FRACTURE TOUGHNESS OF CERAMICS USING THE SEVNB METHOD; ROUND ROBIN

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ABSTRACT

The fracture toughness was measured by the Single-Edge-V-Notched Beam (SEVNB) method on five ceramics in an international round robin with more than 30 participants. Very consistent fracture toughness results were obtained for a coarse-grained alumina-998 and a sintered silicon carbide (3.57 ± 0.22 MPa √m; 2.61 ± 0.18 MPa √m), consistent ones for a gas pressure sintered silicon nitride (5.36 ± 0.34 MPa √m) and reasonably consistent ones for a fine-grained alumina-999 (3.74 ± 0.40 MPa √m), respectively. As predicted, less consistent results were obtained for an yttria-stabilised tetragonal zirconia polycrystal due to its grain size in the submicron range (5.34 ± 0.65 MPa √m). Only the mean for the alumina-998 differed significantly from other credible test methods. A combination of a high sensitivity to subcritical crack growth and a pop-in of small cracks to form a crack "initiation" seems to be responsible for the discrepancy. The SEVNB method proved to be forgiving and robust with respect to the notch preparation, notch width, notch depth or optical notch quality for ceramics with an average grain size or major microstructural feature size of greater than about 1 µm. Most participants had no difficulties and rated the method user-friendly, easy, reliable, accurate and worthwhile for standardisation.

INTRODUCTION

Many methods are currently used to measure the fracture toughness of ceramic materials. Methods based on accepted theories like Surface Crack in Flexure (SCF), Chevron Notch (CN), Single-Edge-Precracked Beam (SEPB) or Single-Edge-Notched Beam (SENB) are often difficult to realise, unreliable, or expensive. Quinn, Gettings and Kübler [1] demonstrated in a Versailles Project of Advanced Materials and Standards (VAMAS) round robin that accurate fracture toughness values can be measured with the SCF method. However, they also showed that making the small cracks and finding their crack front after the test can, depending on the material, range from being simple to very challenging, if not impossible. The CN test is simple to conduct, but Himsolt, Munz and Fett [2] stated that the generation of a sharp crack could not be ensured in all tests. Nishida, Hanaki and Pezzotti [3] concluded that practical problems with the SEPB method make the fracture toughness determination difficult to apply and even unsuitable for some ceramics. The simple SENB method, on the other hand, can be influenced by the notch width, as e.g. Primas and Gstrein [4] discovered in an European Structural Integrity Society (ESIS) round robin. In a more detailed analysis Damani, Gstrein and Danzer [5] suggest that the stress field around the notch tip is responsible for the notch width effect. They show that the notch width must be on the order of the size of the relevant microstructural or machining-induced defects. Nishida, Hanaki and Pezzotti [3] recently reintroduced an interesting technique described earlier by Le Bac [6] to taper a saw cut to a sharp V-notch using a razor blade sprinkled with diamond paste. This method, known as the Single-Edge-V-Notched Beam (SEVNB) method, relates also to basic work conducted by Awaji and Sakaida [7].

With the aim to examine whether the SEVNB method is user-friendly, reliable, and most important, comparable with other recognised methods, a preliminary study with six ceramics, all used in previous international fracture toughness round robin tests was conducted by the author [8]. After this promising study a detailed instruction was written and validated in a mini-round robin with two laboratories. Finally, over 30 companies and institutes from Europe, USA, Japan, Australia and Brazil participated in this fracture toughness round robin conducted jointly under the ESIS and VAMAS organisations.

MATERIAL

In the round robin, five ceramic materials with varying fracture toughness measurability were used. An alumina-999, a silicon carbide (SSiC) and an yttria-stabilised tetragonal zirconia (Y-TZP) were optional, but each participant was required to test an alumina-998 and a gas-pressure sintered silicon nitride (GPSSN). Details of the materials are shown in Table 1.

