The Shear Fracture Toughness, K_{IIc} , of Graphite

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ABSTRACT

The critical shear stress intensity factor, K_{IIc} , here-in referred to as the shear fracture toughness, K_{IIc} (MPa $\sqrt{m}$), of two grades of graphite are reported. The range of specimen volumes was selected to elucidate any specimen size effect, but smaller volume specimen tests were largely unsuccessful, shear failure did not occur between the notches as expected. This was probably due to the specimen geometry causing the shear fracture stress to exceed the compressive failure stress. In subsequent testing the specimen geometry was altered to reduce the compressive footprint and the notches (slits) made deeper to reduce the specimen's ligament length. Additionally, we added the collection of Acoustic Emission (AE) during testing to assist with the identification of the shear fracture load. The means of K_{IIc} from large specimens for PCEA and NBG-18 are 2.26 MPa $\sqrt{m}$ with an SD of 0.37 MPa $\sqrt{m}$ and 2.20 MPa $\sqrt{m}$ with an SD of 0.53 MPa $\sqrt{m}$, respectively. The value of K_{IIc} for both graphite grades was similar, although the scatter was large. In this work we found the ratio of $K_{IIc}/K_{Ic} \approx 1.6$.

1. Introduction

There are three modes of crack propagation and failure recognized in materials [1] as illustrated in Figure 1. Here the focus is on the mode II (plane shear) behavior of graphite.

Figure 1 Failure modes of materials (crack opening mode I, plane shear mode II, and anti-plane shear, or mode III) [1]

Fracture Mechanics has been widely applied to brittle materials such as Rock, cement and concrete, and graphite. Work on mixed mode fracture mechanics began in the 1960s. For example Erdogan and Sih [2] studied the mixed mode fracture of plexiglass using a (i) a centrally located straight cracked large plate tested under equal and opposite shear loads, and (ii) a large plexiglass plate with a centrally located and arbitrarily oriented two inch long crack subjected to uniform tension. A more recent publication by Atkins et al [3] reported a method for determining the combined failure mode of brittle material using centrally slotted discs, or cracked Brazilian disk (CBD) test. Atkins et al [3] tested sample of polymethyl methacrylate (PMMA) and reported slot orientation test angles that gave pure mode II behavior at various crack lengths. Bazant and Pfeiffer [4] determined the shear fracture behavior of concrete using a double notched beam tested in 3 pt. compression (with an unequal loading span so as to maximize the shear force) and demonstrated a size effect for their concrete materials. A numerical and experimental study of the shear fracture of a brittle material was investigated for reinforced concrete beams using the same technique as employed here, namely the Double Edge Notched Compression (DENC) specimen by Xu and Reinhardt [5] and Reinhardt et al [6]. These authors reported a larger K_{IIc} than had previously been reported for concretes. Moreover, Zhu and Tang [7] performed numerical simulation of the double edge notched shear (DEN) specimen (very similar geometry to the DENC specimen) and obtained good agreement between their analysis and experimental data from Schlangen [8]. Reinhardt and Xu [9] and Xu and Reinhardt [5] report a practical testing approach to determine K_{IIc} , and the mode II fracture energy G_{IIF} for concrete [10] that does not suffer from two of the key concerns of the centrally slotted disc specimen, namely, (i) that the volume through which the shear crack propagates is small, and (ii) alignment of the central slot at the correct angle to give pure mode II fracture is critical.

Despite these difficulties the centrally slotted Brazilian Disc (BD) or the CBD geometry specimens have been widely used to determine mixed mode fracture toughness and mode II fracture of graphite [11,12,13,14,15]. Recently, Smith et al [16] published on the subject of the consequences of the T-stress in elastic brittle fracture. Smith et al [16] produced data from mode II shear specimens of PMMA that contained a razor blade sharpened notch to assure shear fracture. Analysis of the loading conditions was made with the finite analysis code ABAQUS and the shear velocity factor and T-stresses were determined. The specimen geometry allowed shear under compression or tensile loading, producing either a –ve or +ve T stress, respectively. Smith et al [16] showed excellent agreement between predicted and experimental K values. They further showed that the role of the –ve T-stress (such as in the BD and DENC specimen geometries) caused the measured K_{IIc} to be as large as about 2.2 times the mode I fracture toughness. Smith et al [16] concluded that the experiment and theory demonstrated that combinations of mode II loading with large –ve T-stress can lead to a mode II fracture toughness greater the twice the mode I toughness. Thus for graphite, if a method such as the DENC specimen described by Xu and Reinhardt [5], Reinhardt et al [6] and Reinhardt and Xu [9,10] that has a large –ve T-stress were used, we might expect a K_{IIc} $>2.0 \text{ MPa}\sqrt{\text{m}}$ might ensue for graphite with a K_{Ic} value of $\approx 1.0 \text{ MPa}\sqrt{\text{m}}$.

While the mixed mode fracture of geo materials, cement and concrete have been widely studied and published, less has been published on graphite. Indeed, nothing could be found for graphite with respect to the use of DENC geometry specimen. Some of the earliest graphite mixed mode fracture toughness studies were by Awaji and Sato [11] using the centrally slotted BD (CSBD) geometry of specimens. Yamauchi et al also researched mixed mode I/II fracture toughness of

graphite using both center-notched disk specimens [17] and edge-notched half disc specimens [18] for an impact fracture test method. Other researchers have studied the mode I fracture toughness of graphite and thermal shock mode I fracture toughness of graphite [12,19,20].

Some of the earliest studies of fracture in polycrystalline graphite were those of Knibbs [21] who observed the crack growth path directly in his graphites using an optical microscope and showed the critical role of filler particles in the fracture process. Coarser textured material (larger grain sizes) were noted to display more tortuous crack paths while the finer grained POCO AXF graphite appeared to exhibit a less tortuous crack path. Acoustic Emission (AE) during graphite fracture was investigated by Burchell et al [22] who examined AE from a six industrial graphites of widely different grain sizes while subjecting the specimens to three point loading. The development of AE with applied load was shown to be associated with the micromechanical events that caused non-linear stress-strain behavior in graphites, and post fracture AE was indicative of the crack prorogation mode at fracture.

Allard et al [23] studied fracture in anthracite based carbons (used in the aluminum smelting industry) and noted R-curve behavior during mode I fracture. The effect of neutron irradiation on the mode I fracture toughness of grade PCEA graphite was examined by Burchell and Strizak [24,25], who showed that K_{Ic} can be expected to increase upon neutron irradiation. Shi et al investigated crack propagation in graphite [26] using electronic speckle pattern interferometry with image correlation to monitor crack propagation. Shi et al observed R-curve behavior and showed that solutions based on simple beam theory could overestimate the mode I fracture

toughness. Finite element analysis (FEA) using a Continuum Damage Mechanics failure model showed the both friction and notch shape played an important part in providing resistance to crack growth. FEA models provided a satisfactory measure of mode I fracture toughness in the small beams being tested.

Studies of fracture in nuclear graphite by Burchell [27] led to a microstructurally based fracture criterion [28]. The Burchell fracture mechanics based model [29] was able to successfully predict the tensile strength failure probability curves of graphite of widely different texture ranging from the fine grained POCO AXF grade to the coarse grained electrode graphite, AGX. In an attempt to extend the Burchell model to biaxial loading scenarios Burchell, Yahr and Battiste [30,31] determined the biaxial strength of grades H-451 and IG-110. Subsequently, Burchell has attempted to combine the Shetty mixed mode fracture criterion [32,33] with his fracture model, but the need for a finite value of K_{IIc} for graphite has become apparent for analyzing the data in all four stress quadrants with an empirical formula [14]. The DENC specimen was attractive for K_{IIc} determination because of its simplicity. Although much of the mixed mode fracture data for graphite is derived from the use of the CSBD specimen geometry. The CSBD is essentially a mixed mode fracture specimen, although at certain slot angles the specimen will yield pure mode (I or II) toughness values. Those angles were tabulated by Atkins et al [3] and more recently pure mode II angles were reported by Ayatollahi, Berto and Lazzarin [13]. The exact slot loading angles are a function of specimen geometry. The latter authors, and Ayatollahi and Aliha [14] have also investigated the mixed mode fracture analysis of polycrystalline graphite. Ayatollahi and Aliha [14] applied a modified maximum tangential stress (modified MTS) criterion and showed that analysis must include the role of T-stresses which explain the higher

than predicted K_{IIc} in many slotted BD tests [34]. Since the DENC type specimen also has a –ve T-stress we would expect the measured K_{IIc} to be greater than K_{Ic} . Ayatollahi and Aliha [14] also note that empirical models of mixed mode fracture are substantially geometry dependent.

