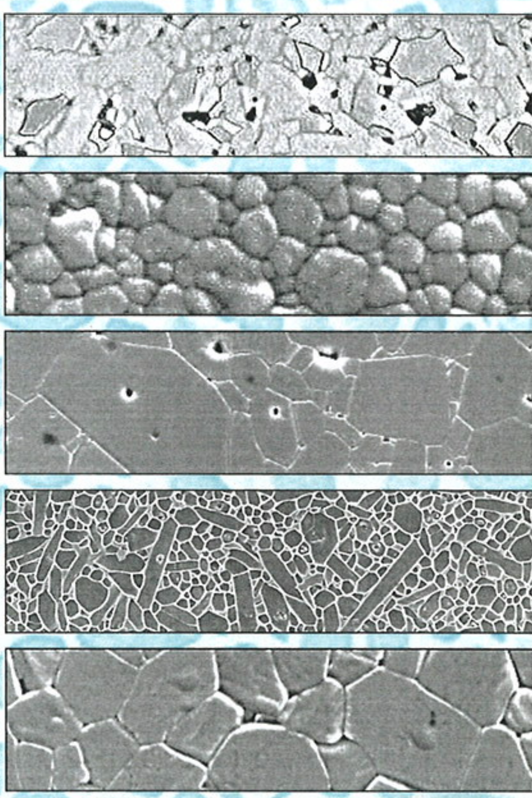
Fracture Toughness of Ceramics using the SEVNB Method; Round Robin



VAMAS Technical Working Area 3

ESIS Technical Ceramics (TC6)

VAMAS Report No. 37

ESIS Document D2-99

by

Jakob Kübler
EMPA, Swiss Federal Laboratories
for Materials Testing and Research
Dübendorf, Switzerland

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(Single-Edge-V-Notched Beam)

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VAMAS

The Versailles Project on Advanced Materials and Standards supports trade in high technology products through international collaborative projects aimed at providing the technical basis for drafting codes of practice and specifications for advanced materials. The scope of the collaboration embraces all agreed aspects of enabling science and technology – databases, test methods, design standards, and materials technology – which are required as a precursor to the drafting of standards for advanced materials. VAMAS activity emphasises collaboration on pre-standards measurement research, intercomparison of test results, and consolidation of existing views on priorities for standardisation action. Through this activity, VAMAS fosters the development of internationally acceptable standards for advanced materials by the various existing agencies.

ESIS

The objectives of the European Structural Integrity Society are to foster research into the prevention of failure of engineering materials, components and structures by fracture or other physical phenomena. These objectives include - among other items - the development and assessment of new testing methods, numerical methods and engineering methods for structural integrity assessment.

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Summary

Fracture toughness was measured by the Single-Edge-V-Notched Beam method on five monolithic advanced technical ceramics in an international round robin with more than 30 participants. These ceramics were coarse- and fine-grained alumina (alumina-998, alumina-999), gas pressure sintered silicon nitride (GPSSN), sintered silicon carbide (SSiC) and yttria-stabilised tetragonal zirconia polycrystal (Y-TZP), and had different degrees of difficulty in the application of this test method. Very consistent results were obtained for the alumina-998. The fracture toughness for the 135 tests accepted (= accepted by the participants and the round robin organiser as valid tests) from 28 participants was 3.57 ± 0.22 MPa √m (mean, standard deviation). Reasonably consistent results were obtained for the alumina-999. The fracture toughness for the 102 tests accepted from 21 participants was 3.74 ± 0.40 MPa √m. Consistent results were obtained for the GPSSN. The fracture toughness for the 129 tests accepted from 27 participants was 5.36 ± 0.34 MPa √m. Very consistent results were obtained for the SSiC. The fracture toughness for the 56 tests accepted from 12 participants was 2.61 ± 0.18 MPa √m. As predicted, less consistent results were obtained for the Y-TZP due to its grain size in the submicron range. The fracture toughness for the 35 tests accepted from 7 participants was 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, slow or stable crack growth near the V-notch tip 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 (< 10 µm), notch depth or optical notch quality for ceramics with an average grain size or major microstructural feature size of greater about 1 µm. Most participants had no difficulties and rated the method user-friendly, reliable and worthwhile for standardisation.

Key Words

Fracture toughness, test method, SEVNB, Single-Edge-V-Notched Beam, flexure, notch, razor blade, diamond paste, ceramics, alumina, silicon nitride, silicon carbide, zirconia,

Contents

0 Nomenclature

The following abbreviations will be used frequently in this report.

a	notch depth
Alumina-998	Material A, coarse grained alumina
Alumina-999	Material B, fine grained alumina
a_{mfs}	major microstructural feature size
ASTM	American Society for Testing and Materials
Avg.	average
CEN	European Committee for Standardisation
CMC	Ceramic Matrix Composite
CN	Chevron Notch
CV	Coefficient of Variance
DGK	German Ceramic Society
EN	Euro Norm
ESIS	European Structural Integrity Society
G.P.Avg.	grand population average
GPSSN	Material C, Gas-Pressure Sintered Silicon Nitride
G.P.Std.Dev.	grand population standard deviation
IF	Indentation Fracture
IS	Indentation Strength
JIS	Japanese Industrial Standard
K_{Ic}	fracture toughness
$K_{measured}$	measured fracture toughness
K_{XX}	fracture toughness measured with a specific method
ρ	notch root radius
S	notch width, V-notch width, or acceptable notch width
SCF	Surface Crack in Flexure
scg	subcritical crack growth
S_{crit}	critical notch width
SENB	Single-Edge-Notched Beam
SEPB	Single-Edge-Precracked Beam
SEVNB	Single-Edge-V-Notched Beam
SSiC	Material D, sintered silicon carbide
Std.Dev.	standard deviation
VAMAS	Versailles Project on Advanced Materials and Standards
W	specimen hight
Y	stress intensity shape factor
Y-TZP	Material E, post hipped Yttria-Stabilised Tetragonal Zirconia Polycrystal

