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Fracture toughness measurements on SiC/Al_2O_3 composite

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Three test techniques are addressed for the measurements of plane strain critical stress intensity factors, K_{IC}, on monolithic Al_2O_3 and SiC-whisker/Al_2O_3 composite: a four-point test on chevron-notched bend bars; a four-point test on single edge-notched bend bars; and a fractometric test on chevron-notched short bars. The tests were performed on 99.80% Al_2O_3 and 30 vol% SiC whisker-reinforced Al_2O_3. Bend bar test techniques yielded more realistic stress intensity factors, K_{IC}, on the SiC-whisker/Al_2O_3 composite than the short-bar test results. Chevron-notched bend-bar tests yielded relatively higher critical stress intensity factors, on both Al_2O_3 and SiC/Al_2O_3, possibly due to *R*-curve effects, suggesting the use of stress intensity factor as a function of crack length instead of using the minimum value. Ambiguous results, K_{IC}, obtained from short-bar tests on SiC/Al_2O_3 composite, strongly suggests the need to run compliance calibration tests on ceramic composites to determine an appropriate *K*-factor.

1. Introduction

The plane strain critical stress intensity factor, K_{IC}, is a measure of the ability of a material to resist crack growth and is an important parameter to be considered to make a fracture-safe structure. Hard and brittle materials have low fracture toughness, and a small crack or damage in such structures can markedly reduce their load-bearing capacity. Ceramic composites are hard and brittle and, therefore, low-toughness materials; their fracture toughness measurement is exceedingly important for design purposes and quality control checks. Dependence of K_{IC} on test methods calls for determination of test technique(s) that are dependable.

Numerous experimental techniques exist to measure the fracture toughness of ceramic materials. There is, however, no standardized test technique for ceramics and ceramic composites. Some of the commonly employed techniques have been reviewed by Sakai [1]. The techniques can be divided into microflaw and macroflaw methods. Two commonly used microflaw techniques are the controlled indentation-produced microflaw, and the indentation microfracture techniques. The macroflaw techniques are: (a) the chevron-notched (CHV) [2], (b) the straight-through-notched bend bar (SENB) [3], (c) the chevron-notched short-rod(SR)/short-bar(SB) [4], (d) the double torsion(DT) and the double cantilever beam (DCB) [5], (e) the compact tension specimen (CT) [5].

The microflaw techniques can be simple and rapid means of evaluation of fracture toughness. However, the general approach and accuracy of the toughness data must be established in certain microflaw techniques before such applications should be contemplated [6]. The macroflaw techniques often have the advantage of the ease of measurements of crack length, or in some instances do not require crack length measurements [7–9]. Nevertheless, significant variations in the critical stress intensity factors, K_{IC}, have been recorded by both the microflaw and the macroflaw test techniques at various temperature and grain-size regions [10, 11]. Thus the test technique dependence of the fracture toughness makes material characterization difficult. A simple, cost-effective, and at the same time reliable, test technique is now a priority consideration for materials qualification for optimum utilization of engineering materials.

Ceramic composites are now considered a major material opportunity. Being hard and brittle, reliable determination of fracture toughness of ceramic composites is an intricate issue to the researchers. Silicon carbide-reinforced alumina can produce a widely applicable range in ceramic composites, but test techniques comparison for K_{IC} determination has, apparently, not been made. Attempts are made in this study to compare three macroflaw test techniques, namely: short-bar(SB), four-point single edge-notched bend-bar(SENB), and four-point chevron-notched bend-bar(CHV) tests, all under ambient conditions.

2. Experimental procedure

2.1. Processing and specimen preparation

The basic raw material used for alumina billets is alumina powder (EBON A) of 99.8% purity and SiC whiskers (TWS-100) of 99% purity. Manufacturer's data for silicon carbide whiskers and alumina powder are presented in Tables I and II. A maximum hot-pressing temperature of 1475 °C and a maximum pressure of 2000 p.s.i. (10^3 p.s.i. = 6.89 N mm^{-2}) were

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TABLE I Manufacturer's data for the physical properties of silicon carbide whiskers TWS-100 (Tokai Carbon Co.)

