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Fractal characterization of brittle fracture in ceramics under mode I stress loading

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ABSTRACT

The self-affine behavior of Brazilian disk test titanium oxide specimens for K_{Ic} measurement was investigated from 3D elevation maps obtained at regular intervals following crack extension. The bifractal approach could be adopted due to the self-affine characteristics of crack propagation under linear elastic regime, determining fractal dimensions associated to micro- (D_T) and macro- (D_S) resolution scales. It was found that fractal dimension data were dependent on crack front positions, and could not be related to fracture toughness.

1. Introduction

Since it was proposed by Mandelbrot, the concept of fractal dimension has been applied to describe surface formation in many phenomena associated to materials structure or failure modes [1,2]. The word “fractal” refers to the multiscale characteristics of surfaces or profiles in nature. For fractal surfaces, the fractal dimension is a statistical quantity that describes how these surfaces fill the space. This introduces the concepts of self-similarity for ideal fractals, when surfaces or boundaries are nondifferentiable and continuous with the same behavior at any size scale, or self-affinity, a general scaling transformation more appropriate to describe real fractal objects, due to the intrinsic anisotropy caused by complexity of the thermodynamics processes involved in the evolution of surface energies changes. Due to their intrinsic complexity, real fractures are not simple fractals, or “monofractals”, with a single fractal dimension, but described by many or multiple fractal values according to the size scale. “Multifractal” surfaces have self-affine characteristics, due to the heterogeneities among different directions.

Mecholsky [3] assumes the monofractal behavior for fracture of brittle materials with controlled crack propagation speed, finding correlations between the fractal dimension and the square root of fracture toughness or theoretical strength. The monofractal approach is clearly restricted by the occurrence of plastic deformation [4] and by the sampling and fractal measurement methods [5,6]. Carpinteri and Pugno [7] proposed that fractal dimension describes toughness and is not dependent on fracture micromechanisms. Sharon and Fineberg [8], Milman et al. [1] and many others have proposed that the establishment of self-similar or self-affine behavior is a function of crack propagation speed. Russ [9], citing Kaye’s concept, described fracture surfaces as mixed fractals, or multifractals, combining the influences of microstructure and applied loading, proposing two scales for fractal analysis: the microscale, or textural, corresponding to the microstructure and micromechanics effects on fine roughness; and the macroscale, or structural, describing the large anisotropic relief behavior due to the evolution of stress fields at crack front. More recently, the multifractal approach has been applied to describe brittle or quasi-brittle fractures, being able to consider the total set of components in fracture processes, from microstructure to environment and loading contexts [10,11], extending the concepts proposed in Russ [9]. In our later experiments for metallic alloys, we concluded that fractal vs. local fracture toughness relations will be dependent on local stress state, at least under elastic-plastic regime, and fractal dimension can be related to local entropy in fracture processes [12]. In this work, fracture surfaces were characterized as bifractals, being evident this behavior in every elevation map measured for each specimen for the self-affine behavior characterization of TiO_2 Brazilian disk test specimens for fracture toughness measurements under mode I loading.

2. Experimental procedure

Central notched Brazilian disc test specimens [13] for determining mode I fracture toughness were prepared by using a commercial

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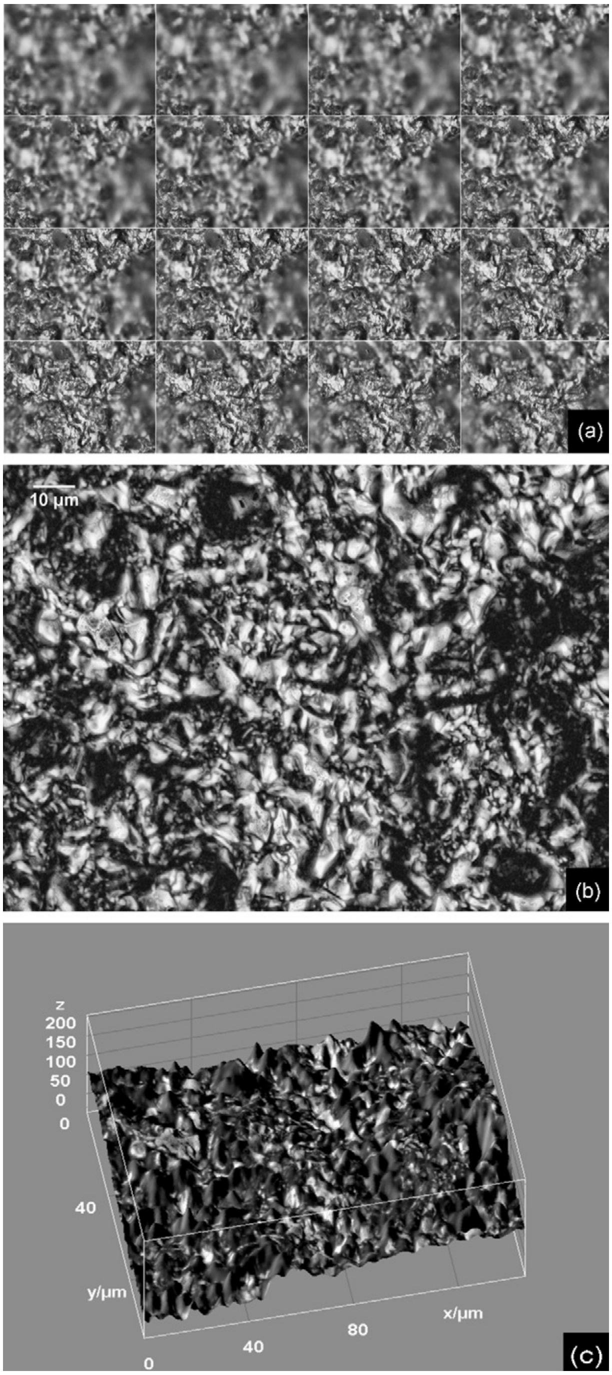