1 Introduction

Many methods are currently used to measure the fracture toughness (K_{Ic}) of ceramic materials. Methods based on a widely accepted theory 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 demonstrated in a Versailles Project of Advanced Materials and Standards (VAMAS) round robin test report [L-1.1, L-1.2] that accurate fracture toughness values can be measured with the SCF method. However, they also showed that making the necessary 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 stated that the generation of a sharp crack could not be ensured in all tests [L-1.3]. Nishida, Hanaki and Pezzotti concluded that practical problems with the SEPB method make the fracture toughness determination difficult to apply and even unsuitable for some ceramics [L-1.4]. The simple and inexpensive SENB method, on the other hand, can be influenced by the notch width, as for example Primas and Gstrein discovered in a European Structural Integrity Society (ESIS) round robin [L-1.5, L-1.6]. In a more detailed analysis Damani, Gstrein and Danzer suggest that the stress field around the notch tip is responsible for the notch effect [L-1.7]. 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 [L-1.4] recently reintroduced an interesting technique described earlier by Le Bac [L-1.8] 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 in order to distinguish it from the SENB method, relates also to basic work conducted by Awaji and Sakaida [L-1.9].

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 [L-1.10]. The fracture toughness values measured with the SEVNB method on alumina, silicon carbide, silicon nitride and a composite compared well with values measured with the SEPB, SCF and CN tests. Values measured on a fine-grained zirconia were in the range of values from the SCF method, but significantly lower than values from the CN method. Further, the results exhibited only a small statistical spread.

After this promising preliminary study and the okay from the VAMAS and ESIS organisations to conduct a fracture toughness round robin jointly, a detailed instruction (Appendix A1: Instructions) was written and validated in a mini-round robin with two laboratories. Next, a questionnaire was sent to potential participants. The questionnaire contained questions like

- Are you experienced with the SEVNB method?
- How are you going to polish the V-notches necessary for the SEVNB method?
- Which fracture toughness test method are you going to use next to the SEVNB method?
- Name your material priorities for using the SEVNB method.
- Would you be willing to do additional studies on the ceramics?

Over 30 companies and institutes from Europe, USA, Japan, Australia and Brazil returned the questionnaire and were willing and able to participate in this exercise. Table 1.1 lists all laboratories that returned test results.

Table 1.1: Participating laboratories

AUSTRALIA	Dr. Mark Hoffman	University of New South Wales	Sidney
AUSTRIA	Mr. Rajiv Damani	Montanuniversität Leoben	Leoben
BELGIUM	Dr. Philippe Descamps	B.C.R.C.	Mons
BRASIL	Prof. Celio Albano Da Costa Neto	Universidade Federal do Rio de Janeiro (COPPE/UFRJ)	Rio de Janeiro
GERMANY	Dr. Kurt Keller	BASF Aktiengesellschaft	Ludwigshafen
	Mr. Volker Knoblauch	Robert Bosch GmbH	Stuttgart
	Dr. Meinhard Kuntz	Universität Bremen, Keramische Werkstoffe und Bauteile	Bremen
	Dr. H.A. Lindner	CFI GmbH & Co. KG	Rödental
	Mr. Gaston Rauchs	Universität Karlsruhe	Karlsruhe
	Dr. Thomas Reich	Fraunhofer-Gesellschaft – IKTS	Dresden
	Dr. Herbert Richter	CeramTech AG	Plochingen
	Prof. Jürgen Rödel	Technische Hochschule Darmstadt	Darmstadt
	Dr. Edith Rudolph	Bundesanstalt für Materialforschung und -prüfung (BAM)	Berlin
	Dr. Ralf Westerheide	Fraunhofer-Gesellschaft – IWM	Freiburg
ITALY	Dr. Martino Labanti	ENEA - CRNM (INN-NUMA-IMAP)	Faenza
	Dr. Goffredo De Portu	CNR – IRTEC	Faenza
JAPAN	Mr. Yasuo Nagano	Japan Fine Ceramics Center (JFCC)	Nagoya
	Mr. Shuji Sakaguchi	National Industrial Research Institute of Nagoya (NIRIN)	Nagoya
NETHERLANDS	Dr. Marc Steen	Joint Research Centre, EC	ZG Petten
RUSSIA	Prof. Sergei Barinov	Russian Academy of Sciences (HTC)	Moscow
SLOVAKIA	Ass. Prof. Ján Dusza	Slovak Academy of Siences (IMR)	Kosice
SPAIN	Prof. Marc J. Anglada	Universitat Politècnica de Catalunya (ETSEIB)	Barcelona
	Prof. José Y. Pastor	Universidad Politecnica de Madrid	Madrid
SWITZERLAND	Mr. Jakob Kübler	Swiss Federal Laboratories for Materials Testing and Research (EMPA)	Dübendorf
UK	Dr. Roger Morrell	National Physical Laboratory (NPL)	Teddington
	Dr. Michael Reece	Queen Mary and Westfield College	London
UKRAINE	Prof. George A. Gogotsi	National Academy of Sciences of Ukraine (IPS)	Kiev
USA	Dr. Kristin Breder	Oak Ridge National Laboratory (ORNL)	Oak Ridge
	Dr. Sung R. Choi	National Aeronautics and Space Administration (NASA)	Cleveland
	Dr. Frank Meschke	Lehigh University (MRC)	Bethlehem
	Mr. George Quinn	National Institute of Standards and Technology (NIST)	Gaithersburg

2 Material

In the round robin, five ceramic materials with varying difficulties for measuring the fracture toughness were used. Each participant was required to test the alumina-998 (Material A) and the gas pressure sintered silicon nitride (Material C: GPSSN). The alumina-999 (Material B), the sintered silicon carbide (Material D: SSiC) and the yttria-stabilised tetragonal zirconia polycrystal (Material E: Y-TZP) were optional. Table 2.1 shows the processing parameters and main properties of the materials as far as they are known. The microstructures are shown in Figure 2.1 to 2.5 together with additional information.