Diameter	0.3–0.6 μm
Length	10–15 μm
Density	3.20 g cm^{-3}
SiC	99 wt %
SiO_2	< 0.5 wt %
Particulate content (< 50 μm)	< 1 wt %
Crystal type	Beta

TABLE II Manufacturer's data for the physical properties of Al_2O_3 matrix* used with SiC whiskers, EBON A (Cercom Inc.)

Purity	99.8%
Average diameter	2.2 μm
Density (min)	3.93 g cm^{-3}
Flexural strength	552 MPa
Compressive strength	2758–3447 MPa
Modulus of elasticity	3.93 GPa

used for alumina, while for the composite, the corresponding temperature of 1755 °C and a pressure of 4500 p.s.i. were employed. In order to ensure material uniformity, both short-bar and bend-bar specimens were cut and machined from a single 102 mm × 75 mm × 14.29 mm billet. Short-bar and bend-bar specimen dimensions were selected to be, as close as possible, to those of the relative dimensions required in testing metallic materials as per ASTM E 1304-89 and ASTM E 399-83, respectively.

2.2. Short-bar tests

The short-bar dimension nomenclature, loading configuration, and relative dimensions of the specimen with respect to the breadth, B, are shown in Fig. 1. Short bars were tested in a fractometer [12] by loading the specimens through inflatable bladder called flatjack. Standard equations [4, 13] were used to calculate stress intensity factors, K_{IC}.

The following validity checks were made [12]:

1. specimen size (breadth for the short bar)

$$B \geqslant 1.25\,(K_{IC(SB)}/\text{yield strength})^2 \qquad (1)$$

where $K_{IC(SB)}$ is the plane strain critical stress intensity for the chevron notched short-bar;

2. plasticity and residual correction factor, P factor

$$-0.05 \leqslant P \leqslant 0.10 \qquad (2)$$

3. the crack must not deviate from the intended plane by more than $0.04B$ at the point where the crack is approximately $0.33B$;

4. at least two unloadings for smooth crack growth materials;

5. for crack jump materials, crack must arrest in the region

$$0.80r_c \leqslant r \leqslant 1.20r_c \qquad (3)$$

where r is the slope ratio corresponding to the crack arrest and r_c is the critical slope ratio (0.55 for the short-bar and short-rod geometry);

6. no obvious pre-existing macroscopic flaws visible in the crack plane.

2.3. Bend-bar tests

The specimen configuration and schematic drawing of loading are shown in Fig. 2. Both the chevron-notched and the straight-through-notched specimens were tested by controlled loading in the Instron. The specimens were loaded at a uniform rate of 0.02 in/min^{-1} to ensure steady crack growth. The load was recorded as a function of machine crosshead displacement autographically. The stress intensity factors, K_{IC}, were calculated using the standard equation [7].

3. Results and discussion

Tests on alumina (EBON A) and silicon carbide-reinforced alumina were consistent with all linear elastic fracture mechanics (LEFM) assumptions. None of the short-bar specimens showed crack jump behaviour. Validity checks as per ASTM E 399, were made in the bend-bar tests and the maximum loads, P_{max}, were used to calculate critical stress intensity factors on the assumption that minimum stress intensity coefficient, Y_m, occurred at the maximum load. In the bend-bar tests, the load–displacement curves were initially non-linear but then became linear up to maximum load, P_{max}, where unstable crack extension occurred. Crack extension in the chevron-notched specimens was relatively smooth except for an occasional discontinuity resulting in a "pop-in" step in the load–displacement record. The scatter in the data and the standard deviations in each group of tests (both on Al_2O_3 and SiC/Al_2O_3) were low, indicating uniformity and consistency of tests in all the groups. Table III presents test result averages with corresponding characteristic life as predicted by Weibull analysis.

3.1. Tests on alumina

The critical stress intensity factors, K_{IC}, obtained from the three test techniques appear to be fairly consistent. Relative to the short-bar tests, the $K_{IC(CHV)}$ averaged 17% higher, while $K_{IC(SENB)}$ averaged 11.85% lower than the average $K_{IC(SB)}$.

