mixed mode I and mode II fracture toughness values of five grades of nuclear graphites were measured at room temperature using a centrally notched disk specimen. The specimens were compressed diametrically with a crosshead speed of 0.125 mm/min in ambient air. The fracture loads of the nuclear graphites decreased as the notch inclination angle increased under mixed mode I-II loading. The nuclear grade graphites with the higher bulk density and coarser coke particles showed higher mode I and mode II fracture toughness. The ratios of K_{IIc}/K_{Ic} were in the range of 1.4 ~ 1.5 regardless of the coke type and size

Key words: Nuclear grade graphite, Notched disk specimen, Mixed mode I-II loading, Fracture toughness, Bulk density, Coke particle.

Introduction

Nuclear graphite is any of a grade of high purity, high density and near-isotropic graphite specifically manufactured for use as fuel elements, moderator or reflector blocks and core support structures in a high temperature gas cooled reactor. Standard specifications including the classification, processing, and properties of nuclear grade graphite are summarized in the ASTM D7219-08 and D7301-08. The former is for fuel elements, moderator or reflector blocks where neutron irradiation induced dimensional changes are a significant design consideration and the latter is for reflector blocks and core support structures where neutron irradiation induced dimensional changes are a not significant design consideration [1, 2]. Nuclear grade graphites are usually manufactured from isotropic filler cokes (petroleum or coal-tar derived). The crushed, milled and graded are blended with a coal tar pitch binder and then formed into block. After baking the block is implagnated with a petroleum pitch and re-baked to density the part. Impregnation and re-bake may occur several times to attain the required density. Graphitization typically occurs at temperatures above 2500 °C with the entire process taking six to nine months.

During reactor operation, graphite core components and core support structures are subjected to complex stresses such as combined loading from neutron irradiation induced dimensional change and thermal gradients. Moreover, static and seismic stresses act on the core components [3, 4]. Fracture mechanics-based structural integrity assessments of graphite components may require understanding the mechanisms and failure criteria of graphite under both single mode and mixed mode loading. In this study, in order to gain an understanding of the effects of mixed mode loading on the fracture behavior, the fracture toughness under mixed mode I and mode II was examined for selected nuclear graphite using a notched disk specimen.

*Corresponding author:
Tel : +82-42-868-8560
Fax: +82-42-868-8767
E-mail: kimes@kaeri.re.kr

Experimental details

Five kinds of nuclear grade graphites were used in this study: IG-110 (petroleum, isostatically molded) and IG-430 (coal-tar, isostatically modled, Toyo Tanso Co, Ltd, Japan) and NBG-17, NBG-18 (coal-tar, vibrationally molded) and NBG-25 (petroleum, isostatically molded, SGL Carbon Group, Germany). According to the ASTM D 7219-08, the IG-110 and IG-430 are classified as superfine-grained graphites, NBG-25 as a fine-grained graphite and NBG-17 and NBG-18 as medium-grained graphites. The main properties of the graphites are summarized in table 1.

Samples for optical microscopy (10 × 10 × 2 mm) were mounted using a vacuum-impregnation apparatus (Struers, EpoVac) to fill most accessible open pores with fluorescent epoxy resin and thus maintain the original structure of the graphite. The sample mounts were initially ground with a P800 grit SiC paper to prevent pulling out the graphite particles, especially the smaller particles. After grinding up to P4200 grit, rough polishing was accompanied on a low napped polishing cloth using 1 μm alumina powder. And then the samples were finally polished for a very short time on a high napped cloth using 0.05 μm alumina powder. The optical pore structure was observed using a polarized light optical microscope (Olympus, GX51).

For mixed mode I-II fracture tests, we used centrally notched disk specimens with a size of 15 mm in radius (R) and 3 mm in thickness (t). The notch length ($2a$)

Table 1. Typical properties of the graphites.