Table 2.1: Processing parameters and material properties

	Material				
	Alumina-998	**Alumina-999**	**GPSSN**	**SSiC**	**Y-TZP**
Code	**A**	**B**	**C**	**D**	**E**
Powder purity / type	> 99.8 %	> 99.9 %	N 3208		TOSOH TZ-3Y (3 mole-% yttria-stabilised tetragonal zirconia polycrystal)
Processing	Metoxit Switzerland	Metoxit Switzerland	Bayer-CFI Germany	Hutschen-reuther Germany	Metoxit Switzerland
cold pressed	isostatic	isostatic			isostatic
hot pressed				unknown parameters	
sintered	oxidising atmosphere	oxidising atmosphere	gas-pressure		oxidising atmosphere
post hipped		gas-pressure			gas-pressure
form & size	bar 4x5x45 mm^3	rod Ø 40 mm length 50 mm	plate 53x47x5 mm^3		rod Ø 50 mm length 200 mm
Avg. grain size	>10 µm	~1.7 µm	< 1 µm with elongated grains	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)] in 4-point bend testing at room temperature

2.1 Alumina-998

The coarse grained alumina–998 is a "general purpose" ceramic produced and used for many years in large amounts by industry for plungers, tubes, plates, insulators, etc.. In 1994 over 500 bend bars with a size of 3x4x45 mm^3 were machined by grinding in accordance with EN 843-1 [L-2.1] from bars with a size of 4x5x45 mm^3. Not all bend bars distributed to the participants were chamfered but with respect to the fracture toughness test method used this should not matter.

About half of the original 500 bend bars were used in a round robin conducted by the German Ceramic Society (DGK) and the rest in the present VAMAS / ESIS round robin. In the DGK round robin a characteristic strength of 342 MPa together with a Weibull modulus of 21, a fracture toughness of 3.64 MPa $\sqrt{m}$ (SCF method) and a subcritical crack growth (scg) parameter n = 44 (static load, water, 20 °C) was measured for the alumina-998. *(Remark: The objective of the DGK round robin was to compare scg parameters measured with different test methods. The results are not published as yet, but an analysis of the alumina-998 results càn be found in EMPA-Report No. 150'960 [L-2.2] or in the proceedings of the Fractography of Glasses and Ceramics III Conference, 1995 [L-2.3]).*

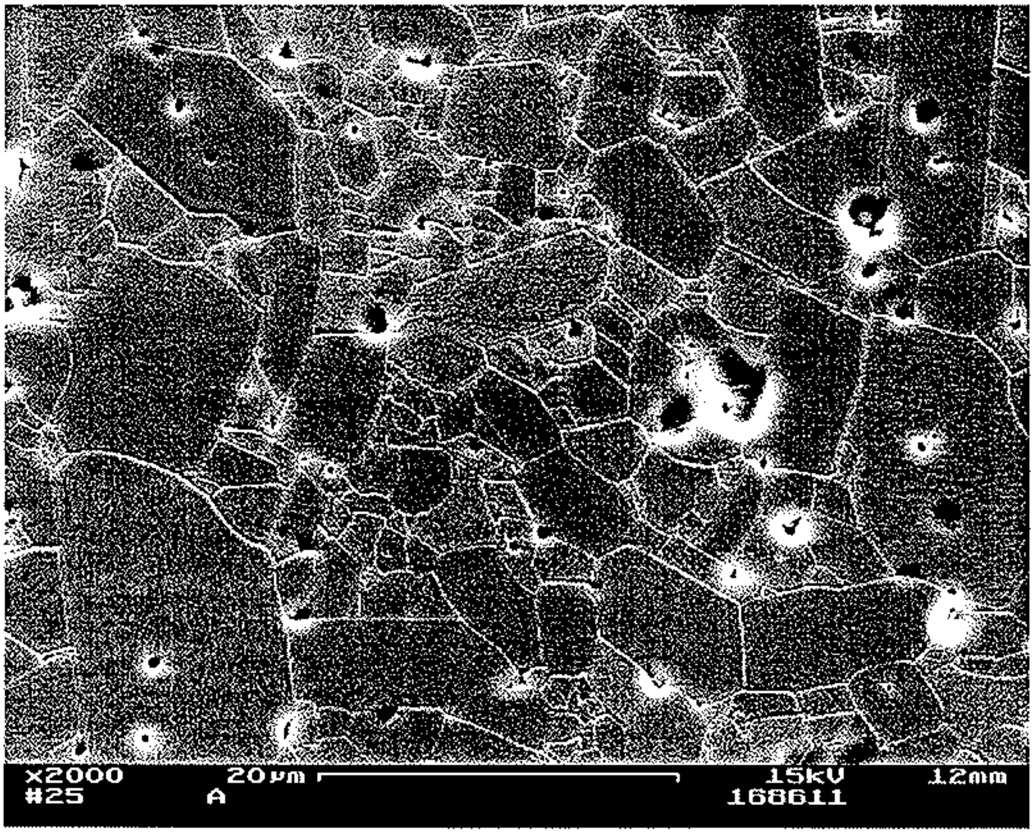


Figure 2.1 (98040047.jpg)
Microstructure of the cold pressed alumina–998 (Material A).

2.2 Alumina-999

The fine-grained alumina–999 has been used for many years by industry for bioceramics, precision parts and spheres. Over 200 bend bars with a size of 3x4x45 mm^3 were machined by grinding in accordance with EN 843-1 [L-2.1] from 10 sintered and post hipped rods with a diameter of 40 mm and a length of 46 mm. All specimens were cut with their long axis parallel to the rod axis.

