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Significance of Specimen Size for the K_R-Curve Behavior of Quasi-Brittle Materials

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Abstract

The influence of specimen size on the crack resistance (K_R-curve) behavior of a coarse-grained carbon is investigated using compact tension samples of varying size. The results are compared to previous measurements on asbestos cement. In order to analyse and estimate the toughening contribution appearing due to crack-wake related processes, the unloading compliance is measured during the K_R curve test. Subsequent renotching of the extended crack and comparison with the behavior of an ideal linear-elastic body enables the crack-closure forces to be estimated.

Der Einfluß der Probengröße auf das Verhalten der Rißwiderstands (K_R-Kurve) von grobkörnigem Graphit wird mit Hilfe von CT-Proben unterschiedlicher Größe untersucht. Die Ergebnisse werden mit früheren Messungen an Asbestzement verglichen. Die Änderung der Probennachgiebigkeit aufgrund des Rißwachstums wird gemessen, um den festigkeitssteigernden Beitrag, der sich als Folge von Prozessen, die in Verbindung mit den Bruchflanken stehen, zu analysieren und abzuschätzen. Anschließendes Nachkerben des gewachsenen Risses und der Vergleich mit dem Verhalten eines ideal linear-elastischen Körpers ermöglichen es, die rißschließenden Kräfte abzuschätzen.

On a étudié l'influence de la taille de l'échantillon sur la résistance à la fracture (K_R) en utilisant des compactes de poudre de taille différente obtenus à partir de poudre de carbone à gros grains. Les résultats sont comparés à des mesures effectuées précédemment sur du ciment amianté. Dans le but d'analyser et d'estimer la contribution au durcissement du aux procédés apparaissant lors de l'extention de la fissure, on mesure durant le test de résistance à la fracture l'amortissement sans charge. On a estimé les forces tendant à maintenir la fissure fermée en pratiqant une entaille supplémentaire sur la fissure déja présente et en comparant les résultats avec ceux d'un matériau idéal linéairement élastique.

1 Introduction

Higgins & Bailey[1] demonstrated with notch bend specimens of cement paste that the apparent fracture toughness of quasi-brittle materials, apart from its dependence on specimen geometry, can increase with increasing specimen size. This stands in contrast to the statistical nature of the fracture strength. In the meantime this phenomenon has been confirmed by a number of other authors[2,3] with various cement composites.

In many following papers[4–8] attempts to explain this effect and the influence of testing geometry on the apparent fracture toughness were developed. A great number of different models have been introduced, such as the 'fictitious crack model' of Hillerborg *et al.*,[4] the further developed 'equiv-

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alent-elastic crack approach' by Bazant and coworkers,[5–7] the 'two-parameter method' proposed by Shah and coworkers,[8–9] and the 'cohesive crack approach' developed by Cook *et al.*[10] Cotterell & Mai[11] and Steinbrech *et al.*[12] independently proposed similar models that enables one to calculate *R*-curves for standard fracture mechanics testing geometries based on the assumption that the *R*-curve behavior is 'wake' controlled, i.e. caused by bridging stresses. Steinbrech *et al.*[12] could correctly predict how the shape of the *R*-curve of a 16-μm alumina changes with increasing notch depth.

The principal purpose of this contribution is not to give a concise review of the numerous size effect approaches, but to select the most simple relationship between specimen size and fracture parameters.

Based on linear-elastic fracture mechanics (LEFM) ideal brittle materials should follow some simple scaling laws of fracture. For a given test specimen, e.g. for a three-point notch beam (NB) specimen, compact tension (CT) specimen, or double cantilever beam (DCB) geometry specimen, if the planar dimensions of such specimens including the crack size are scaled up by X times with the thickness, b, remaining constant, fracture load, P, and fracture displacement, u, should increase with the square root of X. In other words, by increasing the specimen thickness, d, of NB samples, the CT specimen length, W, or the width, H, of DCB plates by a factor of four, P and u will double for any given relative crack length a/d, a/W, or a/H, respectively. Based on the assumption of a linear crack profile these relationships can be further developed to show that the length of a crack bridging zone, identical to the crack extension length over which the *R*-curve rises, should theoretically also increase with the square root of X. Recent *R*-curve measurements of Foote *et al.*[13] on DCB-fiber–cement composites of varying width H seem to confirm this relationship.

