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Comparison of fracture toughness measuring methods applied on silicon nitride ceramics

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Abstract: Different fracture toughness testing methods on unreinforced and with Si_3N_4 whiskers reinforced silicon nitride ceramics were compared. The results revealed that in the reinforced ceramics the K_{IC} rises with the increasing of the crack size, which indicates a rising R-curve behaviour in this system. Using IF testing method equations given by Niihara and by Shetty proved to have the nearest values to these achieved using SENB in both investigated systems.

1. Introduction

Fracture toughness testing of ceramics is hampered by the difficulties associated with introducing a stable straight crack or a sharp notch into these materials. Different testing methods were used for these measurements by different authors. They achieved different K_{IC} values even in the same material [1-4]. The most widespread fracture toughness measuring methods are:

- bending tests with straight through notch (SENB), Chevron notch (CHN) and indentation strength using Vickers indentation cracks (IS) or controlled surface flaw using Knoop indentation cracks (CSF);
- indentation fracture measurements (IF).

Each of these techniques has its advantages and disadvantages. IF and IS are simple, but less reproducible. SENB gives reliable results if the notch is sharp enough ($<100 \mu\text{m}$). By using IF, there are problems with the surface preparation and with the influence of the preparation mode on the measured K_{IC} values.

The aim of this study is to compare and to judge the suitability in using some of these methods on two types of ceramics, i.e. on unreinforced and with $\beta\text{-Si}_3\text{N}_4$ -whiskers reinforced silicon nitride systems.

2. Experimental Procedures

The unreinforced material was a hot pressed silicon nitride with Al_2O_3 and Y_2O_3 as sintering additives. The reinforced system was a hot pressed ($1750^\circ\text{C}/32 \text{ MPa}$) Si_3N_4 with 20 wt% of $\beta\text{-Si}_3\text{N}_4$ whiskers containing the same additives [5]. The characteristic microstructures of both systems are in Fig.1 a,b. Samples for fracture toughness measurements were cut from billets so as their tensile face were

perpendicular to the hot pressing direction, and ground parallel to the longitudinal axis to 3x4x45 mm. The tensile surface was carefully polished and indented with the Vickers pyramid using loads from 49 to 196 N. All specimens were annealed at 1000 °C/2 h in air before indentation. The fracture toughness of the studied systems was assessed using the following methods:

a) Indentation strength in bending (ISB)

The indents were made carefully to cause cracks perpendicular to the longitudinal axis of the specimens. The indented bars were broken in four-point bending with a lower span of 40 mm and upper span of 20 mm, at the cross head rate of 0.5 mm/min. The K_{IC} values were determined according to the equation published by Marshall et al. [6].

b) Modified indentation strength method (MIS)

Each specimen used in this method was indented three times, using the same load. Then they were broken in the way described in a). The crack lengths were measured before and after the specimen's failure using an optical microscope. Fracture toughness was calculated by MIS from the equation developed by Cook and Lawn [7].

c) Indentation fracture (IF)

There were made from 10 to 15 indents at each load on the very carefully prepared surfaces of samples. The diagonals of indents and the crack lengths were measured microscopically using calibrated eyepiece graticule. The crack type in each sample was determined by serial sectioning and the remaining crack length was measured after each step to generate the crack contour. The IF values were computed using equations published in [8-13].

d) Single edge notched beam (SENB)

The samples were notched by a 0.1 mm thick diamond wheel resulting in a notch root radius about 0.05 mm. The notch depth to specimen thickness ratio was about 0.2. The exact depth was measured microscopically. Tests were performed in 4-point bending with an outer span of 32 mm and an inner span of 16 mm at a crosshead speed 0.5 mm/min. The fracture toughness was computed using the equation published in [14].

Fig.1a,b Microstructure of Si_3N_4 and $\text{Si}_3\text{N}_4 + 20\% \beta\text{-Si}_3\text{N}_4$ whisker systems

3. Results and Discussion

Results of serial sectioning revealed that in both systems the indented crack shapes were closer to the Palmquist type (cracks did not remained connected after polishing away the surface layer of the indent), but they were deeper than in the case of typical Palmquist crack. The fracture toughness values are presented in Tabs. I and II. Fracture toughness values achieved by IS method showed a rising tendency with the rise of c/a ratio in both systems. But this tendency is more remarkable in the case of the reinforced system. Comparing the K_{IC} values measured by SENB with those achieved by IF method computed using equation of Niihara et al. for Palmquist crack type [11] and that of Shetty [13] showed that these two equations gave the most reliable results for both investigated materials. Results computed according to the Laugier equation [14] had a falling tendency with rising of c/a . The MIS method overestimated the fracture toughness values in both materials.