TABLE 1

PROCESSING PARAMETERS AND MATERIAL PROPERTIES

	alumina-998	alumina-999	GPSSN	SSiC	Y-TZP
Powder	> 99.8 %	> 99.9 %	Bayer N3208		Tosoh TZ-3Y
pressed sintered post hipped	CIP [2)] air	CIP [2)] air gas-pressure	 gas-pressure	HP [3)]	CIP [2)] air gas-pressure
Grain size	>10 µm	~1.7 µm	< 1 µm, elong.	7 µm	0.45 µm
Density	3.86 g/cm^3	3.97 g/cm^3	3.23 g/cm^3	3.15 g/cm^3	6.03 g/cm^3
Strength	342 MPa [1)]	350 MPa [1)]	> 920 MPa [1)]		> 750 MPa [1)]

[1)] 4-point bending   [2)] cold isostatic pressed   [3)] hot pressed

EXPERIMENTAL PROCEDURE

Each participant received a package containing five 3 x 4 x 45 mm alumina-998 and GPSSN bend bars and instructions detailing how to conduct the round robin. Some participants received bend bars from the other three ceramics, too. All bend bars were prepared in accordance with standard EN 843-1. The participants had to measure the fracture toughness on these bend bars with the SEVNB method. Further, volunteering participants received additional specimens of the aluminas and the GPSSN on which they were asked to measure the fracture toughness using their preferred method (SCF, SEPB, CN, etc.).

The fracture toughness with the SEVNB method had to be measured in 4-point bending tests with spans of 40 / 20 mm. Before testing, each participant was required to cut the V-notches at the centre of the specimens' tensile surface, as shown in Figure 1. The V-notches could be produced either by hand or by machining. The general procedure could be performed in three steps: 1) Mount five specimens parallel and side by side with their compression surface down on a plate, 2) with a thin diamond wheel cut a straight notch to a depth of about 0.5 mm, and 3) polish a second deeper notch into the first one using the slot as a guide with a razor blade sprinkled with diamond paste.

Afterwards, the participants had to compute the fracture toughness K_{Ic} using Eqn. 1:

$$K_{Ic} = \frac{F}{B\sqrt{W}} \cdot \frac{S_1 - S_2}{W} \cdot \frac{3\sqrt{\alpha}}{2(1-\alpha)^{1.5}} Y^* \tag{1}$$

with: $Y^* = 1.9887 - 1.326\alpha - (3.49 - 0.68\alpha + 1.35\alpha^2)\alpha(1-\alpha)(1+\alpha)^{-2}$

where: F, S_x, a, B fracture load, span, notch depth, specimen width, respectively

W, α, Y^* specimen height, a/W, stress intensity shape factor, respectively

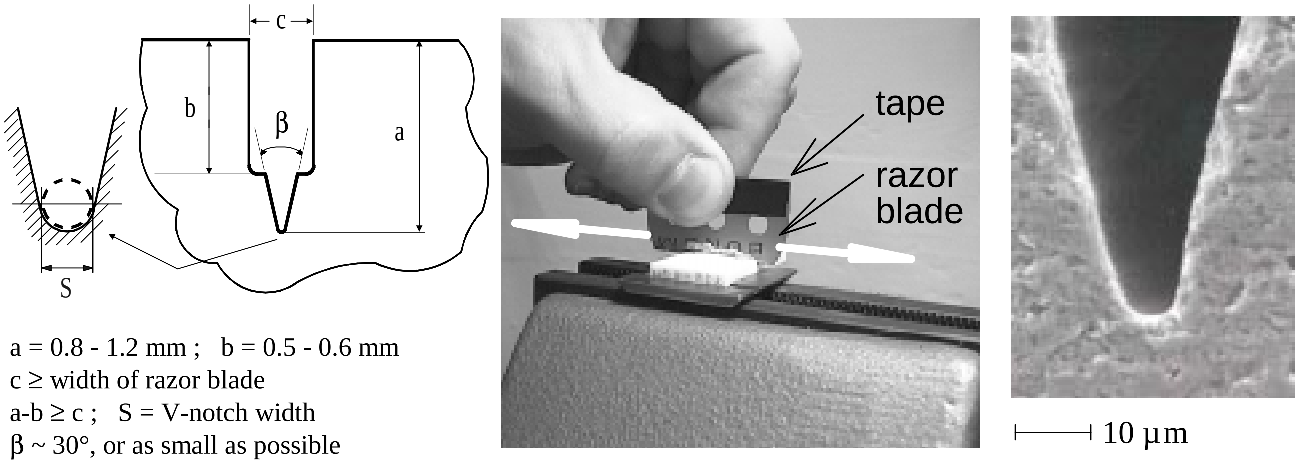


Figure 1:
Left: Schematic geometry of V-notch.
Middle: V-notch polishing by hand.
Right: V-notch tip on a SSiC specimen.