The 'flat' K_R-curve of an ideal brittle material is theoretically a material property, i.e. it does not vary with structure size and geometry. That this is not the case for many quasi-brittle materials is due to the fact that processes in the crack-wake region and ahead of the crack tip are activated and alter the overall macroelastic behavior of the ceramic body. This may lead to a rising K_R-curve. In an early work, Steinbrech *et al.*[14] demonstrated how strongly the apparent *R*-curve is influenced by a variation of notch depth and specimen shape. A frontal process zone, which is large in ceramics, rocks and concretes compared to a plastic-hardening zone characteristic for metals, leads to a deviation from linear behavior. If part of this 'damage zone' does not increase considerably with increasing specimen size, because it is always related to texture, size, and distribution of inhomogeneities and localized residual stresses, it is expected to have a tremendous effect on the elastic behavior of a small body, but only a negligible one for a very large body. A body of infinite size can even be treated as ideal elastic. To what extent the specimen size also affects the K_R-curve behavior of the material all leads back to the question, whether a frontal (microcracked) process zone contributes to toughening or not.

Somewhat different may be the influence of crack-wake related effects, such as compressive wake zones, that enhances friction between rough fracture surfaces, or the presence of elongated grains, fibers and whiskers that bridge the appearing crack. Crack bridging is well known to have a tremendous contribution to the K_R-curve. It is expected that not only the initial K_R value, but also the plateau level and the beginning and extent of the sometimes pronounced artificial rise of the K_R-curve which is often observed when crack bridging is active and the crack approaches the rear end of the specimen, depend on size and shape of the test specimen.

So far, to the authors' knowledge, only one group of researchers, namely Mai *et al.*,[15] has ever experimentally investigated the influence of specimen size on the K_R-curve behavior of quasi-brittle materials using NB specimens of asbestos cement. The use of large specimens is often impractical and, in contrast to concretes, real-size testing has never been practised with ceramics, probably because of the forbidding costs of fabrication. The aim of this work is to experimentally study the *R*-curve size effect by using CT samples of a coarse-grained carbon anode material. The results are compared with the observations of Mai *et al.*,[15] and the role of appearing toughening mechanisms for the size dependence will be discussed.

2 Experimental Procedure

2.1 Material

The tests were performed with a coarse-grained carbon material from an anode supplied by Comalco Aluminium Ltd. The anode had been produced in the normal manner for use in aluminum reduction cells. A heated mixture of coke particles (previously calcined to 1250°C), recycled anode material and 15 wt% pitch is vibroformed into a block and then baked to a temperature of 1100°C. Coke particle size varies from 'dust' (<75 μm) to 4·75 mm. The recycled material, consisting of the portion of anodes unconsumed in

Table 1. Compact tension specimen sizes

Sample number	Thickness (mm)	Width (mm)	Notch length (mm)	(a/W)
1	21·9	28·1	14·0	0·50
2	22·0	46·7	20·7	0·45
3	21·5	85·0	41·5	0·49
4	19·8	144·0	72·0	0·50
5	19·4	240·0	120·0	0·50

the aluminum reduction process, is crushed to a maximum particle size of 19 mm. Granulometry as a whole is optimized for maximum baked density, 1·57 g/cm^3. The material exhibits a four-point bending strength of about 9·3 MPa (obtained with bars of 26 mm width and 20 mm thickness; inner/outer span ratio = 20/90 mm) and an E modulus of about 8·4 ± 1·1 GPa.[16] By increasing the thickness to 80 mm and the outer span region to 320 mm the apparent strength value decreases to 6·8 MPa.[16]

2.2 K_R-Curve tests

The compact tension tests were conducted on samples with relative dimensions as prescribed by the ASTM standard.[17] In all instances the thickness of the plates was held constant at 20·9 ± 1·1 mm. Actual specimen dimensions and initial precrack sizes are listed in Table 1.

The crack propagation was measured directly using a travelling microscope. The crack opening displacement was obtained from a strain gage based clip gage attached to the test pieces. For the compact tension geometry the K_R values were calculated on the basis of the load P, the relative crack length a/W, the thickness b and the appropriate Y function.[17] Prior to the experiment a region about the notch was coated with an alcohol–kaolin suspension. This led to a white strip about the anticipated crack path and enabled microscopic determination of the crack length.