Earlier, the round robin organiser measured on an identical alumina (= identical raw material, processing and bend bar machining) a characteristic strength of 473 MPa together with a Weibull modulus of 8.2, a Young's modulus of 398 GPa and a fracture toughness of 3.13 MPa √m (IF method). Fett and co-workers measured on an almost identical alumina (= identical raw material; but: sintering temperature 1550 °C, post hipping pressure 1000 bar and grain size of 3.25 µm) a scg crack growth parameter n = 73.5 (4-point bending, static load, salt solution, 70 °C) and a fracture toughness of 3.8 MPa √m with the SCF method [L-2.4]. Further, it was reported that 400 ppm MgO was used as a grain refiner.

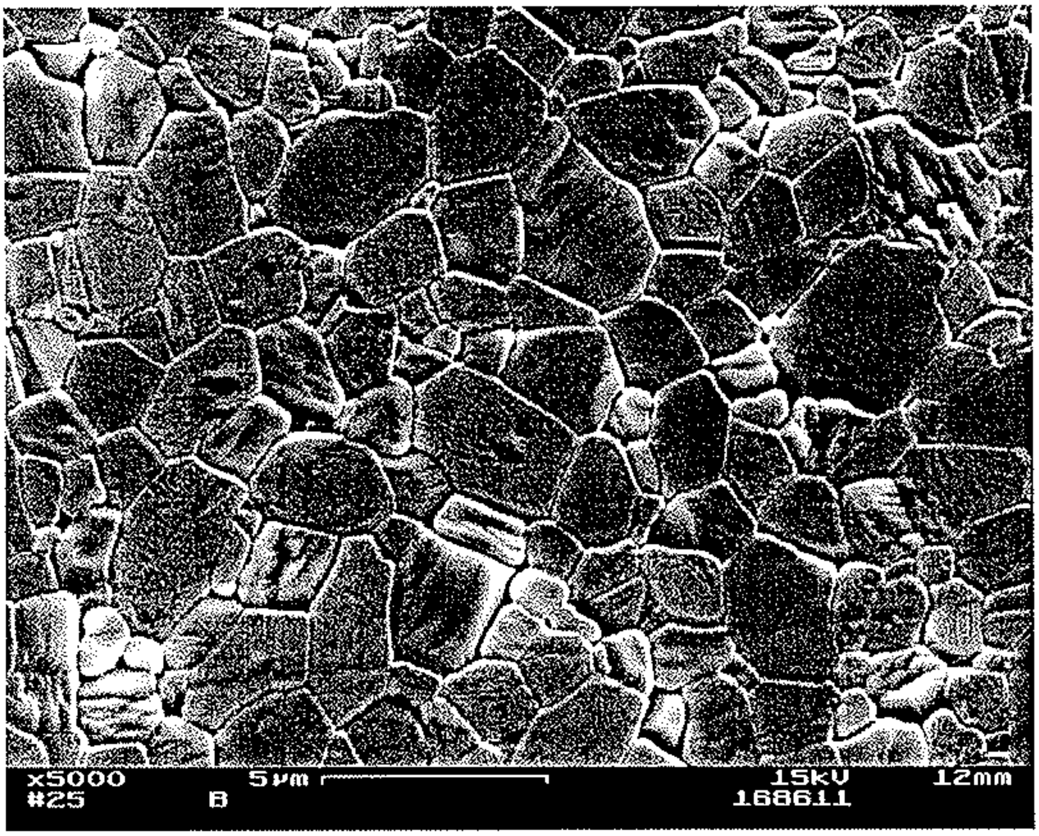


Figure 2.2 (98040050.jpg)
Microstructure of the post hipped alumina-999 (Material B).

2.3 Silicon Nitride

The silicon nitride is used by industry for valves, etc. Almost 300 bend bars with a size of 3x4x45 mm^3 were machined by grinding in accordance with EN 843-1 [L-2.1] from 30 gas-pressure sintered plates with a size of 53x47x5 mm^3.

The manufacturer of the plates reports for the material a 4-point bending strength of 920 MPa with a Weibull modulus >20 at room temperature, a fracture toughness of 6.5 MPa √m (IF method with Niihara equation) and a Young's modulus of 320 GPa.