Li et al [35] reported on “crack-face interlocking/bridging of polycrystalline graphite: The role in mixed mode fracture”. In their study of isotropic fine grained graphite, using the single edge notched beam (SENB), and for mixed modes a pre-cracked SENB referred to as a single-edge pre-cracked beam Li et al [35] et al concluded that bridging/interlocking in the crack wake region reduced the stress intensity factor K_I and K_{II} , leading to a toughening of a cracked body. Li et al [35] further concluded that an essential feature of the microstructural process and mechanism of grain interlocking/bridging in the crack-face sliding or mode II type shear, was the crack face opening produced by the asperities of grain facets on the cracked surfaces. Accordingly, these frictional interactions in the crack-face contact region made mixed mode I/II fracture somewhat complicated [35]. Li et al [35] normalized their data and found their effective stress intensity factor (K_{Ieff}/K_{Ic} vs. K_{IIeff}/K_{Ic}) was in good agreement with experimental data.

Ouagne et al [36] conducted a series of experiments to establish the crack growth resistance in two nuclear graphites (PGA and IM1-24). They used the SENB geometry for determining the linear elastic fracture mechanics parameters K_{Ic} and G_{Ic} and the compact tension specimen (CTS) for their controlled crack growth experiments. The interaction of the crack with the microstructure was observed using dye penetrant and a scanning electron microscope. Ouagne et al [36] applied the method of Sakai et al [37,38] (who also showed non-linear fracture of

graphite) and attributed this phenomena to R-curve behavior caused by microcracking and grain bridging. Significantly, Ouagne et al [36] also noted evidence of shear cracking in the crack wake zone in their CT specimens, again demonstrating the potential for mixed modality in a mode I test. Ouagne et al [36] confirmed the findings of Sakai et al [37,38] who attributed the R-curve behavior of graphite to the non-linear processes, both in the microcracking frontal process zone and in the following wake region, and of grain bridging. Similar behavior was reported earlier for the carbon materials used in Aluminum reduction cells [23]. Sun et al [39] reported an analysis of the distinction between loading mode and fracture mode. Although the DENC geometry was not directly addressed, Sun et al suggest that for a central cracked infinite plate and when the fracture angle is zero the, then the shear stress intensity is expressed as $K_{IIc} = \tau(\pi a)^{1/2}$ and $K_{IIc} < K_{Ic}$. One of Sun et al's [39] conclusions was that the fracture toughness of mode II is larger the mode I fracture toughness, if the experiment is conducted correctly. As reported later $K_{IIc} > K_{Ic}$ for the graphite DENC geometry.

Ayatollahi and Torabi [15] conducted analysis and mixed mode fracture mechanics testing of centrally notched BD specimens. The BD's had a "V" notch at their center. Stressed notched graphite is a scenario that can occur frequently in nuclear graphite core components [40,41]. Ayatollahi and Torabi [15] discuss the fact that some graphites exhibit non-linear stress-strain response in a standard tension or compression test conducted on a plain test specimen. When using a plain specimen with no stress concentration, a considerable volume of the material undergoes non-linear deformation before the final fracture. This may give rise to a non-linear stress-strain curve for the tested material. However, in a specimen containing a stress concentration (like a crack or a notch) the material's non-linearity is often localized around the

crack or notch tip and the load-displacement curve is linear or close to linear for the same graphite grade. In such conditions fracture criterion involving K_I and K_{II} may still apply provided the notched graphite specimen fails in a brittle fashion with a nearly linear load-displacement curve. Ayatollahi and Torabi [15] found good agreement between their experimental data and model predictions.

As previously stated, the Burchell fracture model [29] needed to be extended and combined with a mixed mode criteria [42] such as that due to Shetty [32], Ayatollahi and Aliha [14] or Smith et al [16, 43] to model multiaxial data such as the obtained by Burchell et al [30,31], data that has recently been expanded for grade NBG-18 [44, 45]. To accomplish this, a measure the shear fracture toughness, K_{IIc} , for graphites was required. As reviewed by Xu and Reinhardt [5] and Reinhardt et al [6] there is a bewildering choice of potential test geometries. However, many such as the notched BD are mixed mode specimens rather than pure mode II specimens. Following the path taken by Xu and Reinhardt [5] and Reinhardt et al [6] for concrete and cements the DENC specimen [9, 6] geometry was selected. This geometry has several advantages:

1. It is a pure mode II shear test only not a mixed mode test specimen [6]. Numerical studies by Ozbolt et al [47] confirm the specimen exhibits pure mode II shear behavior.
2. Reinhardt et al [6] measured the K_{IIc} of high strength concrete (with the DENC specimen) and conducted numerical studies on the DENC specimen using a finite element program based upon the microplane material model for concrete [46]. Moreover, the specimen geometry has been extended to normal strength concrete and subjected to displacement

measurements around the notch tip [9]. The numerical studies of mode II geometry have been enhanced by Ozbolt et al [47, 48]. Thus, the DENC geometry specimen does not require any additional analysis or numerical simulation.

3. The specimen geometry is simple (a double notched rectangular prism) which renders it easy to machine compared with other specimen geometries.
4. No special test fixtures are needed for the testing of the specimen.
5. There are no alignment issues with the specimen, such as alignment of the central slot at the appropriate angle for mode II measurement, as described by Ayatollahi and Aliha for the a slotted BD [34].
6. The shear crack propagates abruptly if the geometry is correct for the material being tested. An observation made by Ayatollahi and Torabi [15] for their notched polycrystalline graphite BDs.
7. The shear crack propagates through a large volume of material, which is important for two reasons, (i) it is well known that concrete and some graphite grades are not truly brittle materials and display some pseudo-ductility (e.g., non-linear stress-strain curve) and that classical LEFM cannot be applied. However, LEFM is the asymptote to which Non-Linear Elastic Fracture Mechanics converges for large sized elements [9]. Moreover, LEFM is well suited if qualitative relations are looked for, and (ii) a volume/size effect in graphite is observed for graphite's LEFM parameters [49]. Hence, the large volume offered by the DENC geometry is an advantage over other geometries.

Consequently, a program was initiated to establish a value for the shear fracture toughness, K_{IIc} , of two nuclear grade graphites using the DENC geometry specimen.

Shetty[32], and Singh and Shetty [33], found the K_{IIc} values to be greater than the K_{Ic} values for the ceramics they modelled, including graphite, and that a value of $K_{IIc}/K_{Ic} = 1.25$ best represented the data. Shetty [32], and Shetty and Singh [33], proposed the following relationship between K_{IIc} and K_{Ic} , where C is a constant = 1.25

$$\frac{K_I}{K_{Ic}} + \left(\frac{K_{II}}{C.K_{Ic}} \right)^2 = 1 \quad \text{Equation 1}$$

This relationship has subsequently become known as the “Shetty” equation.

Awaji and Sato [11] studied the fracture behavior on several graphites using the centrally slotted disc method similar to that used by Shetty. The three graphites used were grades 7477 and SM1-24, which are both nuclear grades, and an anisotropic electrode grade, SA, in both the parallel and perpendicular to the extrusion direction. These graphites represented a wide range of textures. They developed the following equation

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

Where $\vartheta=1.6$. Significantly the ratio (K_{IIc}/K_{Ic}) for all the graphites/orientations considered had the same value of ≈ 1.1 .