2.3 Renotching experiment

In order to analyze appearing (toughening) mechanisms that lead to a 'softening' (frontal microcracking process zone) or 'stiffening' (wake-related frictional effects) behavior of the cracked body, a recutting experiment was conducted with specimen number 3. The 31·5-mm long crack was stepwise removed in increments of 2 to 4 mm, and the compliance was measured after each renotching step. Considerable care was exercised to minimize the likelihood of crack extension during each renotching step. The compliance was determined by loading the specimen to 65% of the load which would lead to crack extension of a completely renotched crack. The compliance data were evaluated using the approach of Hu & Wittman.[18]

This approach enables one to measure the range of a K_R-curve and, in particular, the extent of crack tip bridging, which can be considered as the major toughening source in ceramics. The analysis uses the known relationship between normalized crack length a/W and specimen compliance C (inverse stiffness = load point displacement/applied load). Hu & Wittman[18] found that the compliance of a specimen with a bridged crack is always lower than the theoretical value, which would be obtained for an ideal linear-elastic body. This stiffening is caused by closure forces and displacement of the axis of rotation of the sample. After completing the R-curve experiment, the fully developed crack is stepwise renotched and the change of the compliance is recorded. In most quasi-brittle materials it was found that initially the compliance of the specimen does not change as long as the bridging zone is still intact. However, beyond a critical renotch distance from the crack tip, which equals the bridging zone length, the compliance begins to rise. If no softening process zone exists in front of the crack tip the compliance becomes identical with the theoretical value in the moment the entire bridging zone is removed. However, if e.g. a microcracking process zone exists in front of the crack tip that leads to a softening of the specimen, the apparent compliance exceeds the theoretical value upon completely removing the crack.

From the direct comparison of measured and theoretical compliance one is able to calculate the range over which the R-curve rises and the crack closure force distribution along the crack. Hu & Wittman[18] introduced a parameter Φ that quantifies the magnitude and hence the toughening contribution of the appearing bridging stresses. The derivation of Φ is given in detail in two previous papers.[19,20]

3 Results and Discussion

3.1 Size dependence of K_R-curve behavior

The change in fracture toughness with crack length for the various specimens is shown in Fig. 1. The smaller samples display an almost linear rising behavior with crack extension, whereas the larger

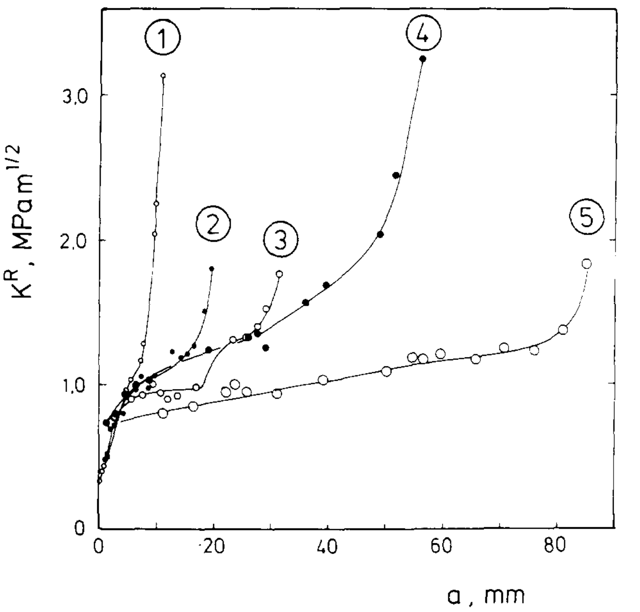

Fig. 1. K_R-curves of carbon anode compact tension specimens of different size.

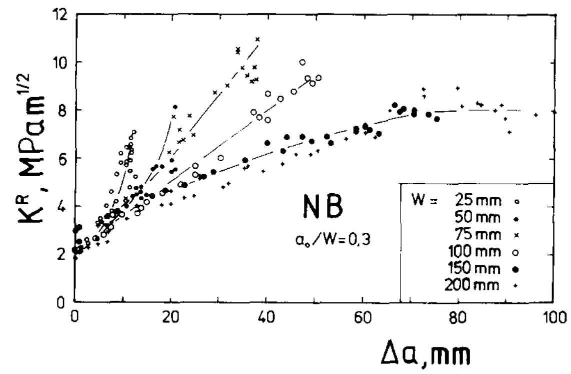

Fig. 3. Reproduction of the K_R-curves of asbestos cement measured by Mai *et al.*[15] with NB specimens of varying width in three-point bending.

samples exhibit a plateau-like region before a sharp rise. The initiation K_R value is about 0·38 MPam$^{1/2}$ for the smallest specimen, 0·45 for specimen number 2, and at 0·7 MPam$^{1/2}$ almost constant for the three larger specimens. This effect of a considerably low initiation K_R value for small specimens has been observed before by Mai *et al.*[15] and was explained by the notch insensitivity of fracture for very small specimens. In such a case the failure is solely determined by the net section stress, which reaches the strength value.