Grade	Density (g/cm^3)	Coke size (μm)	Porosity (%)
IG-110	1.77	20	21.82
IG-430	1.80	10	20.42
NBG-17	1.84	Max. 900 μm	18.87
NBG-18	1.85	Max. 1800 μm	18.27
NBG-25	1.81	Max. 60 μm	20.06

and width were 9 mm (i.e., $a/R = 0.3$) and 1 mm, respectively. The specimens were loaded diametrically using a 30 kN capacity universal testing machine with a crosshead speed of 0.125 mm/min at room temperature. Fracture toughness under mixed mode I and mode II were obtained simply changing the notch inclination angle (β) from 0 ° to 30 ° with respect to the compression load (P). The mode I and mode II stress intensity factors K_I and K_{II} can be written as [5]:

$$K_i = N_i \frac{P_c}{Rt}\sqrt{\frac{a}{\pi}}, \qquad i = I, II \tag{1}$$

The nondimensional coefficients N_I and N_{II} are function of the relative crack size, (a/R), and the crack inclination angle and Hertzian contact width. The width of contact area was measured directly tracing the length which appeared on a pressure sensitive paper (Prescale, Fuji Film Co.) between the specimen and anvils.

Results and Discussion

Fig. 1 shows the typical optical pore structure of the nuclear graphites. The open and closed pores were discernible by small difference in their grey tones. Depending on size, shape and location, three types of pore were identified. One was large acicular-shaped pore or crack in the filler particles which were formed by volumetric shrinkage during calcinations of the filler particles (Type A). They were parallel to the basal planes and normally observed in the medium-grained graphites. The other was curved or round-shaped gas entrapment pores within the binder phase which were formed during the mixing and baking stage of manufacture (Type B). Another was narrow, slit-shaped pores in the binder phase or filler/binder boundary which were formed as a result either of volumetric shrinkage on baking or of anisotropic contraction on cooling from graphitization temperatures (Type C). They were frequently connected to the Type B gas entrapment pores. Most of the Type B and C pores in the binder phase were open, whereas most of the Type A pores in the filler particles was closed. Generally, Type A and C pores had higher aspect ratio than Type B. As can be seen in Fig. 1, more small pores were uniformly distributed in the superfine- and fine-grained graphites than the medium-grained graphites.

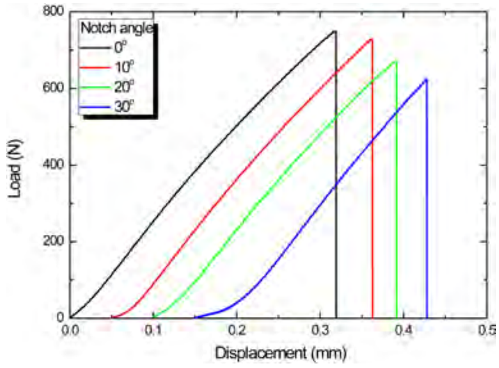

Fig. 2. Load-displacement curves with increasing notch inclination angles for IG-110.

Fig. 2 shows typical load-displacement curves for various notch inclination angles. The fracture load decreased as the notch inclination angle increased. The dependence of the fracture load on the notch inclination angle in the mixed mode loading condition

