Establishing a protocol for measurements of fractal dimensions in brittle materials

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Many techniques have been used to measure the fractal dimension of brittle fracture surfaces. The purpose of this study was to create a protocol for obtaining the fractal dimension using a simplified optical technique for comparison to reported procedures. Four classes of ceramic materials were used in this study: baria silicate (a glass-ceramic), silicon nitride (a fine grain polycrystalline ceramic), zinc selenide (a large grain polycrystalline ceramic), and silicon (a single crystal ceramic). Contours were produced in perpendicular and parallel planes to the fracture surface using three techniques: slit-island (parallel), profile technique (perpendicular), and crack indentation technique (perpendicular). These contours were then analyzed using a method first introduced by Richardson. The slit-island technique produced statistically greater fractal dimensional increments, ranging from 0.08 to 0.28 for all the materials, than either the profile technique (0.01 to 0.03) or the indentation technique (0.02–0.05). This difference is due in part to the fact that many brittle fracture surfaces are self-affine objects and not self-similar objects. A list of recommendations for a protocol and sources of error for this technique are presented in the appendices. © *2001 Kluwer Academic Publishers*

1. Introduction

Fractal geometry is a non-Euclidean geometry that can quantitatively describe irregular shapes and surfaces. Fractal objects are self-similar (or self-affine) and scale invariant and are characterized by non-integer dimensions. A self-similar object is one in which the length scaling is isotropic and remains invariant under the transformation (x, y, z) to (ax, ay, az) where a is a scalar constant. A self-affine object is one that remains invariant under the transformation (x, y, z) to (ax, ay, az^H) where H is called the roughness exponent. A scale invariant object is one in which the geometric surface will be statistically the same at any magnification scale [1].

The fractal dimension has been used to describe many physical phenomena such as Brownian motion, cloud surfaces, surfaces of porous catalysts, soot particles, colloidal silica aggregates, percolation clusters, lengths of coastlines, and fracture surfaces. The topography produced during brittle fracture has been studied extensively using fractal geometry [1–5]. The fractal dimensional increment, the decimal portion of the fractal dimension, has been correlated to fracture toughness through the following equation [4]:

$$K_{IC} = E a_0^{1/2} D^{*1/2}$$

where K_{IC} is the fracture toughness, E is the elastic modulus, D^* is the fractal dimensional increment, and a_0 is a characteristic length. Many different brittle materials have been graphed using this equation as shown in Fig. 1. “Brittle materials” here refers to materials which fail in a brittle manner in the environment in which they are tested. These materials include glasses, glass-ceramics, and fine grained and coarse grained polycrystalline ceramics.

A variety of techniques to obtain the fracture surface contours have been reported [5–11]. Most boundary contours are obtained using one of two contour planes: parallel to the plane of fracture (slit-island) or perpendicular to the plane of fracture (profile). These boundaries are analyzed using a variety of techniques to determine the fractal dimension. In this paper we only consider methods associated with optical techniques. One of the most popular measuring methods was introduced by Louis Richardson [12] in which the length, L, of a coastline is measured repeatably using different ruler units, S. The slope of a log-log plot of L vs. S is related to the fractal dimension.

Some studies have used vertical profiles to produce contours that were measured by a form of Richardson analysis. Clarke [5, 6] obtained a series of vertical profiles which were analyzed using the Richardson technique. He averaged the values and added 1 to obtain a surface dimension. Denley [7] used line scans obtained by scanning tunneling microscopy (STM). From these vertical scans, he determined a parameter from the

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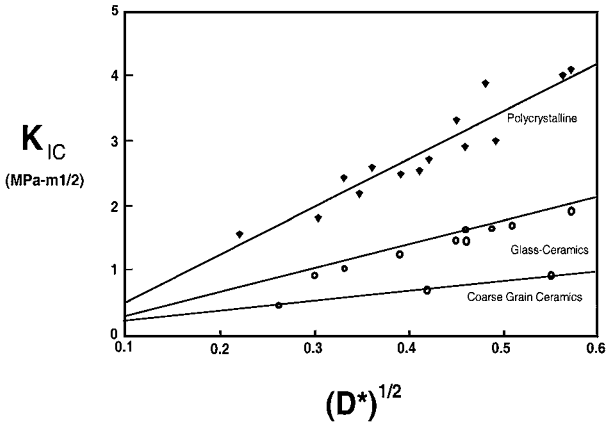