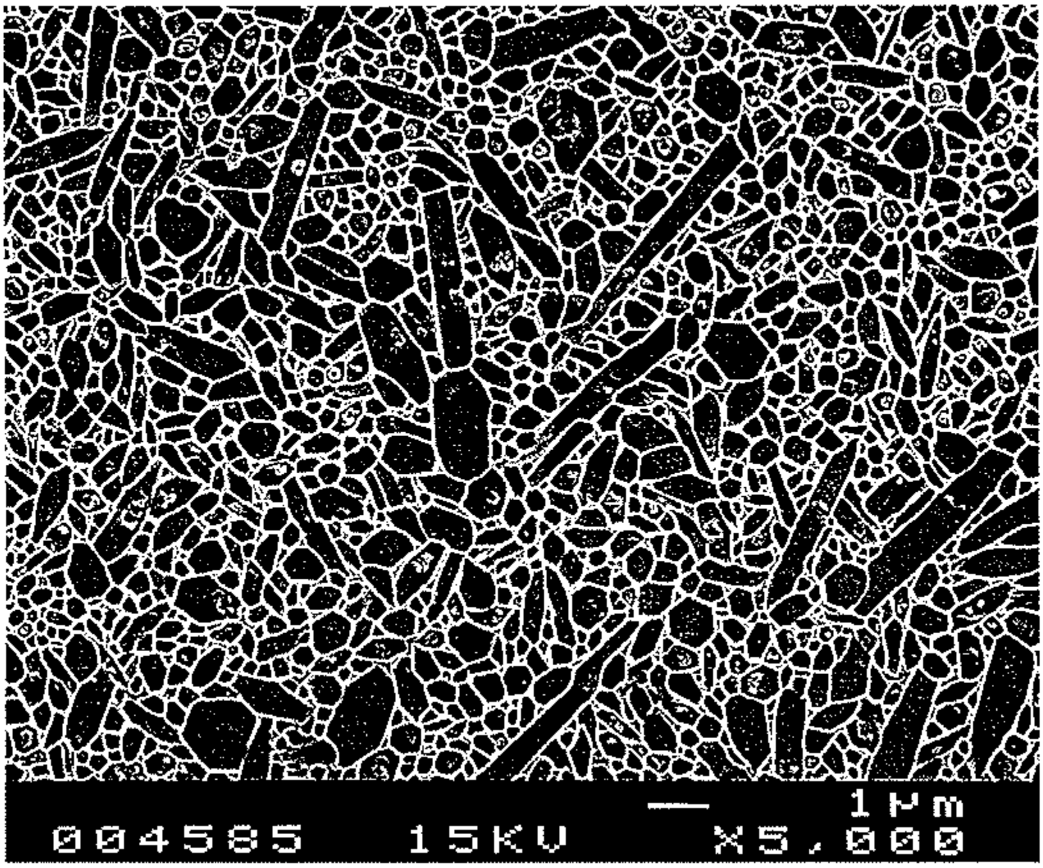


Figure 2.3 (Sn-cfi.tif)
Microstructure of the gas-pressure sintered silicon nitride (Material C).
(Photo courtesy of Bayer-CFI, Germany)

2.4 Sintered Silicon Carbide

The SSiC was previously used in an ESIS TC6 fracture toughness round robin. Primas et al. [L-1.5, L-1.6] report that the microstructure of the SSiC was characterised by some large plate-like grains and neighbouring regions with a high density of pores. The average grain size was 7 µm with grains up to 50 µm. Further, a Young's modulus of 427 GPa measured with the resonance frequency method is reported. The results measured with the five fracture toughness test methods used in the ESIS TC6 round robin are summarised in Table 2.2. The raw material data, processing and machining parameters are unknown. About 70 machined bend bars with a size of 3x4x45 mm^3 were not tested and could therefore be used in this round robin.

Table 2.2: Fracture toughness values measured in the ESIS TC6 round robin.

Test Method	G.P.Avg. [MPa √m]	G.P.Std.Dev. [MPa √m]	Number of Tests [-]	Number of Laboratories [-]
CVN 1)	2.72	0.29	17	2
IF	2.13	0.29	111	4
IS	3.13	0.41	18	2
SENB-B2)	2.09	0.45	13	2
SENB-S3)	3.52	0.46	24	2

1) All tests invalid since no stable crack growth has been observed.
2) Notch (= crack) produced with the bridge technique (= SEPB method).
3) Notch cut with a diamond saw.

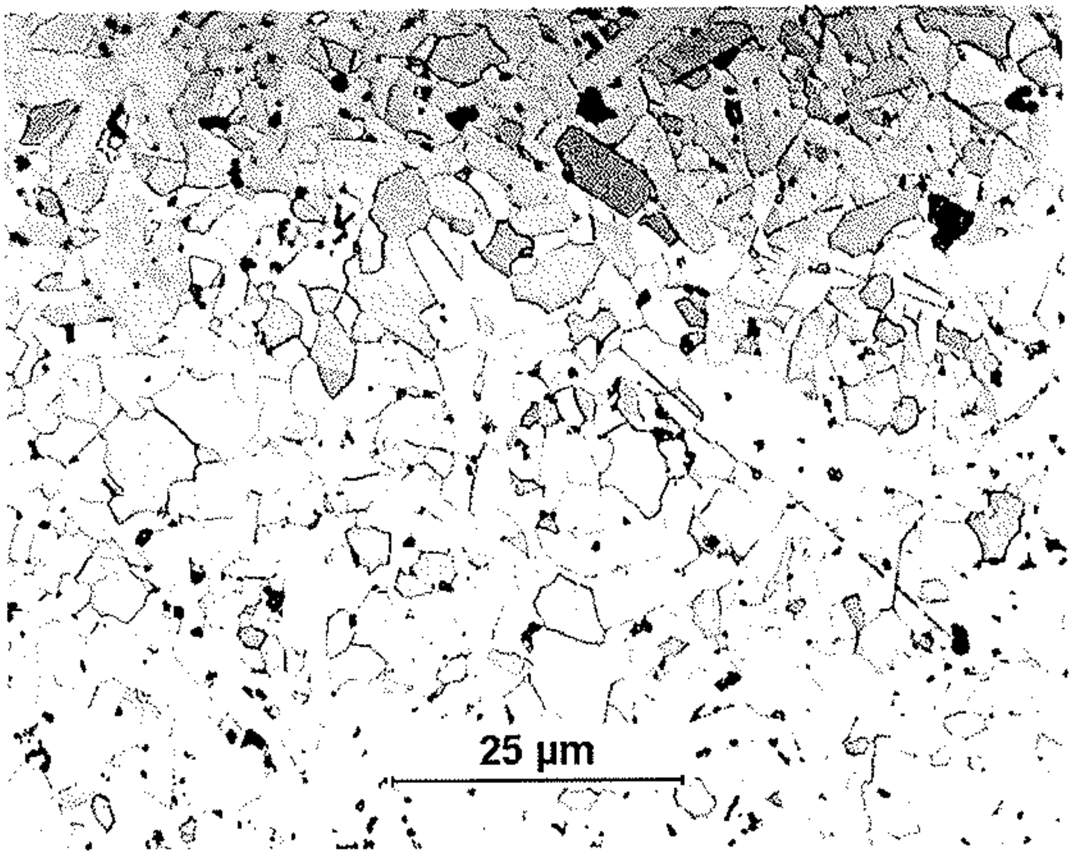


Figure 2.4 (98050682.jpg)
Microstructure of the sintered silicon carbide (Material D: SSiC).