Fig. 1. Example of extended depth-from-focus reconstruction process: (a) images stack at 1.0 mm from machined notch; (b) reconstructed image showing intergranular and transgranular aspects on fracture surface, for the crack growing from bottom to top (scale bar = 10 μm); (c) corresponding 3D elevation map, axes scales in [μm].

titanium oxide powder. The powder was uniaxially pressed, obtaining cylindrical discs with 16.5 mm diameter by 2.0 mm thickness. The plates obtained were calcinated at 1000 °C for 1 h before machining central notches. The resulting pallets were sintered at 1450 °C for 5 h. Diametral compression tests were performed on an electromechanical testing machine at 1 mm/min displacement rate. The pure mode I fracture toughness, K_{Ic}, is computed as:

$$K_{Ic} \cong 1.25 \frac{p_c}{RB} \sqrt{\frac{\alpha}{\pi}} \quad (1)$$

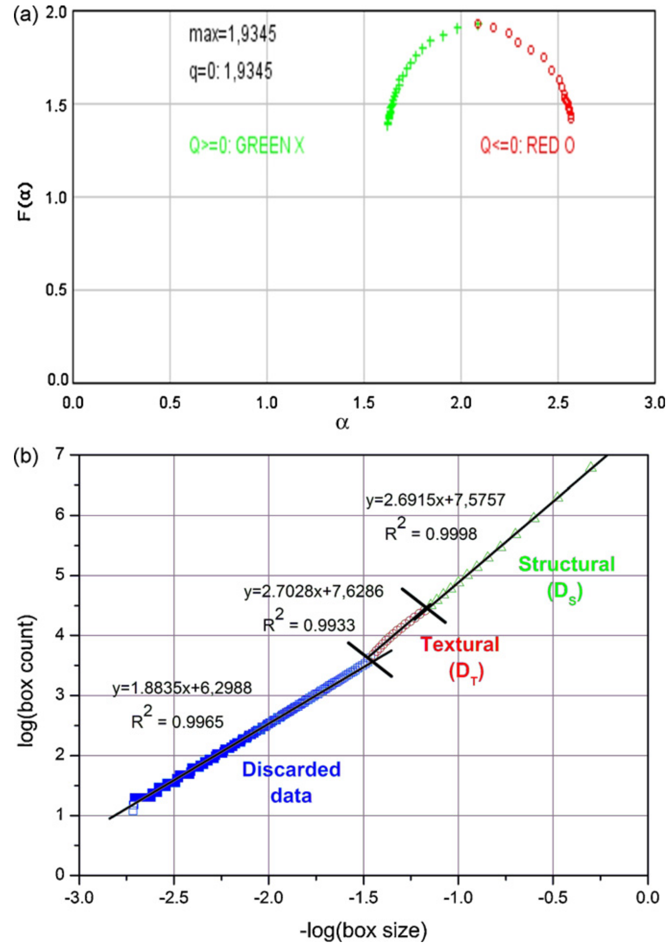

Fig. 2. Multifractal characterization of elevation maps: (a) example of singularities (or multifractal) spectrum revealing heterogeneities on fracture surface formation; (b) the bifractal approach with the determination of textural and structural fractal dimension values.

where the factor 1.25 is related to geometry for pure I mode [14], P_c represents the critical load, R is the disk radius, B is the specimen thickness and a is the half of crack length.