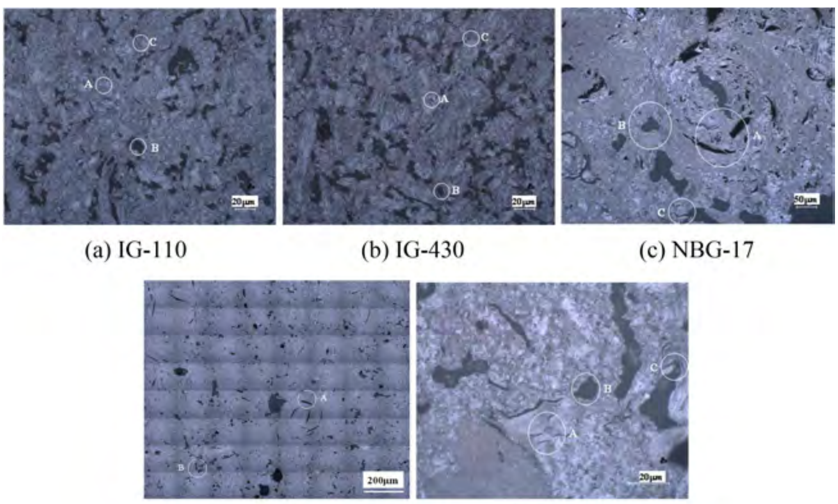

Fig. 1. Optical pore structures.

Table 2. Results of mixed mode I-II fracture toughness tests.

Grade	β (degree)	K_{Ic} * ($MPam^{1/2}$)	K_{Ic} or K_I ($MPam^{1/2}$)	K_{IIc} or K_{II} ($MPam^{1/2}$)
IG-110	0	0.87	0.64	0
	10		0.54	0.48
	15		0.42	0.70
	20		0.25	0.82
	25		0.07	0.95
	30		-0.11	1.00
IG-430	0	1.09	0.78	0
	10		0.64	0.64
	15		0.47	0.79
	20		0.30	1.00
	25		0.08	1.08
	30		-0.18	1.21
NBG-17	0	1.07	0.83	0
	10		0.70	0.63
	15		0.54	0.91
	20		0.31	1.05
	25		0.09	1.27
	30		-0.18	1.32
NBG-18	0	1.34	0.92	0
	10		0.74	0.68
	15		0.56	0.96
	20		0.35	1.19
	25		0.09	1.28
	30		-0.18	1.42
NBG-25	0	1.07	0.80	0
	10		0.66	0.60
	15		0.49	0.83
	20		0.31	1.04
	25		0.09	1.15
	30		-0.17	1.20

* Three point bending test results [9]

can be understood by examining the stress distribution of notch tip formulated by Atkinson [6]. For pure mode I (β = 0°), the tensile tangential stress is maximum but the shear stress is zero so that the crack propagates only under the influence of the tangential stress as shown in Fig. 2(a). However, for mixed mode I-II loading (0° < β < 30°), the shear stress is positive with a maximum value at around β = 30° and the tangential stress is still tensile. The sharp crack initiated by the shear deformation at the notch tip might intensify the tangential stress so that the crack could be propagated in mode I at relatively low fracture load with increasing the notch inclination angle as shown in Fig. 2(b).

The results of mixed mode I and II fracture toughness tests for five grades of nuclear graphite are summarized in table 2. The lowest value of K_{Ic} was 0.64 $MPam^{1/2}$ for the IG-110 with low density, elastic modulus and high porosity. In the case of the IG-430, NBG-17 and NBG-25 which has a higher density than the IG-110, the values of K_{Ic} were around 0.78 ~ 0.83 $MPam^{1/2}$ and higher than that of the IG-110. Although the density of the NBG-18 is nearly equal to that of the NBG-17 but the K_{Ic} value of the NBG-18 was 1.34 $MPam^{1/2}$, much higher than that of the NBG-17. The value of K_{Ic}

(a) (b)

Fig. 3. Typical crack paths under (a) mode I and (b) mode I-II (b = 20°) for IG-110.

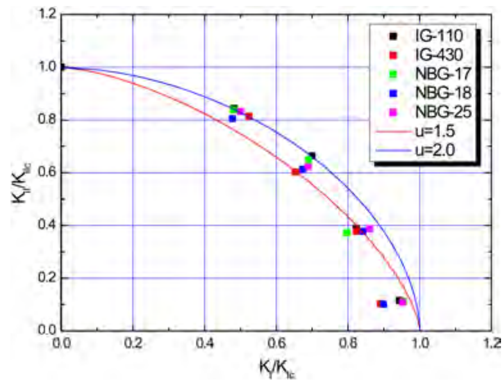

Fig. 4. Fracture criterion in the mixed mode I and mode II for the graphites.