Figure 1 Fracture toughness versus fractal dimensional increment for material classes. [cf. Refs. 3 & 20].

relative surface area obtained as a variation of measuring length. Alexander [8] also applied the Richardson method to elevation profiles. The length of these profiles as a function of ruler lengths resulted in curved lines from which he used the central portion as an estimation of the fractal dimension. Tanaka [9] determined the fractal dimension of soda-lime silica glass and tungsten-carbide cobalt metal using a Richardson analysis of a crack trace produced by a diamond pyramid indentation.

Banerji and Underwood [10] studied the fracture surfaces of high strength steels using vertical sections to determine the fractal dimension. They later reported that the fracture surfaces were not true fractals [11]. To produce their values, they graphed roughness, determined from the ratio of profile length to projected length, versus measuring scale, obtaining a "reverse sigmoidal" curve. Some other studies have since used this technique [13–15]. Pezzotti *et al.* [13, 14], studied the fracture of a SiC/Si_3N_4 isostatically pressed composite. They used the same technique for obtaining the fractal dimension as Underwood and Banerji, obtaining the fractal dimension from a log-log plot of the profile roughness versus the measuring unit. Wasen *et al.* [15] also determined the fractal dimension using this technique for alumina and a series of a SiC/alumina composites. They determined that no correlation was found between fractal dimension and fracture toughness. The fractal dimensional increments for all of the studies mentioned above were relatively low values in comparison to other studies of similar materials [2–4].

Other researchers have used horizontal sections of the fracture surface to obtained profiles for fractal dimension determination, using the slit-island analysis along with the Richardson technique. The first to use this technique were Mandlebrot *et al.* [2]. Their original work used this technique along with Fourier transform analysis of elevation profiles across the surface. Lung [16], in 1985, and Lung and Mu [17], in 1988, also used the slit-island horizontal sections to obtain a fractal dimension which they correlated with fracture toughness values. Fahmy *et al.* [18] used a computer area/perimeter technique, called mosaic amalgamation, to obtain the fractal dimension and compared these values to fracture toughness, finding a positive correlation.

Mecholsky and Plaia [19] used a replication technique to obtain fractal dimensions in the mist and hackle regions of borosilicate glass and calcium aluminosilicate glass. Mecholsky [20] used the slit-island technique to obtain the fractal dimension of a dental glass-ceramic as well as other glass ceramics. Researchers, such as Chen *et al.* [21] and Hsiung and Chou [22] have used slit-island analysis to determine the relationship between the fractal dimension and fracture toughness for materials such as Si_3Ni_4 and high-strength low-alloy steel, respectively. The values obtained by the above researchers resulted in fractal dimensional increments ranging between 0.06–0.33 depending on the material.

Pande *et al.* [23, 24] compared several measurement methods in studying the fracture of titanium alloys. They used slit-island analysis along with Richardson plots on vertical elevation profiles and secondary electron brightness profiles for a line scan across a fracture surface using SEM. They concluded that the methods gave reasonably consistent values. Ray and Mandal [25] used both horizontal and vertical planes to obtain fractal dimension. They compared the fractal dimension from slit-island analysis with impact energy obtaining a positive correlation. Measurements using vertical profiles in Ray and Mandal's work [25] did not produce straight line log-log graphs.

Many different conclusions have been made regarding the relationship between the fractal dimensional increment and the fracture toughness of materials, using either optical or scanning electron microscope techniques for obtaining the fractal boundary or surface. The main purpose of this study was to create a protocol for obtaining the fractal dimension using a simplified optical technique for which the procedures of other studies may be compared. At the same time, some of the discrepancies observed in the literature may be explained. Finally, common procedural errors will be noted as a caution for would-be researchers.