Fractured surfaces were pictured with a digital camera in a light microscope at regular 1.0 mm displacement intervals, from crack initiation to end of fracture, following the centerline relative to specimen thickness. At each position, image stacks were pictured for ordered and successive vertical positions, using 1.0 μm intervals for 3D mapping by an extended depth-of-field reconstruction algorithm (Fig. 1a). Fractal dimension data were computed from elevation maps by using Minkowski–Bouligand method, also known as box-counting dimension. NIH Image J [15], a freeware image processing software developed by Wayne Rasband, was used for overall image processing.

3. Results and discussion

After reconstruction from image stacks, the multifractal character was confirmed for all elevation maps, as can be observed in all singularities spectra (Fig. 2a), denoting the heterogeneity in crack propagation speed, associated to their self-affine scaling laws. This heterogeneity, probably associated to the severe alternation between transgranular and intergranular fracture facets (Fig. 1b) as can be observed on the real fracture surface map at Fig. 1c, can be explained by the large spacing between $Q \geq 0$ and $Q \leq 0$ curve regions in the singularities spectrum presented in Fig. 2a. The box-counting method was applied to evaluate fractal dimensions, resulting in characteristic plots as shown in Fig. 2b. These plots can be approximated to three regions: the low resolution one that represents the microscale range; the intermediary region that is associated to macroscale; and the third one, not representative for fractal description. The boundaries between those three regimes were determined as the following rules:

(a) The discarded data range is defined by the threshold values from box-counting algorithm, as dependent, in practice, on the number of images in each reconstruction stack.
(b) The threshold between textural and structural fractal ranges was determined as the most evident discontinuity in the graph of the first derivative of the log (box count) against – log (box size), after median filtering. This procedure is robust, since the less important discontinuities are naturally discarded, revealing the more regular topographic behavior at both micro- and macro-ranges, validated by the very small dispersion in threshold values for the whole set of fractal curves.