RESULTS AND DISCUSSION

Table 2 lists the Grand Population Average and Standard Deviation for all materials tested. For a first validity check of the round robin test results, graphs as shown in Figure 2 were used. No significant influence of the notch width below 30 µm could be seen. Therefore, a notch width of 30 µm was chosen as criteria to accept a result or not. No results were rejected on the basis of either an incorrect V-notch geometry or the use of another test procedure than 4-point bending. The success rate of the accepted participants conducting the SEVNB method was better than 93 % (4.7 specimens out of 5 per participant) for all ceramics.

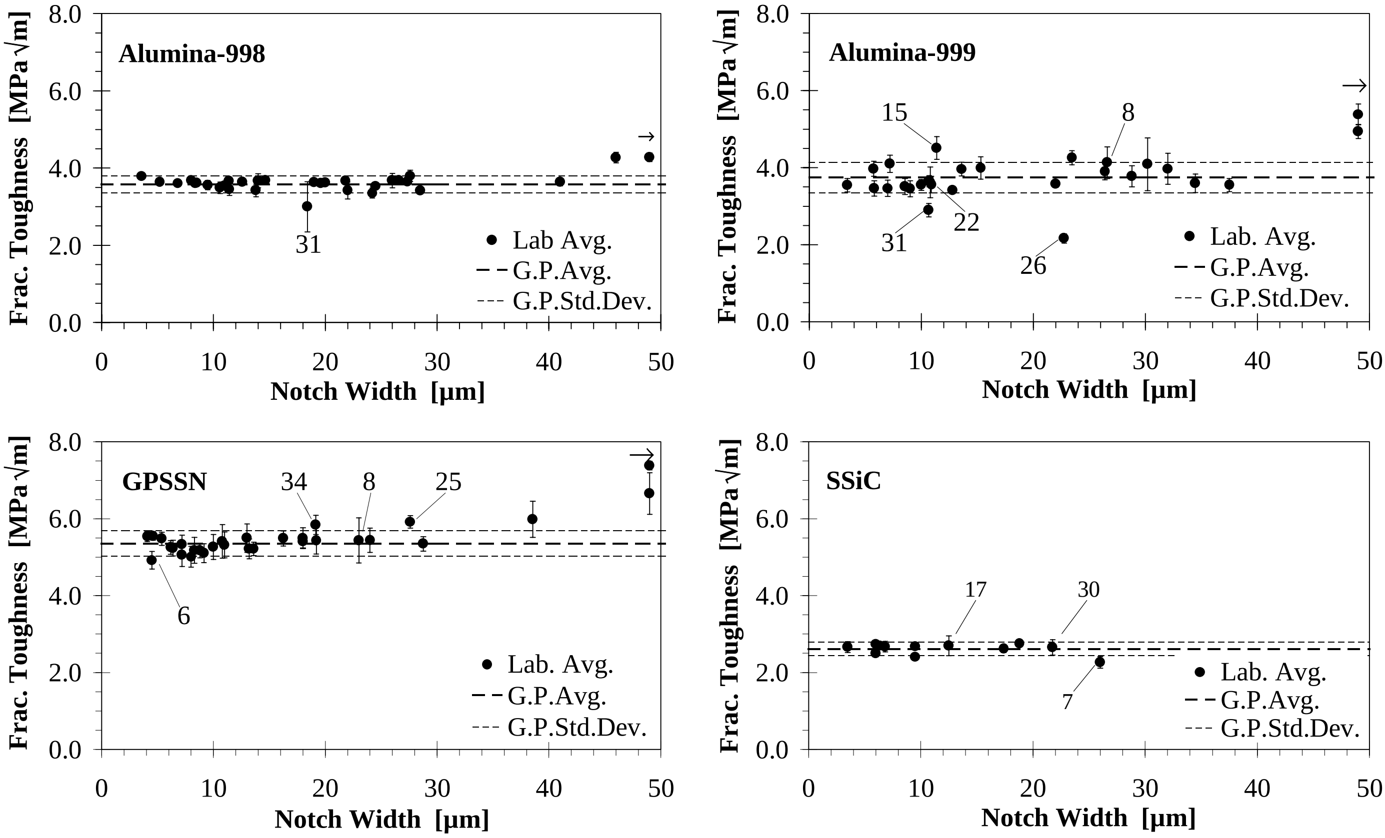
TABLE 2

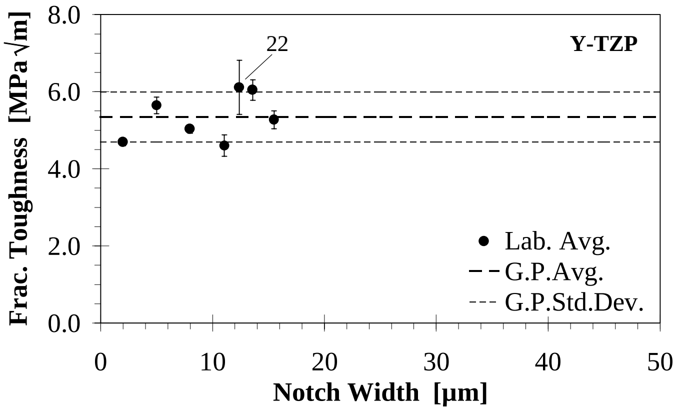
GRAND POPULATION AVERAGE (G.P.Avg.) AND STANDARD DEVIATION (G.P.Std.Dev.).

Material	Participants (total)	Total # of specimens	G.P.Avg. MPa √m	G.P.Std.Dev. MPa √m
alumina-998	28 (31)	135	3.57	± 0.22
alumina-999	21 (28)	102	3.74	± 0.40
GPSSN	27 (30)	129	5.36	± 0.34
SSiC	12 (12)	56	2.61	± 0.18
Y-TZP	7 (7)	35	5.34	± 0.65

Notch quality, width and depth

Twelve participants polished the V-notches by hand and nineteen used a machine. Typical polishing times for 5 specimens were 0.5 h for the aluminas and 1.5 h for the GPSSN and SSiC. To polish the notches, the participants used razor blades with thicknesses ranging from 100 to 250 µm. The polishing stroke length varied from about 4 to 20 mm and the stroke frequencies from 1 to 15 Hz. The loads on the razor blades ranged from 1 to 5 N and the diamond pastes used had grain sizes from 0.5 to 3 µm. An analysis of all information furnished by the participants did not give a clear picture on the influence of the parameters. The general impression is that it is easy to polish V-notches with a width between 20 and 30 µm, but the parameters need to be optimised for each combination of polishing machine or hand, razor blade, stroke length, frequency, and load for notches smaller than 10 µm.