It is interesting that the K_R-curves of small and large specimens appear to be very similar when K_R is plotted versus the normalized crack extension length a/W, as shown in Fig. 2. In this figure the data for the specimens 2, 3 and 5 almost completely overlay one another. Curves 1 and 4 exhibit a deviation from this trend. There is a plateau region at about K_R = 1·0 MPam$^{1/2}$. The sharp rising region of the K_R-curves takes place for about $a/W > 0{\cdot}8$, with the exception of curve 1, for which the K_R curve starts to rise steeply already at $a/W > 0{\cdot}75$. This region is an artifact due to back-surface interaction with the crack tip and an indication of pronounced crack bridging.

From Figs 1 and 2 it is difficult to analyze whether the length of the initial rise of the K_R-curves, which should equal the bridging zone length, increases with the square root of W, as was observed by Foote *et al.*[13] in DCB specimens of fiber–cement composites. Since the plateau region for sample number 5 is lower than that of number 4, it cannot be confirmed that the toughness of quasi-brittle materials characteristically increases with increasing specimen size.

The present results reveal some interesting aspects, in that all curves exhibit a very similar shape and overlay one another when plotted versus the normalized crack length. As mentioned before, Mai *et al.*[15] investigated the influence of specimen size on the K_R-curve behavior by loading asbestos cement NB specimens of varying width (between 25 and 200 mm) in three-point bending. The crack propagation was measured directly using a camera. A replot of their results is shown in Fig. 3.

By size-normalizing the obtained K_R-curves, all data points are found to lie in a band which shows an increasing width with increasing a/W values, as is shown in Fig. 4. For $a/W < 0{\cdot}6$ it seems that the position of the K_R-curves is shifted to higher values with increasing specimen size, which may indicate that the toughness of asbestos cement increases with increasing specimen size. For $a/W > 0{\cdot}6$ the scatter is considerable and the band width finally reaches about 6 MPam$^{1/2}$ at a/W = 0·8. The asbestos cement behaves very similar to the carbon material, in that the smallest specimens (W = 25 and 50 mm) exhibit the lowest toughness (with about 6 MPam$^{1/2}$ for $a/W > 0{\cdot}65$),

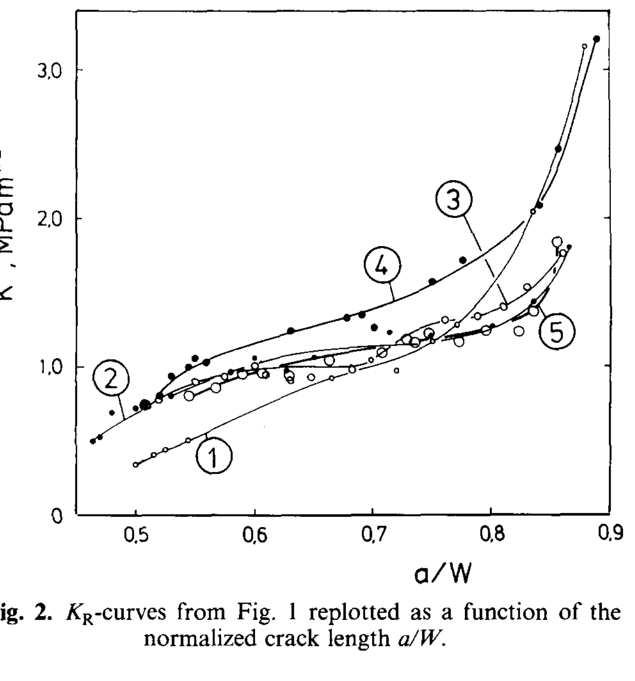

Fig. 2. K_R-curves from Fig. 1 replotted as a function of the normalized crack length a/W.