Smith et al [16, 43] has used the relationship:

$$K_{eff} = \sqrt{K_I^2 + K_{II}^2} \quad \text{Equation 3}$$

An effective K derived from K_I and K_{II} would be most useful in our desire to extend the applicability of the Burchell microstructural model for graphite [29].

In this paper measurements of K_{Ic} for two graphites measured using the DENC specimen are reported. This geometry was previously used by Xu and Reinhardt [5, 6, 9] for concretes.

2. Experimental

The specimens were compression tested on an MTS 4-post servo-hydraulic test machine fitted with a 489kN (100 kip) load cell. The load train was fitted with a spherical bearing plate which floats to allow self-leveling. The specimens were compressed between two steel plates placed between the platens. A 16 bit A/D converter using lab-view software captured the load-time (or displacement) curves. A constant crosshead speed of 7.6 $\mu\text{m}/\text{sec}$ or 0.46 mm/min (0.0003 in/sec) was used. Note the specimen was compression loaded to one side of the two edge notches (Figure 2) only; potential load eccentricities were thus avoided.

Acoustic Emission (AE) was monitored during specimen loading as an improvement in testing using a Vallen AMSY-6 multi-channel system, running two channels with type AE105A high frequency probes [50]. The 8 mm dia. X 8 mm height, ceramic faced, stainless steel mini-probes had a frequency range from 450 to 1150 kHz with a peak response frequency of 800 kHz. The probes frequency response is given in Figure 3.

Figure 2 DENC Specimen loading under compression, arrows indicate locations of the two AE probes

Figure 3 Probe frequency response curve [50]

The two AE probes were placed equi-distant from the notch on the center of the face on the DENC specimen (Figure 2). Sonotrace 40 Ultrasonic Couplant was used between the specimen and the probes.

2.1. Materials

Two graphite grades were selected for K_{Ic} testing, namely PCEA (GraphTech International Ltd) and NBG-18 (SGL Carbon Group). Grade PCEA was selected because we have prior fracture (K_{Ic}) data, including the effect of neutron irradiation on K_{Ic} [25]. Grade NBG-18 was selected because we have multiaxial strength data from the first and fourth stress quadrants [44, 45] which we may wish to use in the future for modelling multiaxial stress failure.

PCEA is extruded, isotropic-petroleum coke graphite, manufactured by GrafTech International (GTI). It has a maximum filler particle size of 0.8 mm. Grade NBG-18 is vibrationally molded, isotropic pitch coke graphite, manufactured by SGL Carbon. It has a maximum filler size of 1.6 mm.

The DENC specimen machining orientation is indicated by the arrow in Figure 4. The PCEA specimens were cut such that the notches were aligned against grain (AG). The NBG-18 DENC samples were prepared with their long axis parallel to the AG direction so that their notches were in the with-grain (WG) orientation. Because the NBG-18 is a vibrationally molded grade the WG directions are perpendicular to the forming axis while the AG direction is parallel to the forming axis. The opposite is true for PCEA since it is an extruded grade.

2.2.Specimen geometry

The DENC specimen geometry is shown Figure 4. Testing was performed by loading one half of the specimen in compression to induce a shear failure between the notches (Figure 2).

If the specimen ligament half-length a (Figure 4) is less than w/π then Tada's [51] elastic solution applies and the shear Fracture Toughness K_{II} , is given by [6]

$$K_{II} = \frac{\sigma}{4} \sqrt{\pi a} \quad \text{Equation 4}$$

Where σ is stress and a is the DENC ligament half length.

If the critical stress σ_c at which instantaneous shear fracture along the ligament $2a$ occurs can be identified, K_{IIc} can be estimated from

$$K_{IIc} = \frac{\sigma_c}{4} \sqrt{\pi a} \quad \text{Equation 5}$$

Where σ_c is the critical shear stress and a is the DENC ligament half length

It is important to recognize that K_I vanishes and only K_{II} remains as a stress component. This theoretical solution is valid for an infinite plate, the question is whether it is also applicable to a specimen of finite size. It can be shown [5, 6, 9] that for a finite sized specimen, where a is greater than w/π the relationship

$$K_{IIc} = \frac{\sigma_c}{4} \sqrt{w} \quad \text{Equation 6}$$

Where σ_c is the critical shear stress and w is the DENC specimen's half width (Figure 4) may be used.

For concretes, typical plots of compressive stress vs. compressive displacement are approximately linear up to the point of the stress discontinuity that is identified as the critical point of mode II fracture. Graphites are more pseudo-plastic (Figure 7) (non-linearity in the stress-strain curve). Reasons for the actual non-linearity are discussed later. Medium and coarse grained graphite are not truly brittle materials, but displays pseudo-plasticity. However, prior to the critical stress a portion of the stress-displacement line is linear. Therefore, the two equations (5 and 6) which are developed from LEFM can be applied to the indicated critical load to give an approximation of K_{IIc} .

Figure 4 DENC specimen drawing

Rearranging equation 5 gives:

$$\sigma_c = \frac{4K_{IIc}}{\sqrt{\pi a}} \quad \text{Equation 7}$$

This means that σ_c is smallest at the beginning of the test and increases when a crack is initiated at the notch tip and is propagating. Therefore, the stress-displacement curve (Figure 5 and Figure 6) should show a discontinuity when the shear crack is generated [9]. Importantly, results gained in a numerical study of the DENC geometry of concrete showed that the shear fracture along the ligament happens prior to compressive failure in the loaded part [5]. Since for all the specimens tested in this work the ratios $h > 2a$, $a < (w/\pi)$ and $w > (\pi a)$ applied, Tada's relationship (Equation 5) was used to calculate K_{IIc} .

3. Results

Early trials concentrated on four specimen sizes, i.e., $2w=20, 50, 100, \& 130$ mm. These initial (scoping) studies met with only partial success. Most of the specimen geometries did not shear as anticipated, but failed in compression. Several specimens showed mixed shear/compression failure modes and others only sheared at the moment of compressive failure. Moreover, it was found impossible to identify the expected load discontinuity that should occur at the moment of shearing. This situation was improved greatly by revising the specimen geometry (thickness, T and notch depth, c) and by adding the collection of the AE signature. Consequently, here the results of three improved geometries and AE are reported. The dimensions of the improved DENC graphite specimens (referred to as type 4, type 2 and type 1 specimens) are given in Table 1 and Table 2.

Table 1 Dimensions of the reworked PCEA DENC specimens.

PCEA DENC Specimen dimensions (mm)								
Specimen ID	Width	Height	Thickness	Notch Depth		Ligament length	notch position	Notch Width mm
	W	H	T	C1	C2	2a	w	b
type 4/1	130.03	100.08	48.19	37.928	37.909	24.146	65.156	0.838
type 4/2	130.02	99.95	49.15	37.932	37.919	24.085	64.287	0.98
type 4/3	130.05	100.05	49.2	37.981	37.984	24.007	64.587	0.822
type 4/4	130.03	100.02	48.3	37.997	37.979	24.017	64.725	0.798
type 4/5	130	100.02	49.1	37.901	37.905	24.19	64.081	1.177
type 4/6	130	100.05	48.12	37.871	37.897	24.229	63.427	1.208
type 4/7	130.05	100.05	48.63	37.989	37.982	24.091	64.526	0.877
type 4/8	130.05	100.05	48.05	37.992	37.982	24.052	64.846	0.803
type 4/9	Block Cut Wrong							
type 4/10								
type2/1	50	25.06	24.98	8.479	8.519	7.995	24.562	1.011
type2/2	50.01	24.99	25.05	8.484	8.533	7.976	24.564	1.017
type2/3	50	25.01	25.03	8.551	8.506	7.955	24.392	1.023
type 1/1	20	15.97	16	5.965	5.95	4.026	9.394	1.035
type 1/2	20.01	16.03	16.01	6.008	6.041	3.974	9.584	1.001
type 1/3	20.01	16.03	16.03	5.959	6.005	4.02	9.54	0.983

Table 2 Dimensions of the reworked NBG-18 DENC specimens.