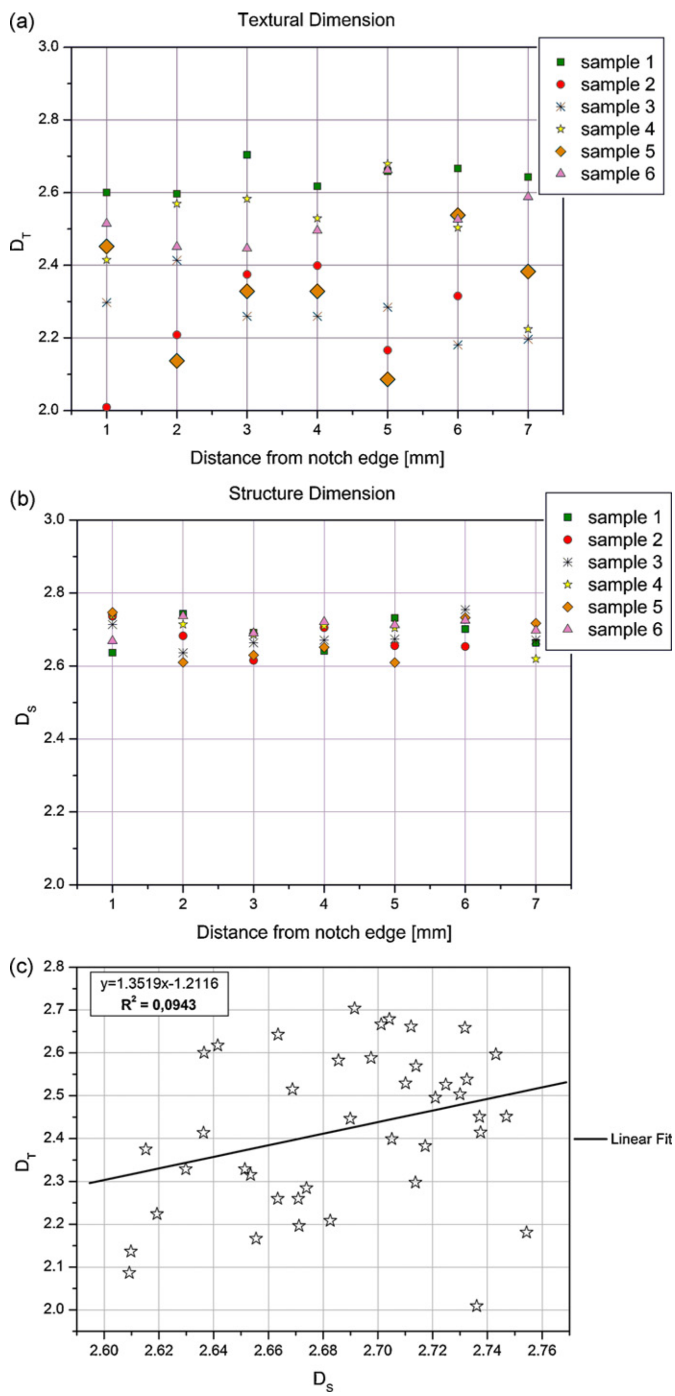

Fig. 3. Behavior of fractal dimension data: (a) textural dimension vs. position on fracture surface; (b) structure dimension vs. position on fracture surface; (c) scattering of textural and structural values.

In both scale ranges, fractal data have presented no evident correlation to investigated positions in each corresponding specimen (Fig. 3a and b), or among the micro- and macroscale fractal data

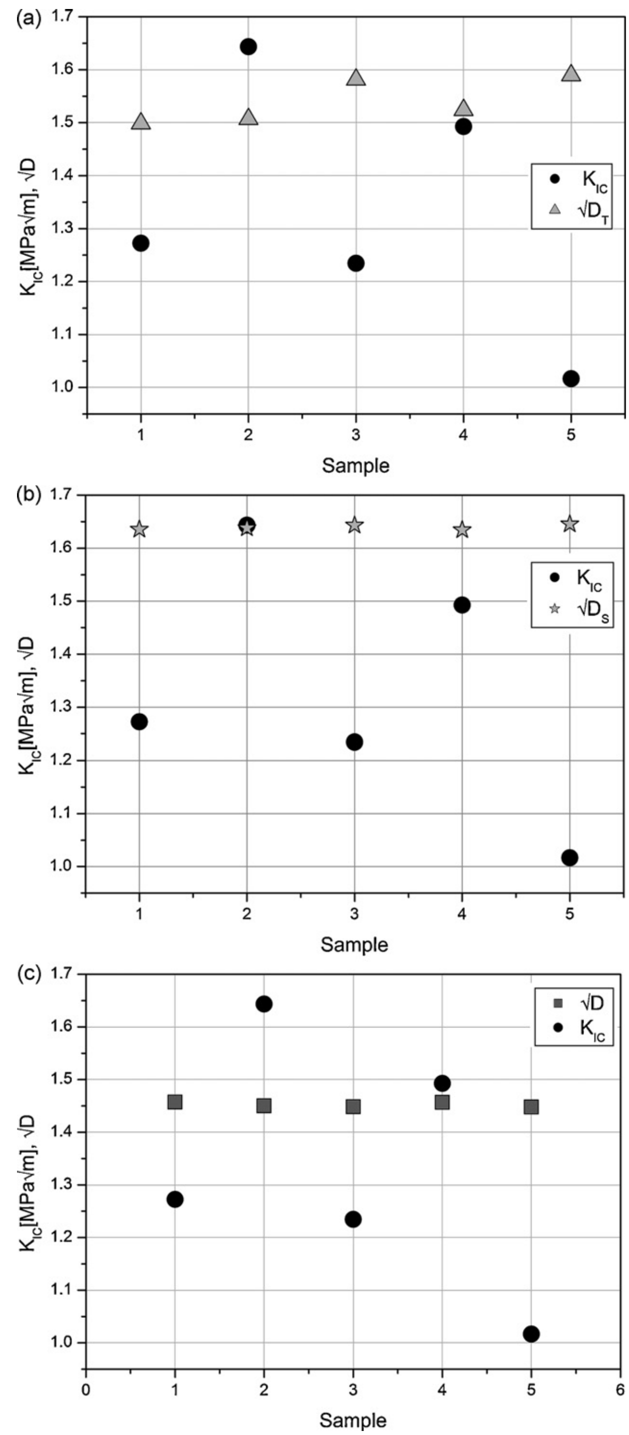

Fig. 4. Fractal and fracture toughness correlations for bifractal and monofractal approaches: (a) K_{Ic} × square root of mean textural dimension; (b) K_{Ic} × square root of mean structural dimension; (c) K_{Ic} × square root of mean fractal dimension in monofractal approach.

and positions on crack advance. It suggests that the monofractal approach is not appropriate to describe the fracture events in these material and testing conditions. Also, fractal dimension data can be considered as local fracture process descriptors, being dependent on crack path, but these may not represent the behavior of entire fracture surface as proposed elsewhere. Also, textural and structural data could not be correlated (Fig. 3c), being independent from each other.