2. Materials and methods

The four ceramic materials studied represent different classes of ceramics: zinc selenide [26] – a large grained ceramic; silicon [27] – a single crystal ceramic; silicon nitride [21] – a small grained polycrystalline ceramic; and baria silicate glass-ceramic [28] – with a large crystal aspect ratio. Each material studied was correlated to lines shown on the K_{IC} versus $D^{*1/2}$ graph in Fig. 1: zinc selenide and silicon on the single and course grain crystal line; silicon nitride on the polycrystalline line; and baria silicate on the glass-ceramic line.

All fracture surfaces were produced from specimens loaded in 4-pt flexure to failure. Two specimens of each material were studied, and each technique was performed on the exact same specimen by using a replication technique detailed later. Two fracture surface boundaries were obtained from each specimen for each technique. Three techniques were used to obtain the boundaries to be measured for fractal dimensional analysis: the indentation technique (IT), the profile technique (PT), and modified slit-island analysis (SIA).

The Richardson technique (Fig. 2) was applied to measure the length of the boundaries between two

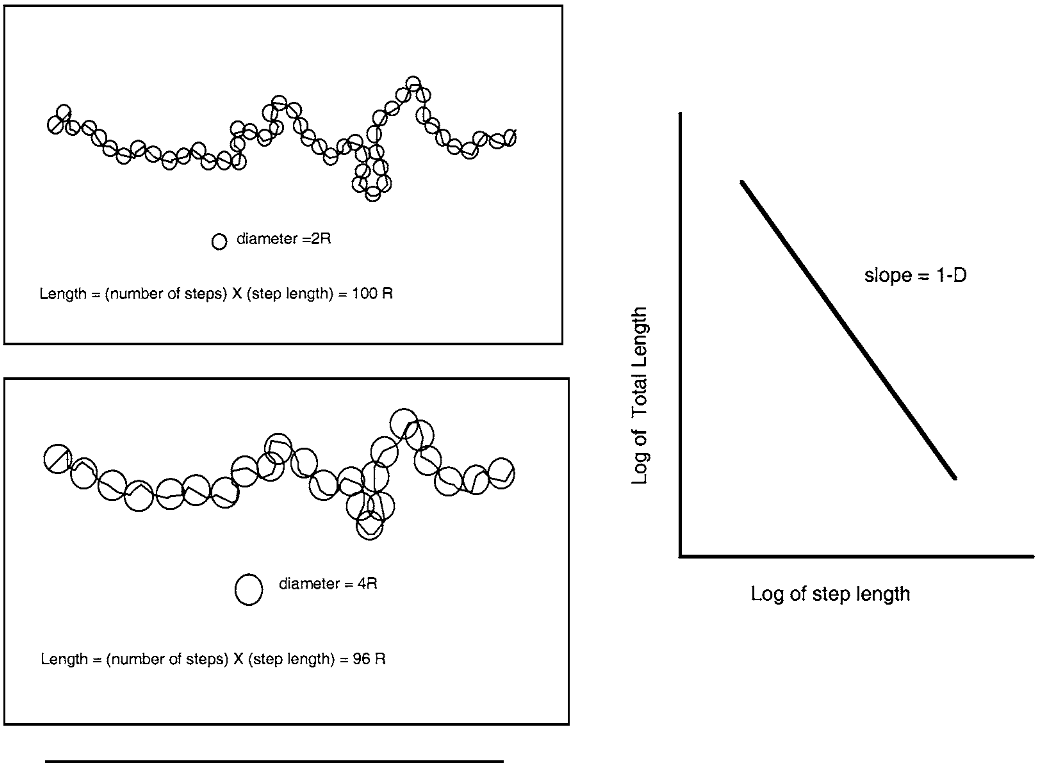


Figure 2 Measuring the total lengths using different step-lengths and plotting these lengths on a log L, - log S graph to obtain (1-D), the fractal dimensional increment.

designated points produced by the three profiling techniques. Calipers set at 4, 6, 8, 16, and 32 mm were step walked along the boundary between two preset points on the collage. The total length was measured using each step length. A smaller step length resulting in a longer total length. Then the total length, L, was graphed versus its step length, S, on a log-log graph, and fitted with a straight line, according to the Richardson equation:

$$L = kS^{1-D}$$

where the slope of this line is equal to 1-D, the fractal dimensional increment, and k is a proportionality constant.