Master result graphs showing the individual participant's (Lab.) average with standard deviation vs. the notch width. The bold dashed lines represent the grand population average (G.P.Avg.) and the fine dashed lines the grand population standard deviation (G.P.Std.Dev.). Results from tests with notches > 30 µm were not used to compute the G.P.Avg. and G.P.Std.Dev. The numbers "identify" the participant responsible for that result.

Figure 2: Master result graphs.

The fracture toughness measured on edge notched bend bars can be influenced by the notch width as well known. From a practical point of view, this had been demonstrated e.g. by Primas and Gstrein [4] in an ESIS round robin. Munz and Fett [9] found that the measured fracture toughness rises above a critical notch width. Munz, Bubsy and Shannon [10] reported a good agreement between critical stress intensity values measured on an alumina by the CN beam and the SENB in bending, provided the notch width was less than 70 µm. Kübler [8] found empirically that fracture toughness values which are comparable with other methods, could be measured if the notch width was less than about twice the size of a major microstructural feature, e.g. the average grain size. An interesting theoretical work by Fett [11], a study by Damani, Gstrein and Danzer [5] and a new yet unpublished analysis by Fett [12] seem to confirm the criteria $S \leq 2 \cdot a_{mfs}$ (S: acceptable notch width; a_{mfs}: major microstructural feature size) to estimate an acceptable notch width for a material. Therefore, additional GPSSN bend bars with varying notch widths were tested to get an idea about the effective notch width influence under exactly the same test conditions. Already at very small notches, an increase of the measured fracture toughness was noticeable. Figure 3 shows firstly that an acceptable notch width of 7 µm calculated from the relation $S \leq 2 \cdot a_{mfs}$ (a_{mfs} = 3.5 µm ~ length of elongated grains) is in good agreement with the test data and secondly that the measured fracture toughness does not drop out of the Grand Population Standard Deviation band up to a notch width S ~ 20 µm.