3.2. Tests on SiC/Al_2O_3

The results of the short-bar and the single edge-notch bend-bar tests are seen to be in closer proximity than the chevron-notched bend-bar tests. The $K_{IC(CHV)}$ of 7.69 MPa m$^{1/2}$ is 60.88% higher than $K_{IC(SB)}$ and 49.90% higher than $K_{IC(SENB)}$; while $K_{IC(SENB)}$ of 5.09 MPa m$^{1/2}$ is only 6.49% higher than $K_{IC(SB)}$. Short-bar tests, however, yielded lower apparent toughness, $K_{IC(SB)}$, on SiC/Al_2O_3, than the toughness obtained from the tests on monolithic alumina. This ambiguity in the results could not be accounted for.

3.3. Comparison with published test results

A comparison of the measured K_{IC} values for Al_2O_3 and SiC/Al_2O_3 of this study with those for the similar materials of earlier research works is presented in Table IV.

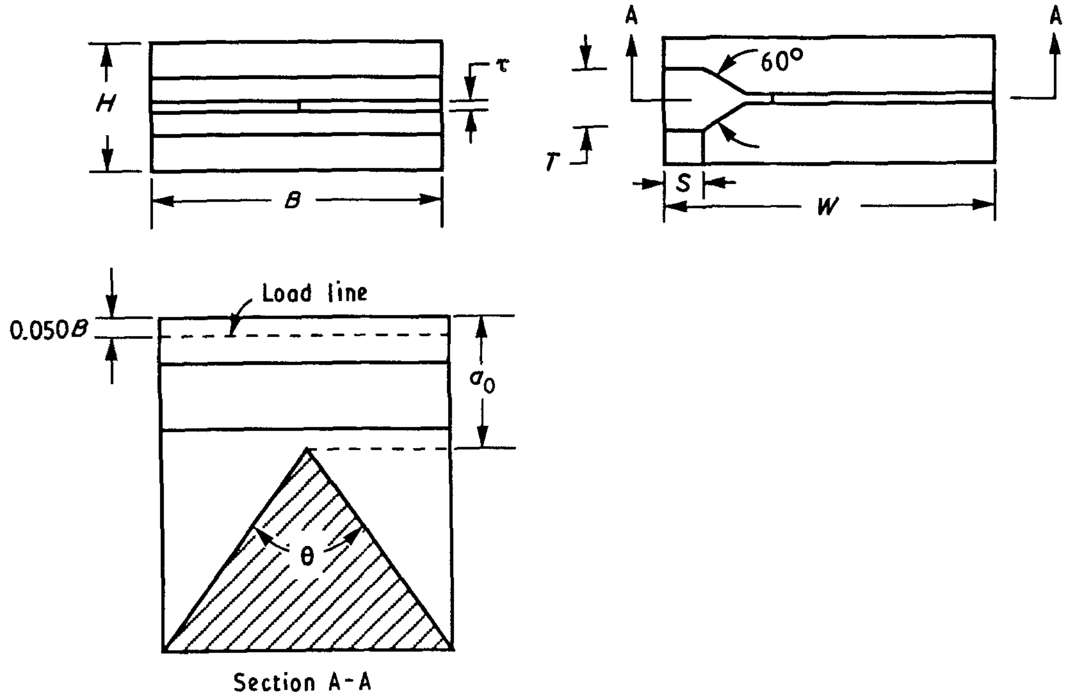

Figure 1 Short-bar specimen dimensions. B, breadth; W, length = $1.5B \pm 0.010B$; H, height = $0.870B \pm 0.005B$; a_0, initial crack length = $0.531B \pm 0.005B$; θ, slot angle = 55.2° ± 0.5°; τ, slot thickness = 0.014 in; S, grip groove depth = $0.130B \pm 0.010B$; T, grip groove width = $0.313B \pm 0.005B$.