NBG-18 DENC Specimen dimensions (mm)								
Specimen ID	Width	Height	Thickness	Notch Depth		Ligament length	notch position	Notch Width mm
	W	H	T	C1	C2	2a	w	b
type 4/1	130	100.03	49.19	37.845	37.858	24.1	64.678	0.998
type 4/2	129.97	100.1	48.15	37.973	37.944	24.045	64.412	0.789
type 4/3	129.86	100.2	48.19	37.961	37.935	24.174	64.347	0.788
type 4/4	130	100.15	49.18	37.931	37.965	24.225	64.378	0.797
type 4/5	130	100.12	48.3	37.888	37.927	24.244	63.948	1.048
type 4/6	130.01	100.09	49.39	37.898	37.928	24.27	64.431	0.805
type 4/7	130.05	100.2	49.2	37.919	37.91	24.294	64.964	1.004
type 4/8	130.08	100.11	48.14	37.888	37.926	24.306	64.513	0.712
type 4/9	130	100.05	48.18	37.981	37.912	24.132	64.969	0.887
type 4/10	129.468	100.11	49.15	37.983	37.927	24.192	64.524	0.8
type2/1	50.1	25.02	25.01	8.494	8.549	7.982	24.613	0.954
type2/2	50.03	25.01	25.01	8.55	8.557	7.956	24.572	1.029
type2/3	50.05	25.02	25.02	8.497	8.558	7.982	24.43	0.953
type 1/1	20.01	16	16	6.025	5.989	3.972	9.38	0.992
type 1/2	19.99	16	16.01	5.968	6.019	4.007	9.389	1.001
type 1/3	19.99	15.99	16	5.978	6.023	4.002	9.597	0.997

Figure 5 and Figure 6 are illustrative of typical specimen loading curves with AE. In Figure 5, on the compressive loading portion of the load-time curve a load-time discontinuity can be observed at 37,000 lbs. (161 kN) after ≈ 400 seconds accompanied by a burst of AE of magnitude approximately 27000 counts. This load discontinuity is associated with the instantaneous shear failure through the specimen's solid ligament (with its accompanying burst of AE). The load is seen to recover, rising slightly, followed by compression failure at approximately 38500 lbs after ≈ 430 seconds. The compressive failure appears to occur in two steps in this specimen, as the compressive failure crack propagates through the remaining half of the specimen. Additional bursts of AE are detected associated with the compressive

failure. The shear fracture face of the uncompressed half of PCEA specimen 4-7 is shown in Figure 8 (a).

Figure 5 Load-Time-AE for compressive testing of a DENC specimen (PCEA 4-3).

Similarly, for NBG-18 (Figure 6) a burst of AE is seen in combination with a load discontinuity on the loading portion of the load-time plot at the moment of shear (critical stress) when the

Figure 6 Load-Time-AE for compressive testing of a DENC specimen (NBG-18 Number 4-7)

instantaneous shear fracture is observed, followed by a load recovery, increasing to the point of eventual compressive failure. The instantaneous shear event can be identified at 360 seconds and a load of 37000 lbf accompanied by a burst of AE of magnitude 20,000 counts in Figure 6 for NBG-18 specimen 4-7. There are several AE peaks visible in Figure 6 that occur prior to the final shear and subsequent compressive failure and that are not associated with load drops or discontinuities (at times of 90, 150, 230, 220, and 350 seconds) and are thought to be associated with subcritical compressive cracking in the specimen, or crack bridging, etc. that have been identified [22, 35, 36, 37, 38] as potential causes of the non-linearity observed is the stress-displacement behavior. A typical NBG-18 shear fracture face of the uncompressed half of the specimen is shown in Figure 8(b).

Figure 7 Compressive stress – displacement plot for specimen NBG-18 PCEA 4-3

Taking specimen PCEA 4-3 as an example (Figure 7), its compressive failure strength would be 54 MPa which is in reasonable agreement with previous data [52]. As shown in the plot (Figure 7) of compressive displacement (mm) against compressive stress (MPa), the curve shows marked non-linearity, thought to be due to sub-critical compressive event [22, 35, 36, 37, 38] (note the compressive stress and displacement are plotted in the positive sense).

The experimentally measured K_{Ic} data are reported in Table 3 and Table 4 for grades PCEA and NBG-18, respectively. The K_{Ic} values for each test are calculated using equation 5 (which assumes linear- elastic behavior), this potential source of error is discussed later and the critical stress identified from the load-displacement curves and AE output e.g., Figure 5 and Figure 6.

Table 3 PCEA test data.

PCEA														
Spec no	fracture comments	Compressive failure force	Compressive failure force	Compressive strength	compressive pertabation (shear force), lbs	Compressive pertabation (shear force), N	H=2h	w	T=2t	$\sigma_c=F/(w*t)$	TADA'S EQUATION $K_{IIC}=(\sigma_c/4)*\text{SQT}(\text{PI}*a*E-3)$	2a	a	1/2 Volume, wxTxH
		lbs	N	MPa			mm	mm	mm	N/mm ²	MPa.v/m	mm	mm	m ³
1.1	AE and load pertabation	2328	10130.8	67.4	2100	9138.6	15.97	9.394	16	60.801	0.61	1.035	0.518	2.40E-06
1.2	slope change with AE	2237	9734.8	63.4	2100	9138.6	16.03	9.584	16.01	59.558	0.59	1.001	0.501	2.46E-06
1.3	AE and load pertabation	2652	11540.8	75.5	1825	7941.9	16.03	9.54	16.03	51.933	0.51	0.983	0.492	2.45E-06
2.1	no pretabation/compressive/AE	6500	28286.2	46.1	6400	27851.0	25.06	24.562	24.98	45.393	1.27	7.995	3.998	1.54E-05
2.2	no pretabation/compressive/AE	6601	28725.7	46.7	6400	27851.0	24.99	24.564	25.05	45.262	1.27	7.976	3.988	1.54E-05
2.3	no pretabation/compressive/AE	8041	34992.1	57.3	6800	29591.7	25.01	24.392	25.03	48.469	1.35	7.955	3.978	1.53E-05
4.1	AE and load pertabation	43800	190605.2	60.7	43000	187123.8	100.08	65.156	48.19	59.596	2.90	24.146	12.073	3.14E-04
4.2	AE and load pertabation	35770	155660.9	49.3	35000	152310.1	99.95	64.287	49.15	48.204	2.34	24.085	12.043	3.16E-04
4.3	Excellent trace to include in pub	38501	167545.4	52.7	37000	161013.5	100.05	64.587	49.2	50.670	2.46	24.007	12.004	3.18E-04
4.4	lot of AE	32779	142644.9	45.6	32000	139254.9	100.02	64.725	48.3	44.544	2.16	24.017	12.009	3.13E-04
4.5	load pertabation AE lost in background	30029	130677.7	41.5	28000	121848.0	100.02	64.081	49.1	38.726	1.89	24.190	12.095	3.15E-04
4.6	AE and load pertabation	33844	147279.5	48.3	31000	134903.2	100.05	63.427	48.12	44.200	2.16	24.229	12.115	3.05E-04
4.7	Clear AE peak with load pertabation	32056	139498.6	44.5	25000	108792.9	100.05	64.526	48.63	34.671	1.69	24.091	12.046	3.14E-04
4.8	load petabation AE lost in background	36115	157162.2	50.4	36000	156661.8	100.05	64.846	48.05	50.279	2.44	24.052	12.026	3.12E-04
4.9	Incorrect machining					0								
4.10	Incorrect machining					0								
										MEAN (Type 4 only)	2.26			
										ST. DEV. (Type 4 only)	0.37			

Table 4 NBG-18 test data.