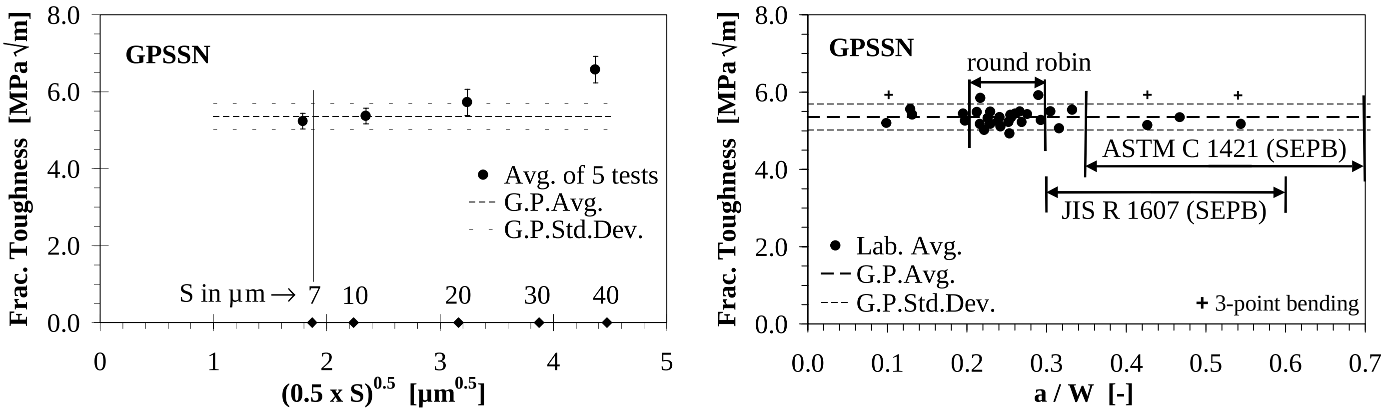


Figure 3:
Left: Influence of V-notch width S on fracture toughness.
Right: Influence of depth ratio a/W on fracture toughness.

The participants were asked to polish their notches to a depth between 0.8 and 1.2 mm resulting in an a/W-ratio between 0.2 and 0.3. This narrow range had been chosen to assure a minimal influence of different notch depths on the measured fracture toughnesses and because the measured fracture toughness can increase with an increasing crack (notch) length as shown by Schindler [13] and because shorter notches are polished faster. Additional bending tests conducted for the aluminas and the GPSSN with a/W-ratios between 0.1 and 0.55 showed no significant influence of the notch depth (Figure 3). Therefore, it is supposed that between a/W = 0.2 and 0.5 none or only a small notch depth dependence of the measured fracture toughness exists.

Repeatability and Reproducibility

To determine the repeatability (within-laboratory) and reproducibility (between-laboratories) of the SEVNB method, the standard ISO 5725-2 "Accuracy (trueness and precision) of Measurement Methods and Results“ was used. The statistical results are shown in Table 3 and compared with results from an earlier VAMAS round robin with the SCF method [1]. The precision and bias statements for the SCF method were computed on the same basis but in accordance with ASTM E 691, which computes repeatability and reproducibility in the identical manner. The data in Table 3 show that the repeatability and reproducibility of the SEVNB method compares very well with the SCF method.

In a next step the results of the participants were assessed with respect to stragglers (test statistic between the 5 % and 1 % critical values) and outliers (test statistic greater than the 1 % critical value) in accordance to Mandel’s *h* and *k* statistics as explained in ISO 5725-2. The specimens and test technique of participants' rated stragglers or outliers were analysed more carefully.

Alumina-998

The within- and between-laboratory consistencies of participant 31 (Figure 2) were so poor that they were rated outliers. The analysis showed that two toughness values were very low. On request the participant analysed the tested specimens fractographically, but could not find any signs of subcritical crack growth (scg) or other defects responsible for the low values.

Alumina-999

Participant 26 reported only two, very low fracture toughness values. His other three specimens failed during handling. The participant was asked to analyse the fractured surfaces fractographically. On both specimens he found large precracks. Because of the precracks the values were discarded.