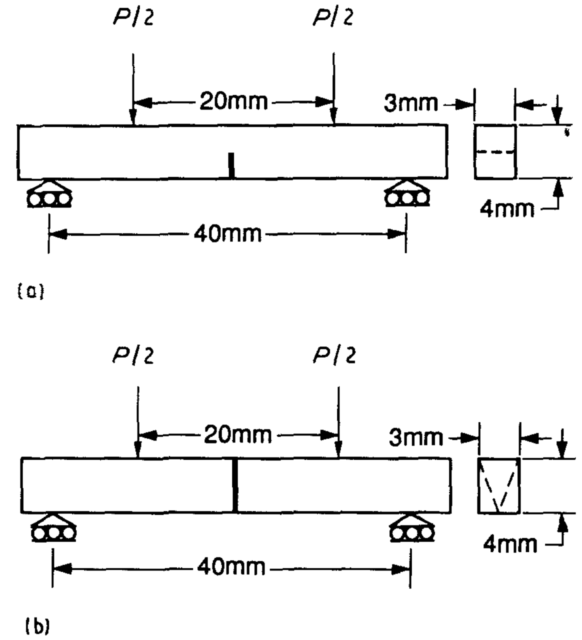

Figure 2 Schematic drawing of bend-bar loading configuration. (a) Straight edge-notched bar; (b) chevron-notched bar.

3.4.1. Alumina (EBON A)

The critical stress intensity factor, $K_{IC(SENB)}$, obtained from these tests compares most favourably with the published results, except with those obtained by Barker [16]. The chevron-notched bend-bar stress intensity factors, $K_{IC(CHV)}$, are higher than the maximum toughness, K_{IC}, obtained from tests on similar polycrystalline materials. The apparent low toughness recorded by Barker was attributed to the use of a smaller calibration factor than would otherwise be required. Subsequently this factor, determined by Barker [23], was increased to match fracture toughness obtained from tests as per ASTM E 399.

3.4.2. SiC (TWS-100)-Al_2O_3 (EBON A)

Table IV shows the widely varying critical stress intensity factors for the SiC/Al_2O_3 composites. The extent to which the materials' physical properties differ, is not known. However, the whisker volume fraction being same (30%), the referenced test results provide a range to check compatibility of the toughness measuring techniques being analysed. Kazi *et al.* [20] determined fracture toughness as a function of whisker volume fraction; the crack plane orientation in his tests is not, however, known. Homney *et al.* [21] measured K_{IC} as a function of composite density, while Mangin *et al.* [22] measured toughness as a function of relative crack length. The relatively high fracture toughness values recorded by Homney *et al.* and Mangin *et al.* vindicate $K_{IC(CHV)}$ obtained in these tests.

The critical stress intensity factor is a function of the measuring technique, processing variables, material properties, and even the testing speed. The crack length and the notch width ratio $\alpha = a/W$, also influence toughness measurements in the bend-bar tests [7, 24]. A review of Mangin *et al.*s' [22] work on SiC/Al_2O_3 and chevron-notched fracture specimens by Newman [25] indicate the effect of the R-curve on fracture toughness. Fig. 3 shows a schematic diagram of the trend of rising R-curve effect on the basis of Newman's study. The R-curve, being also a function of specimen size [26] for stiff materials, calls for the determination of minimum specimen size, B, in testing chevron-notched specimens. The use of chevron-notched specimens with materials that have a rising

TABLE III K_{IC} averages and characteristic values of K_{IC}

	Short-bar K_{IC} (MPa m$^{1/2}$)		Chevron-Notch K_{IC} (MPa m$^{1/2}$)		Straight-notch K_{IC} (MPa m$^{1/2}$)	
Materials	$K_{IC(SB)}$	CL	$K_{IC(CHV)}$	CL	$K_{IC(SENB)}$	CL
Al_2O_3	4.81	4.93	5.63	6.17	4.24	4.27
SiC-whisker/Al_2O_3	4.78	4.85	7.69	7.86	5.09	5.17

TABLE IV Comparison of present K_{IC} of Al_2O_3 and SiC-whisker/Al_2O_3 values with previously published results