The indentation technique was performed on the tensile side of each of the specimens studied. Each surface to be indented was polished through 1 μm. The specimens were indented using a Vickers diamond indenter on a hardness tester (Microton, Model MO, Bridgeport, CT) at the appropriate load to produce a sizable indentation crack to be measured without causing fracture of the specimen as schematically shown in Fig. 3. The loads ranged from 1 kg (zinc selenide) to 6 kg (silicon nitride). A series of scanning electron micrographs were taken of the crack emanating from the indentation at a magnification of 1000×. These micrographs were then pieced together into a collage of the entire crack length to be analyzed by the Richardson technique.

For both the profile technique (PT) and the modified slit-island analysis (SIA), a replica was created [28] for each specimen fracture. This process was performed to allow a direct comparison between the two techniques while not damaging the fracture surface. The fracture surface of the specimen was thoroughly cleaned with ethanol and rinsed with distilled water. A negative impression was made of the fracture surface using a polyvinylsiloxane impression material (Kerr Manufacturing Co., Romulus, MI). First, a thin layer was slowly applied to minimize the trapped air between the fracture surface and the impression. If bubbles or defects were present, the impression was discarded, and a new one created. Then filled in behind the impression with additional impression material. The impression was allowed to polymerize completely before the impression was carefully separated from the fracture surface. A positive epoxy replica was created from the impression according to the manufacturer's instructions (Leco epoxy kit 811-161, St. Joseph, MI). This impression was coated for 4 minutes using gold-palladium to produce a thick high contrast layer. A subsequent layer of epoxy was then poured over the coated replica creating a sandwich type specimen (Fig. 4). Several replicas

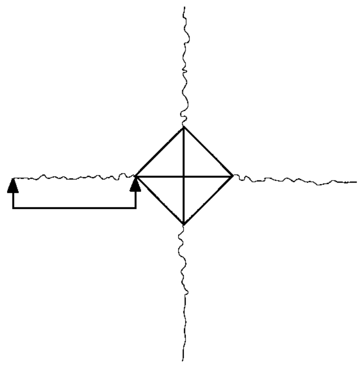


Figure 3 The crack boundary measured from the indentation technique.

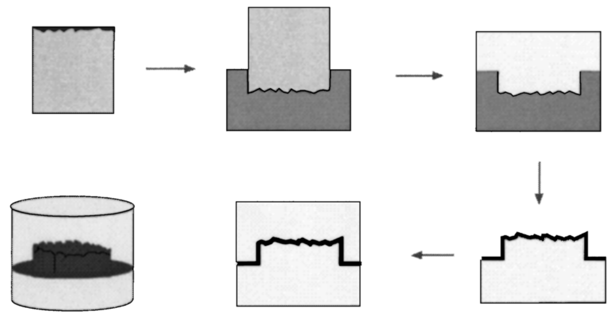


Figure 4 Replication technique of fracture surface.

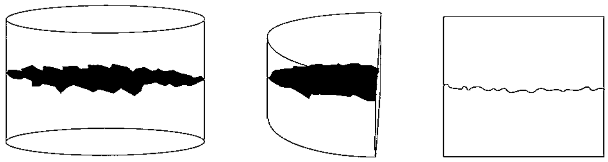


Figure 5 Profile technique cross-section.