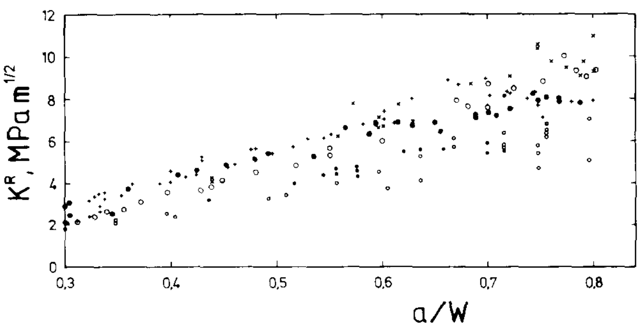

Fig. 4. Plot of the K_R-curves from Fig. 3 as a function of a/W.

followed by the largest samples (W = 150 and 200 mm) with K_R values of about 8 MPam$^{1/2}$. The specimens of medium size (W = 75 and 100 mm) exhibit the highest K_R values (with 9 to 11 MPam$^{1/2}$). Although it seems to be unlikely that this is caused accidentally by local inhomogeneities, which cause the large scatter of the K_R values especially in the smallest specimens, there is no obvious reason for this behavior.

It is remarkable that the shape and position of the K_R-curves of the smallest and the largest specimen are not very different for both materials (Figs 2 and 4), although the specimen width varies by a factor of 8 for the cement and 8.5 for the carbon, and the volume accordingly by a factor of 64 and 73, respectively. This observation may suggest that the size-normalized K_R-curve of the tested carbon and the previously studied asbestos cement may be treated as a 'material property'. If other quasi-brittle materials behave in the same way, their size-normalized K_R-curve would be a reproducible fracture resistance characteristic suitable for the direct comparison with any other quasi-brittle material. However, such a comparison only becomes possible if the same specimen geometry and notch depth are used and if the test body is large compared to existing microstructural inhomogeneities.

However, the above conclusion assumes that the present carbon and cement materials can be considered as typical quasi-brittle materials. This means that their R-curve behavior is caused by the same or similar toughening mechanisms of similar contribution that are characteristic for quasi-brittle materials. The major toughening effect in ceramics, refractories, and concretes is crack bridging, i.e. crack-wake related processes due to the presence of rough fracture surfaces that may be enhanced by a compressive process zone, or alternatively, due to the presence of fibers. Besides crack bridging, stress-induced microcracking is another effect often observed in ceramics and concretes. However, compared to crack bridging, it is generally considered as a minor toughening effect. In the following section an approach is used that enables one to quantitatively determine the 'stiffening' contribution of crack bridging and to separate it from the characteristic 'softening' effect of microcracks appearing in the frontal process zone. This information, which can be gained from the compliance behavior of the specimen, provides valuable hints about the magnitude, distribution and, consequently, the toughening contribution of the bridging stresses.[18-20]

3.2 The contribution of softening and stiffening effects

Observations of the crack tip region revealed significant crack branching and bridging ligament formation along the crack wake and some microcracking about the crack tip. The precise size of the microcracking process zone about the crack tip is difficult to estimate, although there were indications of microcrack development as far as 10 to 15 mm ahead of the crack tip.

The compliance measurements during K_R-curve testing and subsequent renotching of the carbon material in Fig. 1 leads to the results shown in Fig. 5. The upper portion shows the comparison of the measured (C^{exp}) and theoretical (C^{th}) compliance with crack extension during the K_R-curve experiment. There is initially little difference between the two values. However, as a bridging region develops upon crack growth the difference between C^{exp} and C^{th} becomes quite substantial.

The measured and theoretical compliance values obtained during subsequent renotching of the extended crack are shown in the lower portion of Fig. 5 The theoretical curve, which is identical to

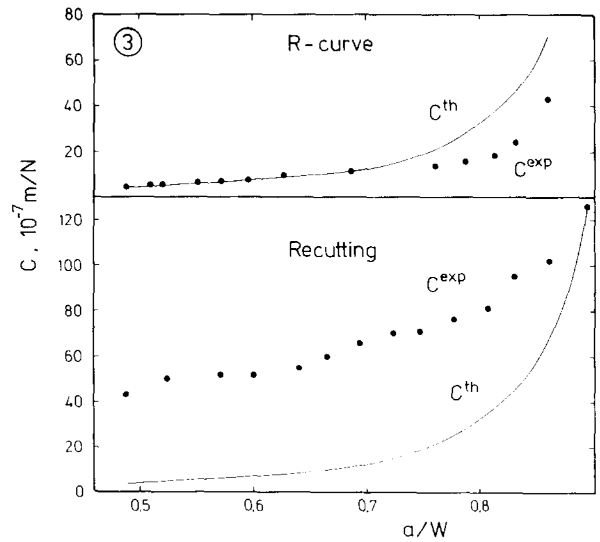

Fig. 5. Development of the compliance upon crack extension (upper part) and recutting (lower part) for sample no. 3. The experimentally determined compliance values C^{exp} are compared with the compliance curves C^{th} which are obtained theoretically for an ideal linear-elastic specimen with exactly the same crack length (upper part) or notch length (lower part) as for the non-ideal test body.

