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The Technical Committee 6 of the European Structural Integrity Society organized a round robin on determining the fracture toughness of ceramic materials. Five materials were tested with five testing methods by eighteen laboratories. The five testing methods were: chevron notched beam in four point bending, direct measurement of the cracks emanating from a Vickers indentation, indentation strength by four point bending, single edge precracked beam in four point bending and single edge notched beam in four point bending. The results are presented and discussed.

INTRODUCTION

Fracture toughness (K_{Ic}) is a critical design parameter for structural components made of ceramic materials. The Technical Committee 6 of the European Structural Integrity Society (ESIS TC 6) organized a round robin on determining the fracture toughness of ceramic materials. The following five testing methods were investigated:

- chevron notched beam in four point bending (abbreviated as CVN method)
- direct measurement of the cracks emanating from a Vickers indentation - indentation fracture (IF method)
- indentation strength by four point bending (IS method)
- single edge precracked beam in four point bending (SENB-B method)
- single edge notched beam in four point bending (SENB-S)

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These testing methods were applied on the following five ceramic materials: Al₂O₃ (Desmarquest, France), HPSN (BASF, Germany), SSiC (Hutschenreuther, Germany), MgPSZ (Friedrichsfeld GmbH, Germany), ZrO₂ (Risø, Denmark).

The objectives of the round robin were to compare the accuracy, reproducibility and practicability of the testing methods for various ceramic materials. The reproducibility of a testing method is reflected in the standard deviation of the results within and between the laboratories. The practicability of a testing method is discussed by comparing the statements received from the participating laboratories.

MATERIALS AND SPECIMENS

The densities, Young's moduli and grain sizes of the five materials used in this round robin are given in Table 1. The Young's moduli needed to evaluate the fracture toughness values with the IF and IS methods were supplied to the participants.

TABLE 1 - Densities, Young's moduli and grain sizes of the ceramic materials used in the round robin

	Al ₂ O ₃	HPSN	SSiC	MgPSZ	ZrO ₂
density [g/cm ³]	3.81	3.16	3.15	5.65	6.12
Young's modulus [GPa]	359	312	427	208	213
grain size [μm]	16	1	7	60	0.4

The specimens were cut to the dimensions of 45 x 4 x 3 mm.

EXPERIMENTAL PROCEDURE

CVN METHOD. The CVN method requires the cutting of a V-shaped chevron notch into the specimen. The specimen is loaded in four point bending, during which a crack is first initiated at the chevron tip. It is necessary that the crack then propagates in a stable manner until catastrophic failure occurs. A general overview of this testing method is given in [1].

The advantages of this testing method are:

- a sharp crack is formed at the chevron notch tip as soon as the crack initiation load is reached
- the maximum load is easy to measure
- no crack length measurements are required

The disadvantage is the comparatively expensive machining of the chevron notch.

IF METHOD. If a sharp indenter like a Vickers diamond is driven into a brittle material, cracks are formed. The fracture toughness of the material can be

determined from the geometry and the dimensions of the cracks, which are measured at the surface of the specimen. More background on this testing method is given in [2].

The correct determination of the fracture toughness requires the formation of straight cracks that are sufficiently larger than the indentation. Branching crack systems can form in ceramic materials with large grain sizes. Lateral cracks, which run parallel to the specimen surface, can cause chipping. This invalidates the test.

The advantages of the IF method are:

- easy to perform, no bending tests are required to determine fracture toughness
- only small specimens are needed
- small and therefore realistic crack sizes are evaluated

and the disadvantages are

- crack length measurements are susceptible to errors
- subcritical crack growth due to residual stresses is possible
- the specimen surface needs to be polished

IS METHOD. The IS method applies a Vickers indentation as a precrack to the specimen that is fractured in four point bending. The precrack experiences some stable crack growth in four point bending before the occurrence of catastrophic failure of the specimen. More information is given in [3].

The advantages of the IS method are:

- no crack length measurements are required
- small and therefore realistic crack sizes are evaluated (the tested crack sizes are typically several hundred micrometers)
- in general no special surface treatment is necessary
- relatively easy to perform

and the disadvantages are:

- the change of the crack geometry during the crack extension before instability length is reached are not considered in the equation for evaluation.
- lateral cracks can cause chipping which invalidates the test

SENB-B METHOD. In the SENB-B method a sharp precrack is introduced into a specimen by the bridging method. This involves placing a Vickers indentation in the middle of a specimen surface that is then loaded with a double-anvil loading fixture. The stress field developed by such a fixture promotes the growth of a crack to a certain depth. The precracked specimen is subsequently fractured in four point bending. The SENB-B method is discussed in [4].

The advantages of the SENB-B method are:

- a sharp precrack
- measurement of the precrack length after test possible
- a well-defined crack size

and the disadvantages:

- large precracks are tested
- the front of the precrack is often not straight and the precrack often does not lie in a plane
- difficult to control the depth of the precrack

SENB-S METHOD. For the SENB-S method a single straight edge notch is machined in the specimen that is then loaded until fracture in four point bending.