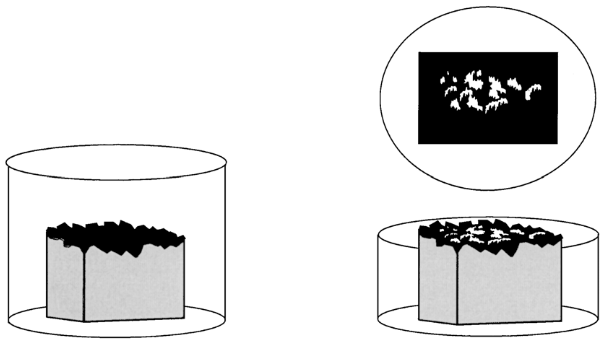


Figure 6 Polishing parallel to fracture surface to produce slit-islands.

were produced from each impression. These specimens can then be used for both techniques.

For the profile technique (PT), the specimen replicas were cut perpendicular to the fracture surface plane producing cross-sections as shown in Fig. 5. Each profile was then polished through 1 μm alumina slurry. A series of 8 to 10 photographs were taken for each profile at a magnification of 400× and then pieced together into a collage of the fracture profile. The Richardson technique was used to analyzed the profile length.

For the modified slit-island analysis (SIA) technique, the specimen replica, as shown in Fig. 6, was polished parallel to the fracture surface until "islands" appeared. The surfaces were then polished through 1 μm alumina slurry to produce a sharper boundary. A series of 8–10 photographs were taken at a magnification of 400× of part of the coastline of one of the islands. These photographs were arranged in a collage where the coastline was measured using the Richardson technique. The SIA technique is modified in that the original presentation [2] measured the area and perimeter of a selected island at different magnifications. In the present study, we measured part of the perimeter (coastline) using several different ruler lengths.

3. Results

The results of all measurements of fractal dimensional increments, D^*, are presented in Table I. The D^* values obtained for both the profile technique and the indentation technique, when analyzed using the Richardson analysis, were less than 0.06 in every case. The modified slit-island technique did not produce any D^* values less than 0.08. The baria silicate glass-ceramic produced the highest value for all three techniques ranging from 0.03 (PT) to 0.28 (SIA), while silicon produced the lowest D^* values for each technique ranging from 0.01 (PT) to 0.08 (SIA). The values for the slit-island technique had the largest scatter of 0.07 maximum between any two measurements (silicon nitride). While the scatter for the indentation technique and the profile technique were lower with a maximum scatter of 0.02.

The boundaries produced by the three techniques display different levels of tortuosity. Both the profile technique (Fig. 7A) and the indentation technique (Fig. 7B) appear much less tortuous when compared to the modified slit-island technique (Fig. 7C) for baria silicate glass-ceramic, which was consistent with the quantitative measurements. This trend is consistent for all materials used in this study.

The D^* values in this study are closer to the predicted lines on the $D^{*1/2}$ versus K_{IC} graph for the modified slit-island analysis values than for the profile technique and the indentation technique for all materials studied as shown in Fig. 8. The baria silicate point falls near the glass-ceramic line; the silicon nitride point is near the polycrystalline line; the single crystal silicon is between the glass-ceramic and course grained ceramic line; and the zinc selenide is on the coarse grained ceramic line as would be expected. In all cases the profile and indentation techniques produced fractal dimensional increments which were much lower than predicted.

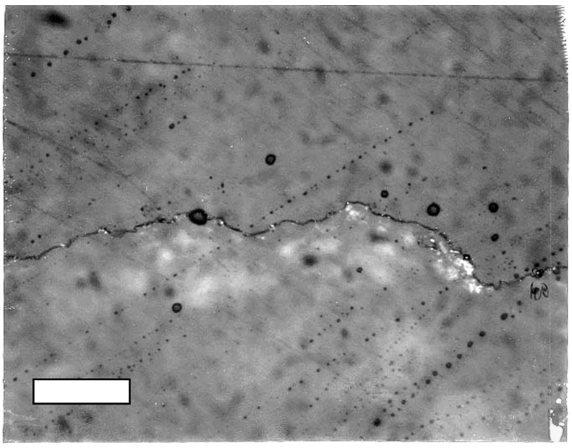
4. Discussion

Many researchers agree that a fracture surface can be a self-affine object, but not a self-similar one [1–3]. This means that as long as a zeroset plane is created through the fracture surface in the (x, y) plane, the values are