The within-laboratory consistencies of participants 8 and 22 and also the between-laboratory consistencies of participants 15 and 31 were rated stragglers but none an outlier. The reason for the stragglers is not clear yet. It might be of interest that participants 8 and 22 had problems resetting the razor blade properly into the notch and therefore polished some small additional notches at the notch root. On the other hand, participant 31 explained that two specimens failed while polishing the V-notches. This, together with the fact that he measured already two very low fracture toughness values for the alumina-998 could indicate that he precracked some or all of his alumina specimens during preparation.

Compared with the alumina-998, GPSSN and SSiC the coefficients of variation for the repeatability and especially the reproducibility are rather high. This could be due to an environmental influence, e.g. subcritical crack growth. Therefore, additional sets of five bend bars each were tested in air, nitrogen atmosphere and also water to suppress scg and support scg, respectively. Interestingly, the measured fracture toughnesses were ranked $K_{air} < K_{water} \approx K_{nitrogen}$ (3.54 MPa √m < 3.97 MPa √m ≈ 4.00 MPa √m). Therefore, if scg influences the measured fracture toughness the notch tips of the specimens tested in water had to be protected, e.g. by polishing paste remains or H_2O-water molecules had more difficulties to reach the notch tip than H_2O-air ones.

GPSSN
The within-laboratory consistency of participant 8 and the between-laboratory consistency of participant 25 were so poor that they were rated outliers. Further, the between-laboratory consistencies of participants 6 and 34 were in the range of stragglers. The analysis showed that participant 25 had used a 4-point bending jig with spans of only 30 / 10 mm instead of the required 40 / 20 mm and that the supporting rollers were set in grooves and thus not free to roll. Constrained rollers can lead to an overestimate of the bending strength of up to 5 % and could explain the high values measured. The advancing route of the V-notch tip on the specimens of participant 34 was approx. 20 °, which is about twice the permitted value set by standard JIS R 1607 for the crack tip if tested with the SEPB method. This will lead to an overestimate of the bending strength and might explain the high toughness values. No irregularities in the test technique of participant 6 and 8 could be found.

TABLE 3
REPEATABILITY AND REPRODUCIBILITY OF THE SEVNB METHOD
(COMPARED WITH THE SCF METHOD)

Material	Method	Total Particip.	Total Spec.	Repeatability (within-lab) Std.Dev. [2] MPa √m	CV [3] %	Reproducibility (between-lab) Std.Dev. [2] MPa √m	CV [3] %
alumina-998	SEVNB	28	135	0.17	4.6	0.22	6.1
alumina-999	SEVNB	21	102	0.23	6.2	0.40	10.7
GPSSN	SEVNB	27	129	0.28	5.3	0.34	6.3
SSiC	SEVNB	12	56	0.12	4.5	0.18	6.8
Y-TZP	SEVNB	7	35	0.33	6.2	0.68	12.7
hot pressed Si_3N_4	SCF [1]	19	102	0.24	5.4	0.31	6.8
hot iso-pressed Si_3N_4	SCF [1]	15	100	0.38	7.7	0.45	8.9

[1] earlier VAMAS round robin [1] [2] standard deviation [3] coefficient of variance

SSiC
The within-laboratory consistencies of participant 17 was rated an outlier and the one of participant 30 a straggler even though their standard deviations were only 0.26 MPa √m and 0.20 MPa √m, respectively. Further, the between-laboratory consistency of participant 7 was in the range of a straggler. This is not too

surprising because absolute values of the within- and also the between-laboratory consistencies were the smallest of all five ceramics tested. It might be of interest that precracks (pop-in) were recognised on the fractured surfaces of two not accepted specimens from participant 17 and that participants 7 and 30 reported specimens which failed before the actual testing.

Y-TZP

The between-laboratory coefficient of variation of 12.7 % was the largest of all five ceramics tested. This is not further surprising because of the notch width dependence discussed earlier. No stragglers and outliers were found with respect to the between-laboratory consistency, but interestingly one outlier with respect to the within-laboratory consistency. Participant 22 who already had difficulties before testing the alumina-999 and GPSSN had reported this outlier.