Spec no	fracture comments	Compressive failure force	Compressive failure force	Compressive strength	compressive pertabation (shear force),	Compressive pertabation (shear force),	H+2h	w	T=2t	$\sigma_c = F/(w*t)$	TADA'S EQUATION $K_{iic} = (\sigma_c/4) * \text{SQT}(\pi * a * E - 3)$	2a	a	1/2 Volume, wxTxH
		lbs	N	MPa	lbs	N	mm	mm	mm	N/mm ²	MPa.vmm	mm	mm	m ³
1.1	AE used to identify pertabation	2630	11700.4	78.0	2400	10677.2	16	9.38	16	71.143	1.40	3.972	1.986	2.40E-06
1.2	AE used to identify pertabation	2515	11188.8	74.4	2480	11033.1	16	9.389	16.01	73.399	1.46	4.007	2.004	2.41E-06
1.3	AE used to identify pertabation	2097	9329.2	60.8	2100	9342.6	15.99	9.597	16	60.843	1.21	4.002	2.001	2.46E-06
2.1	no clear pertabation or AE, only compressive	9900	44043.5	71.5	8700	38704.9	25.02	24.613	25.01	62.876	1.76	7.982	3.991	1.54E-05
2.2	no clear pertabation or AE, only compressive	10648	47371.2	77.1	9700	43153.7	25.01	24.572	25.01	70.220	1.96	7.956	3.978	1.54E-05
2.3	no clear pertabation or AE, only compressive	9918	44123.5	72.2	9300	41374.2	25.02	24.43	25.02	67.689	1.89	7.982	3.991	1.53E-05
4.1	No AE. Different compressive type failure, more AE compressoin crack arest beavior	45000	200197.6	62.9	31500	140138.3	100.03	64.678	49.19	44.048	2.14	24.100	12.050	3.18E-04
4.2	load pertabation inferred from AE	56079	249486.2	80.4	27000	120118.5	100.1	64.412	48.15	38.730	1.88	24.045	12.023	3.10E-04
4.3	AE but no clear load pertabation	43679	194320.7	62.7	42500	189075.5	100.2	64.347	48.19	60.975	2.97	24.174	12.087	3.11E-04
4.4	shear inferred from AE	56185	249957.8	78.9	37000	164606.9	100.15	64.378	49.18	51.990	2.54	24.225	12.113	3.17E-04
4.5	AE and load pertabation, load slope change at 21000	37658	167534.2	54.2	36000	160158.1	100.12	63.948	48.3	51.853	2.53	24.244	12.122	3.09E-04
4.6	lots of AE peaks , load pertabation at 22000 lbf plus AE	36700	163272.2	51.3	22000	97874.4	100.09	64.431	49.39	30.756	1.50	24.270	12.135	3.19E-04
4.7	lots of AE peaks , load pertabation at 37000 lbf	38500	171280.1	53.6	37000	164606.9	100.2	64.964	49.2	51.500	2.52	24.294	12.147	3.20E-04
4.8	compressive only/AE	34802	154828.4	49.9	34000	151260.4	100.11	64.513	48.14	48.705	2.38	24.306	12.153	3.11E-04
4.9	AE only at 21000lbf, better AE at 40500lbf	41730	185649.9	59.3	34000	151260.4	100.05	64.969	48.18	48.323	2.35	24.132	12.066	3.13E-04
4.10	AE and load pertabation at 17500 lbf	34409	153080.0	48.3	17500	77840	100.11	64.524	49.15	24.545	1.20	24.192	12.096	3.17E-04
										MEAN (Type 4 only)	2.20			
										ST. DEV. (Type 4 only)	0.53

Figure 8 (A) PCEA large sized type 4, specimen 4-7 and (B) NBG-18 large sized type 4, specimen 4-8, showing the clean shear fracture faces that occurs through the specimens solid ligament between the notches in the DENC specimens

4. Discussion

4.1. Predicted Size Effect

Equations (5) and (6) predict a size effect [5]. Equation (5) implies K_{II} increases with $a^{1/2}$, i.e., if the ligament of one structure is twice the one of the other structure, the stress needed to reach K_{IIc} is only $(1/2)^{1/2}$ as large. Figure 9 shows the relationship between ligament length and critical stress. This relationship applies to an infinite plate.

Figure 9 Influence of ligament length on critical stress, (from the literature [5]).

However, if the infinite plate is reduced to an infinite strip with finite width 2ω equation (6) does not show a dependence of the critical stress on the ligament length but only on the strip width.

This means that the critical stress is constant for a constant ω and varying ligament length. This is true until $w = \pi a$. Figure 10 shows the critical stress versus the ligament length for different strip widths.

Figure 10 Critical stress versus ligament length and strip width, (from the literature [5])

The transition point moves to the left with smaller widths of the strip. Figure 10 shows that a size effect of the ligament length only exists when $a = w/\pi$ (the condition for stable crack growth).

The same result was shown by Ozbolt [53] for mode I and mixed failure modes. Thus, when assessing a structure, the starting point in Figure 10 is either on the horizontal line or the curved portion of the line depending upon the combination of a and w .

A further consideration of the double-edge notch plate configuration (as in the DENC specimen) that should receive discussion is that equations (5) and (6) predict a critical stress for certain K_{IIc} . If $K_{II} = K_{IIc}$ the crack (notch) will extend and, therefore, the ligament length will decrease. As a consequence the critical stress increases, so $\sigma < \sigma_c$ and the situation becomes stable again. Figure 10 and Equation (6) predict that a crack will propagate until $a \leq w/\pi$ and the crack arrests if the remote stress is kept constant. The crack propagates again only if the stress increases. This may lead to the situation that final shear will not occur but that other mechanisms govern failure, e.g., compressive fracture [6].

4.2.Experimental data

The numerical data (Table 3 , Table 4) and visual evidence suggests that the data from the small (type1) and intermediate (type 2) specimen are unreliable. All of the type 1 specimens failed to fracture along the shear plane between the notches, and the type 2 specimens were only partially successful: NBG-18 2-1 partially sheared along the shear plane, but specimen NBG-18, 2-2 and 2-3 failed compressively without shear and all type 2 PCEA samples failed in compression without shear. The critical shear stress (defined by AE when shear failure was not obvious) was plotted for PCEA and NBG-18 as a function of ligament half length, a , (mm) in Figure 11 and Figure 12. Referring to the literature [5, 6] (Figure 9) the critical shear stress, σ_c , should fall as the ligament half length, a , increases, reaching a steady state value. The data generated here for the three specimen sizes are plotted in Figure 11 and Figure 12. The critical stress is seen to decrease as ligament length increases for these data. However, the small specimen W (types 1

and 2 specimens) may have been such that the compressive strength was less than the critical shear stress and failure occurred by pure mechanical compression of the half specimen that was loaded.

Figure 11 Variation of the critical shear stress with the ligament length, a, for PCEA

Figure 12 Variation of the critical shear stress with the ligament length, a , for NGB-18.

AE was recorded as the compressive stress on the loaded half of the DENC specimen increased (Figure 5 and Figure 6). The occurrence of a load discontinuity at the critical stress, where instantaneous shear fracture occurred was often accompanied by a burst of AE. Moreover, the AE events were noted prior to the critical stress and also after the maximum compressive stress as the remaining (loaded) half of the DENC specimen failed in compression and the load was seen to fall with increasing displacement. This AE is associated with the fracture events occurring as the specimen is crushed and compressive cracks propagate through the loaded half of the DENC specimen. Post fracture examination of the crushed half of the DENC showed

fragmentation of the graphite clearly indicating the ultimate compressive failure crack must have branched and developed on several fronts.

The test data obtained here for K_{IIc} are reported in Table 3 and Table 4 and are plotted in Figure 13 and Figure 14. For the reasons previously discussed the data for the small (type 1 and 2) specimens were considered suspect and only the data from the larger type 4 specimens was considered to be valid.