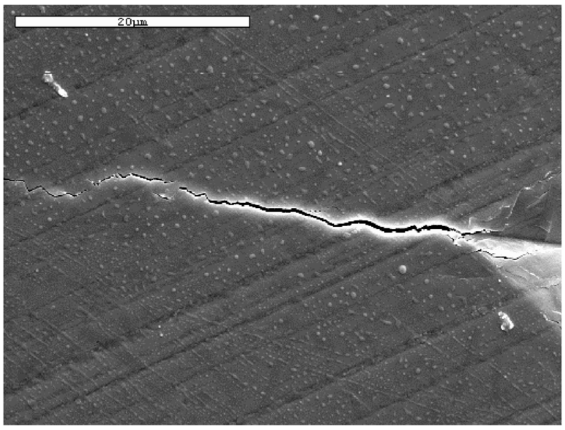
TABLE I Fractal dimensional increments, D^*, measured using three techniques

	Fractal dimensional increment, D^*		
	SIA	PT	IT
Silicon	0.09; 0.07: **(0.08)**	0.01; 0.01: **(0.01)**	0.02; 0.01: **(0.02)**
Baria Silicate	0.31; 0.26: **(0.28)**	0.03; 0.02: **(0.03)**	0.05; 0.05: **(0.05)**
Zinc Selenide	0.13; 0.12: **(0.13)**	0.03; 0.01: **(0.02)**	0.04; 0.02: **(0.03)**
Silicon Nitride	0.28; 0.21: **(0.24)**	0.02; 0.02: **(0.02)**	0.06: 0.04: **(0.05)**

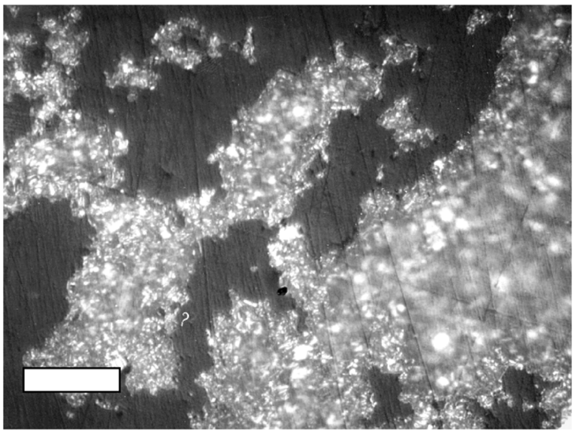
Average values are in parentheses and made bold for each group.



A) Profile coastline of baria silicate.



B) Indentation coastline of baria silicate.



C) Modified slit-island coastline of baria silicate.

Figure 7 Coastline images for baria silicate glass-ceramic using the three techniques A) profile, B) indentation, C) modified slit-island. [Fig. 7A and C are at a magnification 400×. Bar = 25 microns].

valid for the surface using any technique to determine the fractal dimension. Thus, a fracture surface is self-similar in a plane parallel to the surface. Some measurement techniques of the fractal dimension account for the self-affinity of an object or surface while other techniques do not produce valid results. In theory [3], the Richardson technique does not give an accurate fractal dimension unless the plane in which the measurement is being taken is in the zeroset plane. The experimental verification of this statement is the subject of a companion paper [29]. For fracture surfaces, the (x, y) plane would be the plane of fracture in which the slit island technique cuts through to produce the contour boundary. The other two techniques, the indentation and the profile, produce contour boundaries to be measured in the (y, z) plane, which is perpendicular to the (x, y) plane.