In the same context, the correlation of the square root of mean fractal dimension data to fracture toughness was negligible (Fig. 4a and b), even adopting the monofractal approach as proposed by Mecholsky [3] (Fig. 4c).

In synthesis, for microscale related fractal (or textural fractals, D_T, according to Kaye's concept for bifractals), the following characteristics could be observed:

- Large heterogeneity on crack path and no evident correlation with crack positions, or stress intensity factor data (Fig. 3a).
- Large scattering may be explained due to local changes in activation of fracture micromechanisms associated to microstructural heterogeneities, at microscale, or the local changes in micromechanics during fracture process.
- No correlation between K_{Ic} and mean textural dimension data was found (Fig. 4a).

For macroscale or structural fractals (D_S), it was found that:

Heterogeneity on crack characteristics and scattering is reduced at macroscale relative to textural values, or at microscale, but is still significant and there is no significant correlation with crack positions, too (Fig. 3a).

There is no correlation between structural fractal dimension and crack position, which suggests that there is no correlation between structural dimension and the crack propagation mechanics, at macroscale, such as there is no correlation between K_{Ic} and mean structural dimension data.

These results are also coherent with the self-affine character, described by the confirmed multifractal condition, of fractured relief, since crack propagation in brittle materials tends to assume a bidimensional behavior with the growth in the speed of crack tip, being more heterogeneous to better dissipate large amounts of energy. The inherent heterogeneity in crack speed and fracture surface formation also can explain the large scattering in textural fractal values.

4. Conclusions

In summary, the following comments can be done at this time:

- The bifractal approach is a good approximation due to the shape of box-counting curves and the inherent multifractality in elevation maps. Structural fractal values are somewhat stable with crack propagation, but textural ones present larger scattering, being sensible to local micromechanics changes. The lower scattering for structural fractal dimension values is due to the brittle character of fracture, which provides a somewhat uniform evolution of bidimensional crack advance. The larger scattering for textural fractal data can be attributed to the heterogeneous interaction among stress field and local configuration of microstructure.
- The monofractal approach is not reasonable for titanium oxide brittle fracture under mode I loading. Since the crack velocity could not be controlled, no correlation between fractal values and fracture toughness could be established.

It is certainly necessary to conduct more experiments, testing specimens design of many other material systems in order to improve reliability, such as to test other loading modes (II and mixed I/II, at least). However, since verifying the systematic bias, we confide that such results will provide more compelling evidence in order that approaches based on fractal analysis of fracture surfaces for brittle materials find widespread utilization.

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References

[1] V.Y. Milman, N.A. Stelmashenko, R. Blumenfeld, Prog. Mater. Sci. 38 (1994) 425–447.
[2] L.M.F. Ribeiro, A.L. Horovistiz, G.A. Jesuíno, L.R.O. Hein, N.P. Abbade, S.J. Crnkovic, Mater. Lett. 56 (4) (2002) 512–517.
[3] J.J. Mecholsky Jr., Mater. Lett. 60 (2006) 2485–2488.
[4] K.A. Campos, C. Yoshino, L.R.O. Hein, Mater. Sci. Eng. A 525 (2009) 37–41.
[5] S. Stach, J.J. Cybo, J. Chmiela, Mater. Charact. 46 (2001) 163–167.
[6] S. Stach, S. Roskosz, J. Cybo, J. Cwajna, Mater. Charact. 51 (2003) 87–93.
[7] A. Carpinteri, N. Pugno, Nat. Mater. 4 (2005) 421–423.
[8] E. Sharon, J. Fineberg, Nature 397 (1999) 333–335.
[9] J.C. Russ, Fractal Surfaces, Plenum Press, New York, 1994.
[10] K. Klyk-Spyra, M. Sozańska, Mater. Charact. 56 (2006) 384–388.
[11] K. Slámečka, P. Ponížil, J. Pokluda, Mater. Sci. Eng. A 462 (2007) 359–362.
[12] A.L. Horovistiz, L.R.O. Hein, Mater. Lett. 59 (2005) 790–794.
[13] H. Awaji, T. Kato, S. Honda, T. Nishikawa, J. Ceram. Soc. Jpn. 107 (1999) 918–924.
[14] M.R.M. Aliha, M.R. Ayatollahi, Mater. Sci. Eng. A 527 (2010) 526–530.
[15] W.S. Rasband, ImageJ, U.S. National Institutes of Health, Bethesda, Maryland, USA, http://rsb.info.nih.gov/ij/, 1997–2009.