Materials	This investigation		Published results		
	K_{IC} (MPa m$^{1/2}$)	Test techniques	K_{IC} (MPa m$^{1/2}$)	Reference	Test techniques
Al_2O_3	4.81	Short bar	4.44	21 [14]	Not known
Al_2O_3	5.63	Bend bar (CHV)	4.00	22 [15]	Not known
Al_2O_3	4.24	Bend bar (SENB)	3.12	14 [16]	Short rod
Al_2O_3	-	-	3.87	[17]	Three-point SENB
Al_2O_3	-	-	4.0–4.2	[18]	Three-point SENB
Al_2O_3	-	-	3.80	[19]	Four-point SENB
SiC/Al_2O_3 (30 % whskr)	4.78	Short bar	6.00	[20]	Three-point SENB
SiC/Al_2O_3 (30 % whskr)	7.63	Bend bar (CHV)	9.50	[21]	Three-point SENB
SiC/Al_2O_3 (30 % whskr)x	5.09	Bend bar (SENB)	8.70	[22]	Four-point CHV

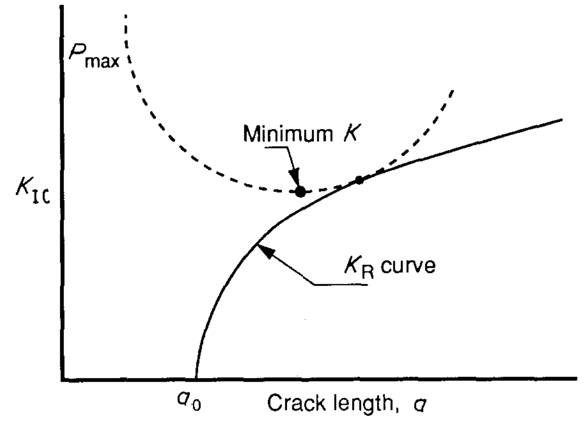

Figure 3 Schematic diagram of K_R curve behaviour.

R-curve may also require stress intensity factors as a function of crack length instead of using only the minimum value [25]. The above considerations further validate the $K_{IC(CHV)}$ results.

3.4.3. Statistical analysis

Variations in the results usually obtained from the fracture toughness tests, including these tests, necessitated statistical analysis of test results. Such an analysis has been made on the results obtained from the SiC/Al_2O_3 tests only, as these were the focal point in these investigations.

The analysis of variance [27] and F distribution [28] made to check statistical equivalence of either all or any combination of two tests, have shown that there is no statistical equivalence in any combination of tests. The statistical dissimilarity in test results very clearly indicates that each test technique bears no relationship to any of the other tests. The statistical analysis re-emphasizes the need for test technique specification when requiring fracture toughness measurements.

4. Conclusions

Monolithic alumina produced acceptable results from both the bend-bar specimen configurations and the short-bar tests. Short-bar and straight-notched bend-bar (SENB) test results on alumina compare well with some published results. Short-bar test results on SiC/Al_2O_3 are not acceptable. Bend-bar tests on chevron-notched (CHV) specimens may have been biased by R-curve effects. Statistical analysis does not indicate "statistical equivalence" in any combination of test techniques employed. From these test results and a review of some published results, the following conclusions can be drawn.

1. The three test techniques employed in this investigation produce comparable results for Al_2O_3. Compliance calibration tests should be conducted on ceramic composite specimens to determine an acceptable K-factor for use in short-bar tests.
2. The use of stress intensity factor as a function of crack length is suggested for chevron-notched bend-bar tests on SiC/Al_2O_3 composite and similar ceramics.
3. Fracture toughness is a test technique dependent property. Toughness data on ceramics and ceramic composites should also include test technique(s) until an accepted standard is available.