Other studies have also examined the fractal dimension applying the Richardson method or variations to measure profile contour boundaries of fracture surface boundaries from the slit island technique. Pande *et al.* [23, 24] used three methods to measure the fractal dimension of titanium alloys: the slit-island technique, the Richardson method on vertical elevation profiles, and the Richardson method applied to a secondary electron brightness profile of a line scan across a fracture surface using SEM. Using these three methods, the authors claimed that the values from all three techniques were "reasonably consistent" in spite of having values of 1.087 to 1.126 for their vertical sections and a value of 1.320 for their slit-island measurements. This range of values is in agreement with the results of this study. They also noted the lack of self-similarity of a fracture surface and concluded that the slit-island technique is fundamentally flawed, a subject which Meisel [30] addressed in his rebuttal on the conclusions of Pande *et al.* [23, 24]. Meisel [30] found that the slit island technique is a valid technique to use for determining the fractal dimension and that there were fundamental errors in the other techniques used by Pande *et al.*

Other researchers [3, 25, 30, 31] have also compared the different techniques and did not agree with the findings above. When Ray and Mandel [25] studied the fractal dimension of steels, correlating it with the impact energy, they found a positive correlation when the fractal dimension was obtained using the slit-island technique. However, a graph of log L versus log S from the vertical profiles did not produce lines that were linear and consistent with a fractal object. Long *et al.* [31] studied the use of vertical sections and criticized the use of parallel vertical sections of an anisotropic fractal surface to obtain the fractal dimension, especially vertical profiles parallel to the direction of fracture. Russ [3] repeatedly states in his section on brittle fracture that using the Richardson method on elevation profiles is inappropriate for self-affine surfaces, which it appears, fracture surfaces are.

The data from the present study seem to support this argument. As can be seen in Fig. 7, the coastlines of the three methods demonstrate that more tortuosity is present using the slit-island technique than for both the other techniques. When compared on the K_{IC} versus $D^{*1/2}$ graph (Fig. 8), the values for each of the four materials are closer to the expected material class lines than the other techniques. The order of the fractal dimension measurements for the materials remained consistent for all the methods studied: baria-silicate glass-ceramic $\geq$ silicon nitride $\geq$ zinc selenide $>$ single crystal silicon.

In conclusion, the fractal dimension measurements for all materials in this study have a greater value for the slit-island technique than values from the indentation technique and the profile technique. These values ranged from 0.08–0.28 for the slit-island, 0.02–0.05 for

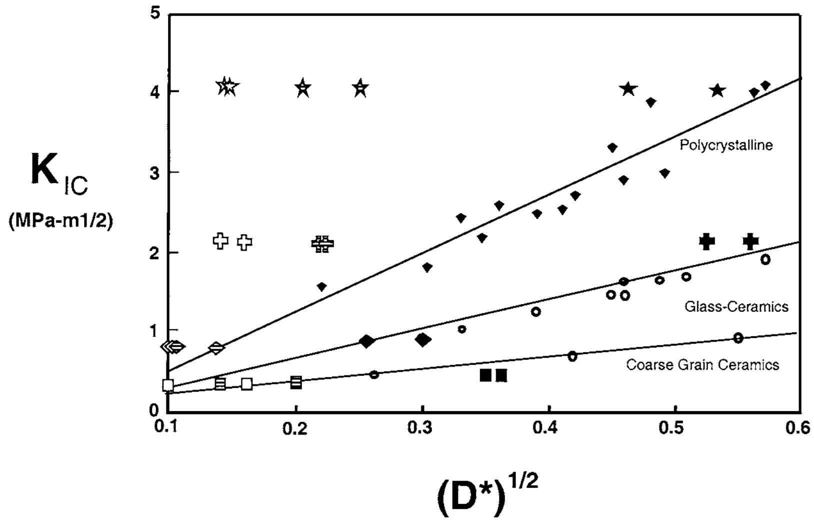


Figure 8 Fracture toughness versus fractal dimensional increment for material classes including materials in this study. (silicon nitride – star; baria silicate – cross, single crystal silicon – diamond; and zinc selenide – square. Open symbols represent the profile technique; symbols with horizontal lines represent the indentation technique; and filled symbols represent the slit island technique.)

the indentation technique, and 0.01–0.03 for the profile technique. This difference in measurements between techniques is thought to be due to the self-affinity of brittle fracture surfaces and thus to the inappropriate method of trying to measure the fractal dimension of a self-affine surface using a vertical profile. The fractal dimension values for the slit-island method were also closer to the predicted material class lines on the K_{IC} versus $D^{*1/2}$ graph.