increased with the coke particle size which may be related with an inherent crack size. Pores and internal cracks are inherent to the microstructure and develop during thermal processes (baking and graphitization) due to gas evolution or anisotropic thermal shrinkage during cooling. When a cracked body is subjected to a far-field load, very large strains are generated in a frontal process zone. In the process zone, microcracks are generated from pre-existing pores and sub-critical crack growth occurs between inherent pores. The crack extension takes place when the main crack reaches a critical length which is known to depend on filler particle size in graphite [7].

The values of K_{Ic} in this experiment were somewhat smaller than those which were measured using a single edge notched three-point bending specimen [8]. In disk tests, slow crack growth occurs with difficulty prior to dynamic crack propagation, because tensile stress acts almost uniformly on a diametral line in the disk. Another reason is the difference in specimen size. Fracture toughness values of small volume specimen have a tendency to decrease somewhat especially in a comparatively small volume specimen [9]

The ratios of K_{IIc}/K_{Ic} were in the range of 1.4 ~ 1.5 regardless of the coke type and size. These ratios were a little larger than the reported value of 1.24 for IG-11 [10]. Fig. 4 shows the results of mixed mode fracture toughness for all materials. This shows that the mixed mode fracture criterion is also applicable in spite of the material difference. Representing the relationship of mixed mode fracture criterion as follows [11],

$$\left(\frac{K_I}{K_{Ic}}\right)^u+\left(\frac{K_{II}}{K_{IIc}}\right)^u=1 \quad (2)$$

All the measured data are between the curves with u = 1.5 and u = 2.0.

Summary

The mixed mode I and mode II fracture toughness tests were performed for selected nuclear grade graphites at room temperature and the results are summarized as follows; The fracture loads of the nuclear graphites decreased as the notch inclination angle increased under the mixed mode I and mode II loading due to the sharp crack initiation by the shear deformation at the notch tip. The nuclear grade graphites with the higher bulk density and coarser coke particles showed higher mode I and mode II fracture toughness, since they has less inherent defects like pores. The values of K_{IIc}/K_{Ic} were in the range of 1.4 ~ 1.5.

Acknowledgment

This study was supported by Nuclear Research & Development Program of the National Research Foundation of Korea (NRF) grant funded by the Korean government (MSIP). (Grant code: NRF-2012M2A8A2025682).

References

1. ASTM D 7219-08, Standard Specification for Isotropic and Near-isotropic Nuclear Graphites. Annual book of ASTM Standards, 2009.
2. ASTM D 7301-08. Standard Specification for Nuclear Graphite Suitable for Components Subjected to Low Neutron Irradiation Dose. Annual book of ASTM Standards, 2009.
3. T. Iyoku, S. Shiozawa, M. Ishihara, T. Arai, and T. Oku: Nucl. Eng. Deg. 132 (1991) 23.
4. T. Shibata, M. Eto, E. Kunimoto, S. Shiozawa, K. Sawa, T. Oku, and T. Maruyama, in ‘JAEA-Research 2009-042’.
5. C. Atkinson, R.E. Smelser, and J. Sanchez, Int. J. Frac. 18 (1982) 279.
6. T. Burchell, T. Yahr and R. Battiste: in Proceeding of 3rd International Topical Meeting on High Temperature Reactor Technology, Johannesburg, South Africa, 2006.
7. E.S. Kim and Y.W. Kim: in Proceeding of Carbon 2010, Clemson, USA.
8. M. Ishihara, H. Awaji, S. Sato and S. Kiyono, Tanso, 117 (1984) 124.
9. S. Sato, H. Awaji, K. Kawamata, A. Kurumada, T. Oku, Nucl. Eng. Deg. 103 (1987) 291.
10. M. Li, M. Tsujimura and M. Sakai, Carbon 37 [10] (1999) 1633.