Figure 13 Variation of K_{IIc} with the ligament half length, a , for grade PCEA graphite

Figure 14 Variation of K_{Ic} with the ligament half length, a , for grade NBG-18 graphite

The mean values of K_{Ic} for PCEA and NBG-18 are 2.26 MPa√m with a standard deviation of 0.37 MPa√m and mean of 2.20 MPa√m with a standard deviation of 0.53 MPa√m, respectively. The measured value of K_{Ic} for both graphites was similar, although the scatter was large and may be subject to some uncertainty (discussed subsequently).

Mean K_{Ic} values for PCEA and NBG-18 are (from relatively large ASTM D7779 SENB samples) [54]: PCEA K_{Ic} (AG notch) = 1.43 MPa√m (S.D.=0.04 MPa√m, n=20) and PCEA K_{Ic} (WG notch) = 1.29 MPa√m (S.D.=0.03 MPa√m, n=37); K_{Ic} of NBG-18 (WG notch) = 1.36 MPa√m (S.D.=0.10 MPa√m, n=25) and NBG-18 K_{Ic} (AG notch) = 1.41 MPa√m (S.D.=0.05 MPa√m, n=37). Recently, Singh *et al* [49] showed that K_{Ic} is subject to size effects and thus the K_{Ic} values quoted here are subject to some uncertainty.

For the extruded graphite PCEA we expect the material to be tougher when the crack is propagating against grain (AG). Thus our DENC specimens of grade PCEA, cut parallel to the extrusion direction (Figure 4), have a shear plane that is oriented perpendicular to the specimen axis and thus shear cracks are propagating in the tougher AG direction. Thus, if K_{Ic} and K_{IIc} follow the same trends we are measuring the toughest shear direction for PCEA and, thus, might expect it to be tougher (larger K_{IIc}) than NBG-18 which is a vibro-molded grade and, thus, has one AG and two WG directions.

Comparing the mean K_{IIc} values obtained from our larger (type 4) graphite specimens we see that the ratio K_{IIc}/K_{Ic} is approximately 1.6. Xu and Reinhardt [5] found K_{IIc} was twice the value of K_{Ic} reported by other researchers for their concrete materials and higher ratios of K_{IIc}/K_{Ic} have been reported for marble ($K_{IIc}/K_{Ic} \sim 2$) [55].

Table 5 K_{IIc}/K_{Ic} Ratio for PCEA (AG) and NBG-18

Graphite and Notch Orientation	K_{Ic}	K_{IIc}	Mean Of Ratios
	MPaVm (± 1 S.D.)		K_{IIc}/K_{Ic} (± 1 S.D.)
PCEA(AG)	1.43 ± 0.04	2.26 ± 0.37	1.58 ± 0.26
NBG-18 (WG)	1.36 ± 0.1	2.2 ± 0.53	1.62 ± 0.39

Thus it appears that for brittle material $K_{IIc} > K_{Ic}$ should be expected. Other researchers [32, 33, 11, 14] have reported a lower ratio of K_{IIc}/K_{Ic} of 1.1-1.25. Although, our larger values of K_{IIc} and K_{IIc}/K_{Ic} , appear more appropriate because K_{IIc} is derived from only the larger block specimens, where the failure was clearly first by shear fracture (Figure 8 (a) and (b)) and was identifiable by the fracture mode, or by the existence of shear cracks, or by the AE signal. Thus,

we are confident the large DENC specimens are valid for shear fracture toughness determinations whereas the two smaller volumes may not be valid.

Li et al [35] measured the critical K_{II} value of, which they referred to as the apparent mode II toughness, K_{IIc}^{app} , using the straight-through-cracked-notched specimen geometry. There specimens were subjected to combined mode I/II or pure mode II loading under asymmetric four-point bend test, and to pure mode I loading under symmetric four-point bend testing. In the straight-through-notched-specimen geometry they found crack face friction in mode II sliding from the grain interlocking/bridging surface caused by mode I influences. Hence [35], with this geometry an increase in mode I stress intensity and a decrease in the mode two stress intensity can be expected. Despite the problems with the specimen geometry Li et al [35] reported a $K_{IIc}^{app}/K_{Ic}^{app}$ value for a polygranular graphite, IG-11 (with a reported 14 μm grain size), measured with a notched specimen of $K_{IIc}^{app}/K_{Ic}^{app} = 1.11 \pm 0.15$, which is in reasonable agreement with our values for K_{IIc}/K_{Ic} of 1.58 ± 0.26 and 1.62 ± 0.39 from DENC specimen geometries using larger grain size graphites. Perhaps there is less decrement in mode II deformation with the DENC geometry of graphite specimen.

We hope to expand our AE studies to include crack location capability and show that (a) the majority of the AE is associated with the propagation of compressive cracks, and (b) that the AE spike at the load perturbation is from the shear crack propagation.

4.3. Potential error sources in the K_{IIc} data

Because of the important role of graphite in high temperature structural applications its fracture behavior has been extensively studied. Graphite behaves in a pseudo-brittle fashion, with a limited degree of plasticity within a “process zone” [56]. The existence of a “process zone” in-part accounts for the observed non-linearity of some graphite grades [22]. If the graphites are not linear-elastic the validity of the fracture mechanics theories used here must be questioned. Graphite grades are noticeably more non-linear as they increase in textural coarseness (grain size) [22]. Another contributing factor to the pseudo plasticity (non-linearity) of graphite is the R-curve (crack bridging) behavior noted by several researchers [35, 36, 37, 38]. Consequently, for non-linear elastic graphites, it can be expected that the graphite is actually tougher than it may appear. This introduces added conservatism to any cracking situation, which is generally desirable in the nuclear industry.

In defense of the DENC specimen geometry, the shear crack samples a large area of material ($\approx 1000 \text{ mm}^2$) (Table 1 and Table 2). This is an area that will be more representative of the graphite texture for a medium-grain size nuclear graphite than, for example, the notched BD specimen, where the sample ligament area is typically a few tens to a few hundreds of mm^2 (Table 6). Graphite heterogeneity is well known, and is caused by textural variation resulting from filler particle size, distribution, and orientation, forming method and other processing details. Thus sampling a large ligament area is desirable, and will result in a more representative value of K_{IIc} .

A size effects on graphite fracture properties, specifically K_{Ic} , has been previously reported [49, 57, 58]. But as Romanoski and Burchell pointed out the increasing K_{Ic} due to size effects [58], which was attributed to rising crack growth resistance with crack extension, provided a conservative measure of fracture toughness for nuclear graphites when smaller diameter specimens were used.

Moreover, different grades of graphite were studied here than in prior work. Here the two nuclear grades, NBG-18 and PCEA (1.6 mm and 0.8 mm grain size, respectively) were examined and had K_{Ic} values $>1.3 \text{ MPa}\sqrt{\text{m}}$ (measured using ASTM recommended SENB specimens). Prior work by Awaji and Sato [11] reported K_{Ic} values $<1.0 \text{ MPa}\sqrt{\text{m}}$ for various grades the studied (using BD) and Ayatollahi and Torabi [15] used a medium-grained (mean grain size = 0.32 mm) polycrystalline graphite with $K_{Ic} = 1.0 \text{ MPa}\sqrt{\text{m}}$. The K_{Ic} value of graphite is clearly grade specific and test specimen geometry dependent [59]. Perhaps, as Shetty [32] has proposed, the critical value of K_{II} not a unique materials property.

A final reason for our value of K_{IIc} being greater than other values predicted in the older literature [11, 32] was that some prior analyses did not include the effects of T stress. Ayatollahi and Aliha [14] included the negative T-stress in their Modified Maximum Tensile Strength criteria predictions of mixed mode fracture and showed a much improved prediction of Awaji and Sato's data [11]. The presence of a negative T-stress in the [16] causes the measured value of K_{IIc} to be larger than in a specimen geometry where the T-stress is positive and the K_{IIc} value may be underestimated. The maximum effect from the T-stress is expected to take place in

mode II dominated loading conditions [34]. This makes the resultant K_{IIc} from a –ve T-stress specimen unconservative when used for the prediction of critical stress. Belief that the graphite is more shear tolerant than it really is must be considered when designing graphite reactor cores.