2.5 Yttria-Stabilised Tetragonal Zirconia Polycrystal

The very fine grained, yttria-stabilised ceramic is used by industry for example for watch casings, precision parts and bioceramics. The material was fabricated in the form of two rods with a diameter of 50 mm and a length of 200 mm. Afterwards they were cut into pieces with a length of 50 mm. From those rods about 500 bend bars with a size of 3x4x45 mm^3 were machined by grinding in accordance with EN 843-1 [L-2.1]. All specimens were cut with their long axis parallel to the rod axis.

In an earlier study using some of the 500 bend bars the round robin organiser measured for the Y-TZP a characteristic strength of 774 MPa together with a Weibull modulus of 14 and a Young's modulus of 211 GPa. Further, 240 bend bars were used in a VAMAS fracture toughness round robin [L-1.1]. With the SCF method used, a fracture toughness of 4.4 ± 0.4 MPa √m was determined. The round robin revealed further that the material had sintering defects with sizes of up to 20 µm and that the ground surfaces of the bend bars showed mostly a tetragonal and/or cubic phase. About 50 bend bars were left and could be used in the present round robin. Additional mechanical properties of this material, but from an earlier produced batch were presented at the American Ceramic Society Annual Meeting, Minneapolis, MN, USA, April 1992 [L-2.5].

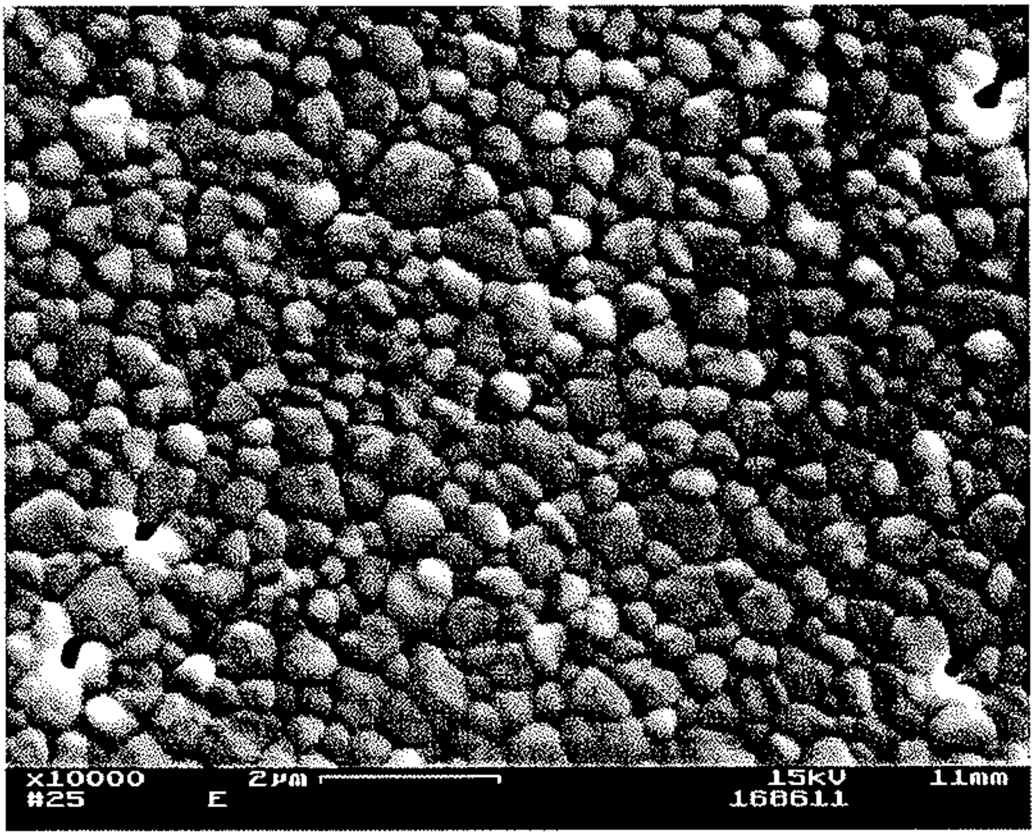


Figure 2.5 (98040061.jpg)
Microstructure of the post hipped yttria-stabilised tetragonal zirconia polycrystal (Material E: Y-TZP).

3 Experimental Procedure

Each participant received a package containing five 3x4x45 mm alumina-998 and GPSSN bend bars prepared in accordance with standard EN 843-1 [L-2.1] on which the fracture toughness with the SEVNB method had to be measured, and instructions detailing how to conduct the round robin. Only participants who asked for (in the questionnaire) received additional specimens from material B, D or E (alumina-999, SSiC or Y-TZP). Further, some participants received additional five specimens of materials A, B, or C on which they were asked to measure the fracture toughness using their preferred method (SCF, SEPB, CN, etc.).