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References

1. M. SAKAI, *Bull. Ceram. Soc. Jpn* **23** (1988) 480.
2. G. C. WEI and P. F. BECHER, *Am. Ceram. Soc. Bull.* **64** (1985) 289.
3. T. N. TIEGS, P. F. BECHER and G. C. WEI, "SiC whisker Reinforced Ceramic Composites", Ceramic Technology for Advanced Heat Engines, Semi Annual Report, December (1984).
4. L. M. BARKER, *Engng Fract. Mech.* **9** (1977) 361.
5. J. H. UNDERWOOD, "Fracture Tests for Metals; Can they apply for Ceramics and Glass-based Composites", Presentation at ASME E-24 Committee Week Workshop on Testing Method for Ceramic Matrix Composites, Nashville, TN, November (1985).
6. A. G. EVANS and E. A. CHARLS, *J. Am. Ceram. Soc. Discuss. Notes* July-August (1976) pp. 371–372.
7. D. MUNZ, R. T. BUBSEY and J. L. SHANNON Jr. *J. Amer. Ceram. Soc.* **63** (5, 6).
8. *Idem, J. Test Evaluation* **8** [3] (1980) 103–107.
9. R. F. KRAUSE Jr, *et al.*, "Fracture Toughness of Polymer Concrete Materials Using Various Chevron-Notched Configurations", ASTM STP 855 (American Society for Testing and Materials, Philadelphia, PA, 1984) pp. 309–23.
10. W. L. VAUGHEN, unpublished thesis, University of Illinois, Urbana-Champaign.
11. B. MUSSLER, M. U. SWAIN and N. CLAUSSEN, *J. Amer Ceram. Soc.* **65** (1982) 566.
12. Terra Tek Systems, "Chevron-notched Short Bar Fracture Toughness Testing of Alumina Ceramics for Tuskegee University", University Research Park, 360 Wakara Way, Salt Lake City, UT 84108, USA.
13. L. M. BARKER, *Int. J. Fract.* **15** (1979) 515.
14. G. D. SWANSON, *J. Amer. Ceram. Soc.* **55** (1972) 48.
15. L. A. SIMPSON, *ibid.* **56** (1973) 610.
16. L. M. BARKER, paper presented at the "International symposium on Fracture Mechanics of Ceramics", Pennsylvania State University, July (American Ceramic Society, 1977).
17. M. G. JENKINS, A. S. KOBAYASHI, M. SAKAI, K. W. WHITE and R. C. BRADT, *Amer. Ceram. Soc. Bull.* **66** (1987) 1734.
18. P. L. PRATT, *Fracture* **3** (1977) 909.
19. H. HUBNER and W. JILLEK, *J. Mater. Sci.* **12** (1977) 117.
20. M. KAJI, S. KOSHAKA and K. KOGA, "Fracture Behavior of Alumina matrix Composites", 91st annual meeting, April 25, Indianapolis (American Ceramic Society, 1989) pp. 23–27.
21. J. HOMNEY, W. L. VAUGHEN and M. K. FERBER, *Amer. Ceram. Soc. Bull.* **67** (1987) 333.
22. C. MANGIN, J. HOMNEY and S. S. WANG, Unpublished thesis, University of Illinois, IL (1989).
23. L. M. BARKER, "Compliance Callibration of A Family of Short Rod and Short Bar Fracture Toughness Specimens" (Terra Tek, University Research Park, 420 Wakara Way, Salt Lake City, UT 84108, 1981).
24. *Idem, J. Test. Evaluation* **17** [4] (1989) 218–223.
25. J. C. NEWMAN, "A Review of Chevron-Notched Fracture Specimen", Chevron notched specimens: Testing and Stress Analysis, ASTM STP 855 (American Society for Testing and Materials, Philadelphia, PA, 1984).
26. ASTM E 646-78. "Standard Practice for Plane-Strain Fracture Toughness Testing of Aluminium Alloys", Annual book of ASTM standards, Section 2, Vol. 02.02 (American Society for Testing and Materials, Philadelphia, PA, 1984) pp. 661–5.
27. RONALD L. IMAN and W. J. CONOVER, "Modern Business Statistics" (Wiley, 1983).
28. W. ALLEN WALLIS and HARRY V. ROBERT, "Statistics A New Approach" (The Free Press, Glencoe, IL, 1957)
29. M. V. SWAIN and N. CLAUSSEN, *Commun. Amer. Ceram. Soc.* **C-27** **66** (2) (1983) C-27.

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