It is necessary to determine the critical notch root radius ρ_{crit} , which is a material parameter. The relation between ρ_{crit} and the microstructural features of the material is given in [5]. Above ρ_{crit} the measured value increases linearly with $\rho^{1/2}$. Therefore the results of the tests in which the measured ρ values were above this critical value should not be taken into account.

The advantages of the SENB-S method are:

- a well-defined notch
- easy measurement of the precrack depth
- relatively inexpensive machining

and the disadvantages are:

- no sharp notch tip
- the need to determine the critical notch root radius ρ_{crit}
- a small notch width may be required depending on the material

RESULTS

The results for the tested materials are given in Table 2. The scatter in the measured fracture toughness values for each testing method applied can be described with 3 statistical quantities [6]: the standard deviation of all data for each testing method, s_{tot} , the deviation of the weighted mean values determined in different laboratories from the overall mean value, $s_{between}$, and the deviation of all data from the mean values obtained in single laboratories, s_{within} . The deviation 'within' laboratories s_{within} gives a rough estimate of the inherent material variability of the measured fracture toughness, whereas the deviation between laboratories $s_{between}$ gives an estimate of the systematic variability of the specific testing method applied to one specific material. A comparison of those quantities will reveal which variability (the inherent material variability or the systematic variability) is more influential on the measured fracture toughness.

The detailed results are listed in [7].

TABLE 2 - Summary of statistical data of the round robin

Al_2O_3	average [MPa $\sqrt{\text{m}}$]	s_{tot} [MPa $\sqrt{\text{m}}$]	$s_{between}$ [MPa $\sqrt{\text{m}}$]	s_{within} [MPa $\sqrt{\text{m}}$]	number of tests	number of labs
CVN	3.92	0.45	0.77	0.39	56	7
IF	3.90	0.78	2.06	0.64	178	10
IS	4.22	0.41	0.71	0.36	105	12
SENB-B	4.67	0.86	2.31	0.51	64	7
SENB-S	3.17	0.19	0.39	0.16	80	9
HPSN						
CVN	8.22	1.28	–	–	9	1
IF	4.50	0.51	1.18	0.45	43	3
IS	4.73	0.32	0.46	0.30	22	3
SENB-B	4.38	0.72	2.12	0.40	13	2
SENB-S	7.48	1.22	1.59	1.17	33	4
SSiC						
CVN	2.72	0.29	0.79	0.22	17	2
IF	2.13	0.29	1.38	0.18	111	4
IS	3.13	0.41	1.21	0.30	18	2
SENB-B	2.09	0.45	1.15	0.32	13	2
SENB-S	3.52	0.46	0.19	0.47	24	2
MgPSZ						
CVN	10.66	0.66	0.17	0.68	17	2
IF	7.68	1.89	4.82	1.81	68	2
IS	10.22	1.54	2.58	1.39	32	4
SENB-B	8.93	2.17	4.62	1.76	35	4
SENB-S	8.18	0.90	2.20	0.68	52	5
ZrO₂						
CVN	6.78	0.92	2.28	0.73	16	2
IF	4.35	0.46	–	–	60	1
IS	6.43	1.13	1.26	1.12	19	2
SENB-B	4.63	0.83	3.16	0.39	19	2
SENB-S	7.83	0.67	1.28	0.58	20	2

CONCLUSIONS AND DISCUSSION

The following conclusions are drawn from the round robin:

The measured fracture toughness value depends on the testing method and testing conditions.

The determination of a consistent fracture toughness that is comparable between testing laboratories requires a detailed prescription of specimen preparation, testing method and testing condition.

The CVN method results in reproducible fracture toughness values if stable crack growth occurs during the four point bending test. The occurrence of stable crack growth is seen from the load vs. displacement plot provided the testing machine is

sufficiently stiff. This is a reliable method of judging the validity of the CVN test. The machining of the CVN specimen is expensive.

The results from the IF method are less accurate and exhibit larger scatter compared to other methods because of inaccuracies in the measurement of the length of small cracks. In addition, the results of the IF method are sensitive to sub-critical crack growth. On the other hand this testing method is very fast and inexpensive requiring only small specimens. The method is recommended for material development purposes and material comparisons within one laboratory, but can not be used to compare fracture toughness values between laboratories.

The IS method gives reproducible results. Realistic crack sizes are tested. The obtained data scatter is generally larger than that obtained for correctly applied CVN and SENB-S methods. Chipping can invalidate the test results.

The SENB-B method tests unrealistically large cracks. Reproducible results can be obtained if the crack depth is limited to a narrow range.

The SENB-S method produces reliable results if the used notch width does not exceed the critical notch width. The critical notch width depends on the tested material. If this condition is met the scatter obtained with the SENB-S method is small. The experimental determination of the critical notch width is seen to be impractical.

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