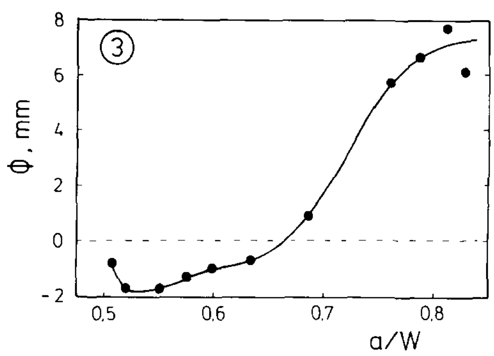

Fig. 6. Φ-curve calculated from the results shown in Fig. 5 using the approach of Hu & Wittman.[18]

the theoretical curve in the upper portion, reflects the development of the compliance of an uncracked ideal linear-elastic (uncracked) specimen being stepwise notched to the same depth as the actual cracked body. The actual measured compliance begins to increase when the recutting has extended the original notch some 18 mm or at about $a/W > 0{\cdot}7$. This indicates that the length of the crack bridging zone is about 13·5 mm or 0·16 a/W. On continued recutting C^{exp} continues to increase, as the bridging zone is removed, but does not finally coincide with the theoretical compliance when the extended crack has been removed completely. The initial precrack was measured to be 31·5 mm long ($a/W = 0{\cdot}86$). However, agreement between both values is not achieved until the notch is extended to 34 mm ($a/W = 0{\cdot}89$). This may indicate that there is a microcrack process zone in front of the crack tip that causes the material to behave 'more softly' i.e. exhibiting higher C values, than was expected. It had to be removed to achieve an equality of C^{exp} and C^{th}.

The approach of Hu & Wittman[18] enables one to calculate a Φ-curve as a function of crack extension length that reflects the incremental change of the compliance contribution of microcrack 'softening' and crack bridging 'stiffening' with crack extension. It was found for many quasi-brittle materials that the Φ-curve rises almost parallel to the K_R-curve of the material, and hence, serves as a measure for the contribution of bridging stresses to the K_R-curve.

The Φ-curve calculated from Fig. 5 for the present carbon sample number 3 is shown in Fig. 6. The initially negative curve indicates that microcracking takes place in front of the crack tip. The influence of microcracking is compensated after 16-mm crack extension by the much stronger stiffening influence of crack bridging.

The present Φ-curve exhibits a shape that is not uncommon for quasi-brittle materials. A similar shape has been observed for coarse-grained alumina and various duplex structures.[20] Maximum Φ values of about 7 mm are slightly higher than values of 4 mm obtained for alumina. In one duplex ceramic that exhibits pronounced quasi-ductility due to the unusual effect of multiple crack branching, Φ values of more than 18 mm were measured. All of these observations indicate that the present carbon can be considered as a characteristic quasi-brittle material.

4 Conclusions

In the present work the influence of specimen size on the K_R-curve behavior of quasi-brittle materials is investigated using a coarse-grained carbon of compact tension geometry. The rising K_R-curve of the carbon is thought to be mainly caused by the appearance of frictional effects in the crack wake region, as for many other quasi-brittle materials. Some microcracking is observed in the frontal process zone, but despite its softening effect on the compliance behavior of the sample, its toughening contribution is considered to be low. It is found that even an 8·5-fold increase of the specimen width (and 73-fold volume increase) does not lead to a considerable change of the K_R-curve when it is plotted versus the relative crack extension length. A similar behavior can be demonstrated with a previously tested asbestos cement. This suggests that the size-normalized K_R-curve of a quasi-brittle material for a given constant notch depth may be treated as a material property, as long as the specimen size is large compared to the grain size of the material or existing inhomogeneities. It hence seems that this approach is suitable for directly comparing the fracture resistance behavior of differently composed ceramics, refractories and concretes.

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