Comparison of results with other methods

All fracture toughness values measured with the SEVNB method, excluding the alumina-998 ones (Figure 4), compared well with values determined by other methods like SCF, CN or SEPB. Additional SEVNB tests on the alumina-998 showed no environmental, test speed or notch depth influence (dry N_2, silicon oil, 100-times slower test speed, dynamic fatigue tests with SEVNB specimens using test rates of 0.005 and 50 mm/min, increased notch depth of 2.1 mm). The discrepancy might be explained by a study from Nishida, Pezzotti, Mangialardi and Paolini [14] performed with SEVNB specimens to detect the near-tip R-curve behaviour and to discern it from the long-crack R-curve behaviour. In a coarse grained (18 µm) alumina with a microstructure similar to the one of the alumina-998, they detected and quantified the increase in the measured fracture toughness as 1.5 MPa √m within the first 10-20 grains just beyond the notch tip.

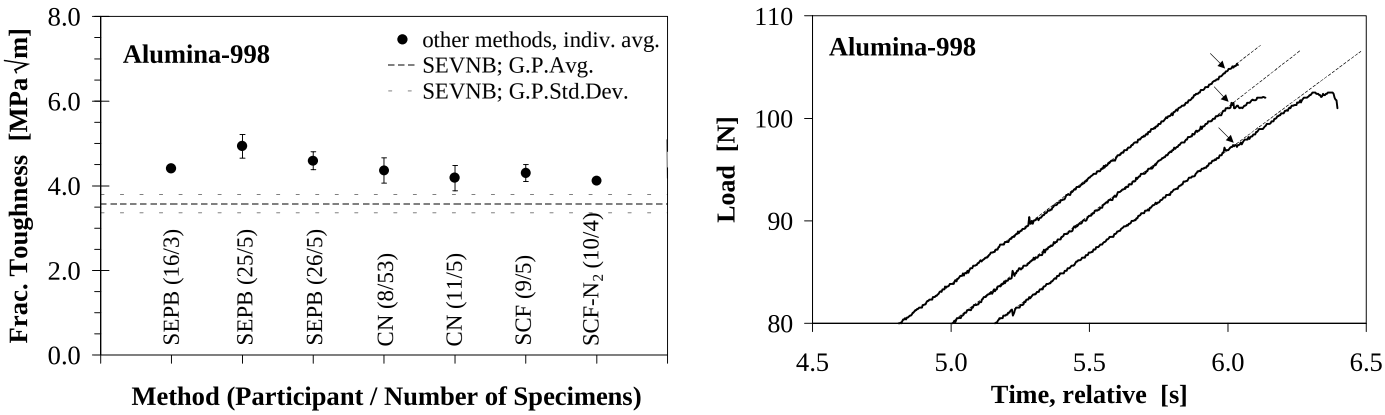


Figure 4:

Left: Comparison of fracture toughness values measured with other test methods with values from the SEVNB method.

Right: Load-time curves (◊ first deviation from linear behaviour).

Another possibility could be stable crack growth or a pop-in during all SEVNB tests. An undetected crack growth of ≈ 250 µm would increase the fracture toughness from 3.6 to 4.2 MPa √m and would reach the value measured with the SCF method. One participant, an experienced fractographer, saw evidence of a 50 to 150 µm crack "initiation" region at the root of the notches. A closer look at digitally recorded load-time curves showed evidence of stable crack growth, as can be seen in Figure 4. The load-time curves of the alumina-998 do not have a linear behaviour in the final part before the failure of the specimen.

CONCLUSIONS

With the SEVNB method very consistent results were obtained for the coarse-grained alumina-998 and the sintered silicon carbide. For the gas pressure sintered silicon nitride and the fine-grained alumina-999 consistent and reasonably consistent results were obtained, respectively. As predicted, the results for the yttria–stabilised tetragonal zirconia polycrystal were less consistent due to its grain size in the submicron range. Only the mean of the coarse-grained alumina-998 did not compare well with results from other test methods. A combination of a high sensitivity to stable crack growth and a pop-in of small cracks to form a crack "initiation" region might be responsible for the discrepancy. The repeatability and reproducibility of the method are equal or better than those of other methods, e.g. SEPB and SCF. "Good" fracture toughness values can be measured for ceramics with an average grain size or major microstructural feature size of greater about 1 µm if the V-notch width is less than 10 µm. The V-notch depth had no influence on the measured fracture toughness over a wide range. Participants unfamiliar with the SEVNB method had in general no difficulty. The method proved to be forgiving and robust with respect to the notch preparation or optical notch quality. Participants rated the method user-friendly, easy, reliable and accurate. They are interested in a SEVNB standard.

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