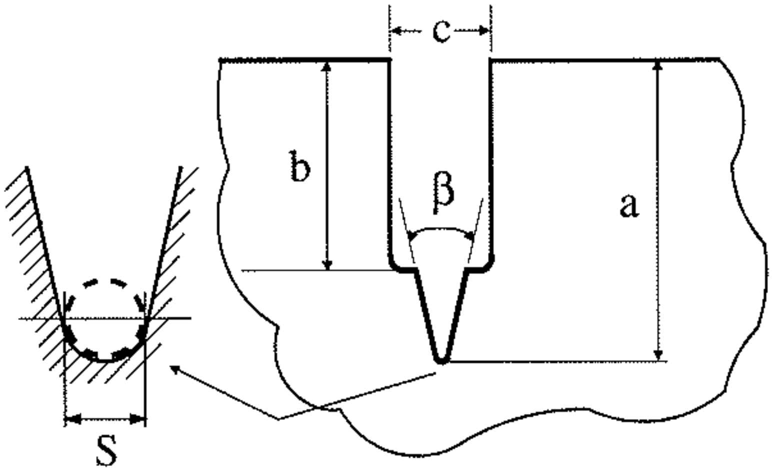


Figure 3.1
Schematic geometry of V-notch
a = 0.8 mm ... 1.2 mm
b = 0.5 mm ... 0.6 mm
c ≥ width of razor blade
a-b ≥ c
β ~ 30°, or as small as possible
S ≈ V-notch width

The fracture toughness with the SEVNB method was to be measured in 4-point bend 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 3.1. The V-notches could be produced either by hand (Figure 3.2) or by machining (Figure 3.3). The general procedure could be performed in three steps:

1) Mount five specimens parallel and side by side with their later 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.

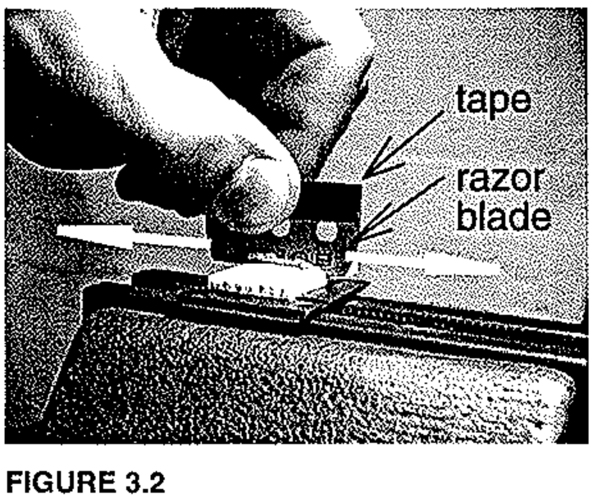


FIGURE 3.2
V-notch polishing by hand

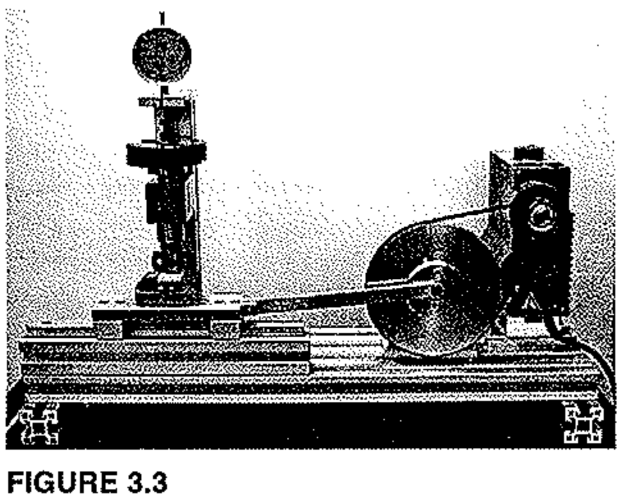


FIGURE 3.3
Example of a machine to polish the V-notches

Finally, the participants had to compute the fracture toughness K_{Ic} using the following formula [L-3.1, L-3.2]:

$$K_{Ic} = \frac{F_C}{B\sqrt{W}} \cdot \frac{S_1 - S_2}{W} \cdot \frac{3\sqrt{\alpha}}{2(1-\alpha)^{1.5}} Y^* \qquad \text{(F 3.1)}$$

with: $$Y^* = 1.9887 - 1.326\alpha - (3.49 - 0.68\alpha + 1.35\alpha^2)\alpha(1-\alpha)(1+\alpha)^{-2} \qquad \text{(F 3.2)}$$

where:

F_C	fracture load
S_X	span *(x=1 : outer span ; x=2 : inner span)*
B	specimen width
W	specimen height
a	notch depth
α	a/W
Y^*	stress intensity shape factor

The package sent to each participant, the shape and dimensions of the specimens, the preparation of the specimens by hand or machine, the test machine and procedure, the analysis of the test results, and how to report the results is described in great detail in Appendix A1: Instructions.

4 Results

4.1 General

All accepted fracture toughness values together with the V-notch width from the individual participants are listed in Appendix A2: Individual Fracture Toughness Results. Further, all results reported by the participants are summarised in Table 4.1.1. Comments to the SEVNB method and the round robin reported by the participants are listed in Appendix A4: Comments by participants to the SEVNB method.

Five of the original 36 participants quit the round robin due to the following reasons:
- change in field of work (participant #13)
- test machine failure (participant #18)
- necessary modifications on equipment not possible (participant #20)
- unknown (participant #27)
- lack of manpower (participant #33)

All participants were instructed to furnish photos showing the V-notch tip from at least two specimens per material tested. In the Appendix A3: Notch and Notch Tip Geometry a typical notch and V-notch tip from each participant and tested material is shown.

Appendix A2: Individual Fracture Toughness Results, shows also the results that were declared as "not valid" by the participants themselves and those "not accepted" by the round robin organiser. Only in one case a single result of a participant was "not accepted" (participant #7, GPSSN). In all other cases, the whole data set was either included or not. For each material a grand population average (G.P.Avg.) fracture toughness together with a grand population standard deviation (G.P.Std.Dev.) was computed using all accepted results. For a better visualisation of the results, frequency histograms for all five materials are included in Appendix A2.

Participant #22 measured the notch depth for each test on both specimen halves and computed always two notch depths, two fracture toughness values, etc. for each specimen. For the tables and figures the organiser averaged all values.