Table I. Fracture Toughness Tests Results on Unreinforced Si_3N_4

Indent.Load [N]	49	98	147	196	294
c/a	2.30	2.65	2.80	2.93	3.16
K_{IC} -IF [MPa.m ^{1/2}]					
Anstis (M) [8]	4.1±0.2	3.9±0.1	4.0±0.2	4.0±0.1	3.9±0.1
Evans-Char.(M) [9]	4.1±0.2	3.9±0.1	4.0±0.1	4.0±0.1	4.0±0.1
Evans (M) [10]	5.2±0.1	5.4±0.1	5.7±0.2	5.8±0.1	5.9±0.2
Niihara (M) [11]	4.8±0.2	5.0±0.2	5.3±0.2	5.5±0.2	5.8±0.2
Niihara (Pq) [11]	5.8±0.1	5.6±0.2	5.7±0.2	5.7±0.1	5.6±0.2
Langford (Pq) [12]	6.3±0.3	6.0±0.2	6.9±0.2	6.9±0.2	6.0±0.2
Shetty (Pq) [13]	5.1±0.1	5.4±0.2	5.7±0.2	5.9±0.2	6.1±0.2
Laugier (Pq) [14]	5.6±0.2	4.7±0.2	4.6±0.1	4.5±0.1	4.2±0.1
K_{IC} -ISB,MIS,SENB [MPa.m ^{1/2}]					
ISB [6]	5.1±0.1	5.3±0.2	5.4±0.2	5.4±0.2	
MIS [7]	5.9±0.3	6.8±0.3	7.8±0.3	7.0±0.3	
SENB [15]			5.8±0.2		

TABLE II. Fracture Toughness Tests Results on Reinforced Si_3N_4

Indent.Load [N]	49	98	147	196	294
c/a	2.05	2.20	2.34	2.59	2.76
K_{IC} -IF [MPa.m ^{1/2}]					
Anstis (M) [8]	5.0±0.2	5.3±0.1	5.3±0.2	4.8±0.1	4.9±0.1
Evans-Char.(M) [9]	5.3±0.2	4.5±0.1	5.4±0.1	4.9±0.1	4.9±0.1
Evans (M) [10]	5.8±0.1	6.5±0.1	6.8±0.2	6.6±0.1	6.9±0.2
Niihara (M) [11]	7.2±0.2	7.6±0.2	7.6±0.2	6.9±0.2	7.0±0.2
Niihara (Pq) [11]	5.5±0.1	6.0±0.2	6.3±0.2	6.2±0.1	6.5±0.2
Langford (Pq) [12]	7.8±0.3	8.2±0.2	8.2±0.2	7.5±0.2	7.5±0.2
Shetty (Pq) [13]	6.2±0.1	6.6±0.2	6.9±0.2	6.6±0.2	6.9±0.2
Laugier (Pq) [14]	7.4±0.2	7.4±0.2	7.1±0.1	6.0±0.1	5.7±0.1
K_{IC} -ISB,MIS,SENB [MPa.m ^{1/2}]					
ISB [6]	6.5±0.2	7.0±0.2	7.3±0.2	7.3±0.3	
MIS [7]	7.8±0.3	8.7±0.4	9.2±0.4	9.1±0.4	
SENB [15]			6.7±0.2		

c = crack length, a = diagonal length, (M) - equation for median crack type, (Pq) - equation for Palmquist crack type

Fractographic analysis of fracture surfaces and fracture lines revealed a very fine grain size in the unreinforced system. The observed fracture micromechanisms correspond with this fine microstructure. The intercrystalline fracture was identified predominantly, with the exception of some cases in which the transgranular fracture of larger Si_3N_4 grains was detected. From the characteristic toughening mechanisms, which include crack deflection, crack bridging, and pull out of whisker shaped grains, only the crack deflection was identified in limited cases. The fine grained globulitic microstructure limits the occurrence of toughening mechanisms and this causes relatively low fracture toughness values.

In the reinforced Si_3N_4 ceramics different types of toughening mechanisms were found (crack deflection, crack bridging by Si_3N_4 whiskers, mechanical interlocking and sliding between both separated fracture surfaces). This system showed a rising R-curve behaviour ($\beta=0.19$), while the low grained unreinforced Si_3N_4 had a nearly flat R-curve ($\beta=0.32$). This is also of importance when comparing the results of K_{IC} measurements of both systems, especially those obtained by IF and ISB methods.

4. Conclusions

The indentation crack profile was more or less of Palmquist type in the whole range of used indentation loads (49-294 N) in both investigated materials. By using indentation fracture measurements the best correlating values with the SENB method gave equations published by Shetty and by Niihara for both systems. In case of reinforced system the determination of the true K_{IC} value is further complicated by its rising R-curve behaviour.

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