Table 6 Comparison of ligament area for the centrally slotted or notched BD and the DENC Type 4 specimen used here

Authors	Reference Number	Specimen type	Disc Radius, R	Central slot length, 2c	c/R	Disc thickness, t	Calculated Ligament Area
			mm	mm		mm	mm ²
Yamauchi et al	[17]	Central Chevron Notched BD	25	25	0.5	5	125
Yamauchi et al	[18]	Central Chevron Notched BD	25	25	0.5	5	125
Sato et al	[12]	Centrally slotted BD	15	12	0.4	3	54
Awaji and Sato	[11]	Centrally slotted BD			0.3 to 0.6	4	
Ayatollahi and Aliha	[14]	Used data from Awaji and Sato [11]					
Shetty	[32]	Used data from Awaji and Sato [11]					
Ayatollahi, Berto and Lazzarin	[13]	Centrally slotted BD	25	25	0.5	8	200
Ayatollahi and Torabi	[15]	V-notched BD	30	30	0.5	8	240
This work	-	DENC Specimen Type 4	ligament Area = $2a \cdot T$ (Figure 4)				1176

5. Conclusions

The DENC geometry appears to be applicable to graphite for the determination of the shear fracture toughness, K_{IIc} (if a sufficiently large specimen is used). Using a specimen of $\sim 0.65 \times 10^{-3} \text{ m}^3$ in volume and with dimensions of at least 48-50 mm thickness, 100 mm height and 130 mm length, with two notches cut into the faces 65 mm from the end to a depth of approximately 38 mm leaving a shear ligament of approximately 24 mm gave the following values for K_{IIc} : Mean K_{IIc} for PCEA (AG) = $2.26 \text{ MPa}\sqrt{\text{m}}$ with a standard deviation of $0.37 \text{ MPa}\sqrt{\text{m}}$ and for NBG-18 a mean K_{IIc} of $2.20 \text{ MPa}\sqrt{\text{m}}$ with a standard deviation of $0.53 \text{ MPa}\sqrt{\text{m}}$. Both graphite grades return similar K_{IIc} values. The values obtained for K_{IIc}/K_{Ic} for PCEA (AG) and NBG-24 (WG) were 1.58 ± 0.26 and 1.62 ± 0.39 , respectively, in reasonable agreement with the previous value of 1.11 ± 0.15 reported by Li et al [35].

In agreement with previous work on concrete [5] and marble [55] a larger graphite K_{IIc} value was observed, in excess of previously published graphite data. Moreover, our work suggests the ratio K_{IIc}/K_{Ic} value (C in the Shetty Equation) is ≈ 1.6 and not 1.25 as previously reported [32]. Various reasons for the discrepancy have been proposed here. However, the possibility that the DENC specimen geometry gives an artificially high value of K_{IIc} must be considered when designing graphite reactor cores.

Based upon the experimental method, techniques and data obtained here we propose a larger study of K_{IIc} to obtain additional data, thus providing a larger, more statistically relevant data set. We also propose to expand the number of graphite grades examined to include isomolded fine grain grades (whose behavior is noticeably more linear-elastic). Moreover, a separate study shall

be undertaken to determine companion K_{Ic} values from large-200 x 20 x 15 mm ASTM D7779 compliant specimens. Finally, we hope to expand our AE studies to include crack location capability.

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References

- [1] Knott, J. F. and Elliot, D., “*Worked Examples in Fracture Mechanics*” The Institute of Metallurgists, Monograph No. 4, p. 4 (1979)
- [2] F. Erdogan and G.C. Sih,, “*On the crack extension in plates under plane loading and transverse shear*”, J, Basic Eng. Trans. ASME Vol 85, pp. 519-525 (1963)
- [3] Atkinson C., Smelser, R. E. and Sanchez, J., “*Combined mode fracture via the cracked Brazilian Disc*”, International Journal of fracture, Vol.18, No.4, pp279-291 (1982)
- [4] Bazant, Z. P., and Pfeiffer, P.A. Matériaux et Constructions, “*Shear Fracture Test of Concrete*” Vol.19, No. 110, pp. 11-1121, (1986)
- [5] Xu, S. and Reinhardt, H. W., “*Shear Fracture on the Basis of Fracture Mechanics*”, Otto-Graf-Journal, Vol. 16, pp.21-77 (2005)
- [6] H.W. Reinhardt, J. Ozbolt, S. Xu and A. Dinku, “*Shear of Structural Concrete Members and Pure Mode II Testing*”, Adv. Cem. Bas. Mat., 5, pp. 75-85 (1997)
- [7] W.C. Zhu and C.A. Tang, “*Numerical simulation on shear fracture processes for concrete using mesoscopic mechanical models*”, Construction and Building Materials, 16, pp. 453-463 (2002)
- [8] Schlangen E, *Experimental and numerical analysis of fracture processes in concrete*”. Ph.D. Thesis, Delft University of Technology, Netherlands (1993)

- [9] H. W. Reinhardt and S. Xu, “*Experimental determination of K_{IIC} of normal strength concrete*”. Materials and Structures/Matériaux et Construcions 31, pp. 296-302 (1998)
- [10] H. W. Reinhardt and S. Xu, “*A practical testing approach to determine mode II fracture energy G_{IIF} for concrete*”, International journal of fracture 105, pp.107-125 (2000)
- [11] H. Awaji and S. Sato, “*Combined mode fracture toughness measurement by the disk test*”, Transactions of the ASME: Journal of Engineering Materials and Technology 100, pp175-182 (1978)
- [12] S. Sato and K. Kawamata, H. Awaji, M. Osawa and M Manaka. “*Thermal shock resistance and fracture toughness during the graphitization process*”, CARBON 19, pp. 111-118 (1981)
- [13] M. R. Ayatollahi, F. Berto and P. Lazzarin. “*Mixed mode brittle fracture of sharp and blunt V-notches in polycrystalline graphite*”, CARBON 49 pp. 2465-2474 (2011)
- [14] M. R. Ayatollahi and M. R. M. Aliha. “*Mixed mode fracture analysis of polycrystalline graphite – A modified MTS criterion*”, CARBON 46 pp 1302-1308 (2008)
- [15] Ayatollahi M.R. and A. R. Torabi, “*Failure assessment of notched polycrystalline graphite under tensile-shear loading*”, Materials Science and Engineering A 528, pp. 5685-5695 (2011)
- [16] D. J. Smith, M. R. Ayatollahi and M. J. Pavier., “*On the consequences of T-stress in elastic brittle fracture*” Proc. R. Soc. A 462’ pp .2415-2437 (2006)
- [17] Yamauchi Y., Nakano M., Kishida K. and Okabe T., “*Measurement of fracture toughness for brittle materials under mixed-mode impact loading using center-notched disc specimens*”, J. Soc. Mat. Sci., Japan, Vol. 49, No.12, pp.1324-1329 (Dec 2000) – In Japanese
- [18] Yamauchi Y., Nakano M., Kishida K. and Okabe T., “*Measurement of mixed mode fracture toughness for brittle materials using edge-notched half- disc specimen*”, J. Soc. Mat. Sci., Japan, Vol. 50, No.3, pp.229-234 (Mar 2001) – In Japanese
- [19] Latella B. A and Liu. T., “*The initiation and propagation of thermal shock cracks in graphite*”, Carbon 44 (2006) 3043-3048
- [20] Kim J. H., Lee Y. S., Kim D. H., Park N. S., Suh J., Kim J. O. and Moon S. I., “*Evaluation of thermal shock strengths for graphite material using laser irradiation method*”, Materials Science and Engineering A 387-389 (2004) pp. 385-389
- [21] Knibbs, R. H., “*Fracture in Polycrystalline Graphite*”, J. Nucl. Mater. 24 (1987) 174-187
- [22] Burchell T. D., Cooke R. G., McEnaney B. and Pickup I. M., “*Acoustic Emission from polygranular graphites*”, Carbon 23 (1985) 739-747
- [23] Allard B., Rouby D., Fantozzi G., Dumas D. and LaCroix p., “*Fracture behavior of carbon materials*”, Carbon 29 (1991) 457-468
- [24] Burchell T. D. and Strizak J. P., “*The effect of neutron irradiation on the fracture toughness of graphite*”. In Proc. of the HTR 2012, Tokyo, Japan, Oct 28-Nov 1, 2012. Paper No. 12-4-004
- [25] Burchell T. D. and Strizak J. P., “*The effect of neutron irradiation on the fracture toughness of graphite*”. Nuclear Engineering and Design 271 (2014) 262-269
- [26] Shi, L., Haiyan L., Zhemnin Z., Fok A. S. L., Marsden B. J., Hodgkin A., Mummery P. M. and Marrow J., “*Analysis of crack propagation in nuclear graphite using three-point bending of sandwich specimens*”, Journal of Nuclear Materials 372 (2008) 141-151
- [27] Burchell T. D., Ph.D. Thesis “*Studies of Fracture in Nuclear Graphite*”, University of Bath (1986)
- [28] Burchell T. D., “*A microstructurally based fracture model for polygranular graphite*”, IAEA-TECDOC-690. Proc of a Specialists Meeting, Tokai-mura, Japan 9-12 Sept 1991.49-58