In performing this study, sources of error were noted which could alter the measured fractal dimension value obtained. These sources of error, along with a recommended procedure, are given in Appendices 1 and 2.

Appendix 1: Recommended procedure

(1) Produce fracture surfaces.

(2) Clean fracture surface carefully with ethanol to remove any dust or debris and rinse with distilled water.

(3) Make an impression of the fracture surface by first applying a thin layer of polyvinylsiloxane over the fracture surface and then fill in the rest of the impression container. Carefully remove the polyvinylsiloxane negative replica from the fracture surface.

(4) Make a positive epoxy replica of the fracture surface impression. Be sure to remove all trapped bubbles that may occur at the interface of the impression material and the epoxy in the area of the fracture surface. Also be sure to have low enough viscosity of the epoxy mixture to allow for good flow, but high enough so that it will harden completely.

(5) After careful separation of the impression and epoxy replica, sputter coat the epoxy replica for a long enough time to produce a substantial uniform layer. Do not coat long enough for the epoxy replica to heat up and alter the fracture surface.

(6) Add more epoxy to form a sandwich type structure of epoxy/coating/epoxy. This will make producing island contours from progressive polishing easier.

(7) SLOWLY grind parallel to the fracture surface until first islands appear. Many specimens have been lost due to grinding through the fracture surface replica too rapidly.

(8) Once the first islands appear, polish surface through 1 μm finish. This approach produces smoother surfaces and clearer contours.

(9) Obtain images of coastline at a magnification appropriate to equipment and microstructure. For our studies, a magnification of 400–500× has been optimum.

(10) Construct collage of fracture coastlines. Piece the images together to form a continuous representation of the coating/epoxy interface.

(11) Measure coastline using systematic step lengths. Use a consistent ruler length to measure the coastline for each set of measurements. Be careful to maintain an exact ruler length throughout the entire step-walk process. Always measure multiple times with each ruler length.

(12) Use the Richardson method of graphing log total length versus log ruler length to determine the fractal dimension from the slope.

Appendix 2: Procedure recommendations

(1) Replicate the fracture surfaces using high resolution impression material.

By replicating the surface, the information from the fracture surface is retained for future research and verification. In our studies, a dental impression material was used which was able to copy the information of the fracture surfaces to be analyzed. Try to eliminate any pores and inclusions during this step.

(2) Use a thin high contrasting coating for your boundary layer.

By using a thin coating, the details of the epoxy replica are more visible and easier to measure. The coating must be thick enough to be uniform and allow for some tolerance of polishing through the silt-island surface.

(3) Polish surface of replica to at least 1 micron finish.

Chen [32] has demonstrated that the final polishing roughness of the replica surface can have an effect on the measured fractal dimension with a finer surface finish giving a more accurate value.

(4) Make the slit-island plane as parallel as possible to the fracture plane.

A slight deviation from the plane of fracture has been shown to give a large deviation in measured fractal dimension. The polishing plane should be as parallel as possible to the fracture plane to ensure accurate measurements. This deviation is thought to be due to the self-affinity of the fracture surface.

(5) Use enough ruler lengths to increase accuracy ($r^2 = 0.98$).

It is known that at least five ruler lengths are needed to ensure an accurate measurement for the Richardson method. If the number of ruler lengths selected is not large enough, then the line formed on the Richardson log-log graph may be invalid and result in an erroneous value. To insure accuracy, use enough points to give a straight line on the Richardson log-log graph with a correlation coefficient of 0.98 or higher.

(6) Insure that the step length remains constant.

When the step walk process is being performed, a minor deviation in perceived step length can cause a large deviation in the measured total length. This can greatly affect the calculated fractal dimension. An imaging program may be better for this part of the process if the resolution is accurate enough. Duplicate or triplicate measurements are required to increase precision.

(7) Multiple replicas of the same specimen should be made to increase precision.

At least two replicas should be made of each fracture surface to provide repeated measured values. This replication procedure will increase accuracy and reduce the sources of variation.

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