- [29] Burchell T. D., “*A microstructurally based fracture model for polygranular graphites*”, CARBON 34 (1996) 297-316
- [30] Burchell T., Yahr T. and Battiste R., “*Modelling the multiaxial strength of nuclear graphite*”, Proc HTR2006, 3rd International Topical Meeting on High Temperature Reactor Technology, Oct 1-4, 2006, Johannesburg, South Africa, paper 00000206, pp.1-13
- [31] Burchell T., Yahr T. and Battiste R., “*Modelling the multiaxial strength of H-451 nuclear grade graphite*” Carbon 45 (2007) pp. 2570-2583
- [32] Shetty, D. K., “Mixed-Mode Fracture Criteria for Reliability Analysis and Design with Structural Ceramics” Transactions of the ASME: Journal for Engineering for Gas Turbines and Power, Vol.109, No.3, pp. 282-289 (1987)
- [33] Singh, D. and Shetty, D. K., “*Microstructural Effects on Fracture Toughness of Polycrystalline Ceramics in Combined Mode I and Mode II Loading*”, Transactions of the ASME: Journal for Engineering for Gas Turbines and Power, Vol. 111, No.1, 174-180 (1989)
- [34] Ayatollahi, M. R. and Aliha, M. R. M., “*Wide range data for crack parameters in the disc-type specimens under mixed mode loading*”. Computational Materials Science 38, pp.660-670 (2007)
- [35] Li M., Tsujimura M. and Sakai M., “*Crack-face interlocking/bridging of a polycrystalline graphite: The role in mixed mode fracture*”, Carbon pp.1633-1639 (1999)
- [36] Ouagne P., Neighbour G.P. and McEnaney B., “*Crack growth resistance in nuclear graphites*”, J. Phys. D:App Phys 35 (2002) 927-934
- [37] Sakai M., Yoshimura J-I., Goto Y. and Inagaki M. “*R-curve behavior of a polycrystalline graphite: Microcracking and Grain bridging in the wake region*”, J. Am. Ceram. Soc. 71(No.8) (1988) 609-616
- [38] Sakai M., Urashima K. and Inagaki M., “*Energy principle of elastic-plastic fracture and its application to the fracture mechanics of polycrystalline graphite*”, J. Am. Ceram. Soc. 66 (No.12) (1983) 868-874
- [39] Sun Z., Rao Q. and Chen F., “*Analysis of distinction between loading mode and fracture mode*”. In Proc. 11th Conf. on Fracture, Turin, Italy, March20-25 (2005)
- [40] Burchell T.D (1999) “*Fission reactor application of carbon*” .Chpt. 13, In: Timothy D. Burchell (ed.) Carbon Materials for Advanced Technologies, 429-484, Pergamon
- [41] Burchell T.D. (2012) *Radiation Effects in Graphite*. In: Konings R.J.M., (ed.) Comprehensive Nuclear Materials, volume 4, pp. 299-324 Amsterdam: Elsevier
- [42] ORNL/TM-2011/175 Burchell T., “*Modeling the biaxial failure of zNBG-18 graphite in the first and fourth stress quadrants*” (2012)
- [43] Smith D.J., Ayatollahi M.R. and Pavier M.J. “*The role of T-stress in brittle fracture for linear elastic materials under mixed-mode loading*”, Fatigue Fracture in Engineering Materials and Structures, 24 pp 137-150 (2001).
- [44] ORNL/TM-2009/026, Battiste R., Burchell T. and Strizak J., “*The biaxial strength of NBG-18 graphite*” (2009)
- [45] ORNL/TM-2010/143, Battiste R., Burchell T. and Strizak J., “*Biaxial strength testing of NBG-18 graphite in the fourth stress quadrant*” (2010)
- [46] Ozbolt J. and Bazant Z. P. “*Numerical smeared fracture analysis: nonlocal microcrack interaction approach*”, Int. J. Numerical Methods in Engineering 39, pp. 635-661 (1996)

- [47] Ozbolt J.,Reinhartd H. W. and Xu S., “Numerical studies on the double-edge notch mode II geometry” In Fracture Mechanics of Concrete Structures, Proceedings FRAMOS-3, ed. H. Mishashi and K. Roguko, Aedificatio Publishers, Frieberg, Germany pp. 773-782 (1988)
- [48] Ozbolt J., Li Y. and Kozar I., “*Microplane model for concrete with relaxed kinematic constraint*”, Int. J. of solid and Structures 38 (2001) 2683-2711
- [49] Gyanender Singh, Hiayan Li, Alex Fok, and Susan Mantell, “*Size Effect on the Fracture Properties of Nuclear Graphite*”. In ASTM STP1578 on *Graphite Testing for Nuclear Applications*, Tzelepi and Carroll (Eds), 2014.
- [50] Vallen Systeme , *Acoustic Emission Sensors Specification* Released Germany Dec. 2012, <http://www.vallen.de>
- [51] Tada H., Paris P. and Irwin G., “*The stress analysis of cracks handbook*” Second Edition, Paris Productions Inc., St Louis, Mo (1985)
- [52] ORNL/TM-2009/025. Burchell T., Nunn S, Strizak J. and Williams A.M., “*AGC-1 sister specimen Testing data report*” , May 18, 2009
- [53] Ozbolt J. “*Maßstabeffekt und Duktiliävon Beton und Stahlbeton Konstruktion*”, Habilitationsschrift, 1995, Stuttgart University, Stuttgart, Germany
- [54] Burchell T.D., Unpublished Data
- [55] Aliha, M.R.M., Ashtari, R. and Ayatollahi, M. R., “*Mode I and Mode II “Fracture Toughness Testing for Coarse Grain Marble*”, Applied Mechanics and Materials. Vol. 5-6, pp. 181-188 (2006)
- [56] Romanoski G.R. and Burchell T.D (1999) “*Fracture in Graphite*” .Chpt. 14, In: Timothy D. Burchell (ed.) Carbon Materials for Advanced Technologies, 485-534, Pergamon
- [57] Li H., Li j., Singh G., and Fok. A, “*Fracture Behavior of nuclear graphite NBG-18*” CARBON 60 (2013) 46-56
- [58] Romanoski G. R. and Burchell T. D., “*The effect of specimen geometry and size on the facture toughness of nuclear graphites*”, IAEA-TECDOC-690. Proc of a Specialists Meeting, Tokai-mura, Japan 9-12 Sept 1991, 241-247
- [59] Burchell T. D., “*A comparison of fracture toughness measurement techniques as applied to nuclear graphite*”, Ext Abs. and Prog. CARBONE 90, International Carbon Conference, Paris, France, 16-20 July 1990.