Participants #6, #8 and #11 reported that they used rollers with diameters of 4.5 mm, 4.25 mm, and 3 mm, respectively, with the 40 / 20 mm 4-point bend test jigs instead of the required 5 mm ones. Participant #14 mentioned that he used a 3-point test jig with a roller span of 30 mm. Participant # 25 used a 4-point bend test jig with spans of 30 / 10 mm and rollers set into grooves and thus not free to roll. Finally, participant #35 used a test jig as asked for in the instructions except that two from the four loading rollers were set into grooves and therefore not free to roll. No results were discarded because of the roller size, the test jig dimensions or fixed rollers.

The organiser asked some participants to check for suspicious results. If the check showed a typographical error, a miscalculation of the results, a mix up of results or other "minor" problems, the result was corrected without further notification. In two cases, the organiser corrected the reported notch width in accordance with the furnished photos (participant #21, GPSSN and participant #28, alumina-999).

Considering the large number of participants, the amount of specimens was rather meagre. Therefore, the alumina-998, SSiC, and Y-TZP results of the round robin organiser (participant #1) and also the alumina-998 result of participant #6 were taken from the previous mini-round robin.

Table 4.1.1.: Fracture toughness results for the round robin. Each block shows a participant's mean result, the standard deviation, both in (MPa √m) and the number of specimens tested (in parentheses). Values marked with an asterisk * were not used to compute the average and deviation (reasons see paragraphs 4.2 to 4.4).

Participant #	Alumina-998	Alumina-999	GPSSN	SSiC	Y-TZP
1	3.67 ± 0.07 (5)	3.54 ± 0.17 (5)	5.23 ± 0.21 (5)	2.62 ± 0.10 (5)	4.69 ± 0.09 (5)
2	3.61 ± 0.08 (5)	4.09 ± 0.69 (5) *	5.49 ± 0.20 (5)	-	-
3	4.27 ± 0.14 (5) *	5.38 ± 0.27 (5) *	7.38 ± 0.11 (5) *	-	-
4	3.45 ± 0.16 (5)	3.51 ± 0.21 (5)	5.48 ± 0.17 (5)	2.67 ± 0.14 (5)	5.03 ± 0.10 (5)
5	3.42 ± 0.16 (5)	3.55 ± 0.14 (5)	5.11 ± 0.24 (5)	2.41 ± 0.02 (5)	-
6	3.68 ± 0.19 (5)	3.47 ± 0.21 (5)	4.92 ± 0.23 (5)	2.49 ± 0.08 (5)	5.27 ± 0.23 (5)
7	3.54 ± 0.07 (4)	3.55 ± 0.17 (5) *	5.01 ± 0.27 (3)	2.26 ± 0.15 (4)	-
8	3.41 ± 0.07 (4)	4.13 ± 0.41 (5)	5.43 ± 0.59 (5)	-	-
9	4.28 ± 0.11 (5) *	3.59 ± 0.24 (4) *	5.98 ± 0.47 (4) *	-	-
10	3.54 ± 0.09 (5)	3.45 ± 0.20 (5)	5.27 ± 0.33 (5)	-	-
11	3.62 ± 0.04 (5)	3.41 ± 0.03 (5)	5.43 ± 0.35 (5)	-	6.04 ± 0.27 (5)
12	3.68 ± 0.09 (4)	3.97 ± 0.40 (2) *	5.32 ± 0.34 (5)	-	5.65 ± 0.22 (5)
14	3.35 ± 0.13 (5)	-	-	-	-
15	3.79 ± 0.07 (5)	4.51 ± 0.30 (5)	5.54 ± 0.13 (5)	2.66 ± 0.14 (5)	4.60 ± 0.28 (5)
16	3.61 ± 0.07 (5)	3.56 ± 0.21 (5)	5.17 ± 0.34 (5)	2.68 ± 0.07 (5)	-
17	3.61 ± 0.07 (5)	3.97 ± 0.19 (4)	5.41 ± 0.17 (2)	2.70 ± 0.26 (3)	-
19	3.49 ± 0.15 (5)	3.46 ± 0.20 (5)	5.33 ± 0.23 (5)	-	-
21	3.42 ± 0.22 (5)	3.58 ± 0.08 (5)	5.44 ± 0.32 (5)	-	-
22	3.66 ± 0.03 (5)	3.62 ± 0.40 (5)	5.41 ± 0.44 (5)	2.75 ± 0.05 (5)	6.11 ± 0.70 (5)
23	3.80 ± 0.14 (5)	3.90 ± 0.22 (5)	5.35 ± 0.19 (5)	-	-
24	3.64 ± 0.06 (5)	4.10 ± 0.22 (5)	5.55 ± 0.11 (5)	2.74 ± 0.05 (5)	-
25	3.68 ± 0.03 (5)	3.78 ± 0.28 (5)	5.92 ± 0.16 (5)	-	-
26	3.68 ± 0.07 (4)	2.17 ± 0.12 (2) *	5.25 ± 0.18 (4)	2.70 ± 0.08 (5)	-
28	3.64 ± 0.06 (5)	3.99 ± 0.29 (5)	5.49 ± 0.37 (5)	-	-
29	3.64 ± 0.10 (4) *	4.94 ± 0.18 (4) *	6.65 ± 0.54 (5) *	-	-
30	3.60 ± 0.06 (5)	3.96 ± 0.18 (5)	5.22 ± 0.17 (5)	2.66 ± 0.20 (4)	-
31	3.00 ± 0.65 (5)	2.90 ± 0.17 (3)	5.17 ± 0.19 (5)	-	-
32	3.56 ± 0.12 (5)	3.65 ± 0.14 (5)	5.06 ± 0.30 (5)	-	-
34	3.65 ± 0.06 (4)	4.26 ± 0.18 (5)	5.84 ± 0.25 (5)	-	-
35	3.67 ± 0.18 (5)	-	5.49 ± 0.27 (5)	-	-
36	3.63 ± 0.08 (5)	-	5.22 ± 0.26 (5)	-	-
G.P.Avg.	**3.57 ± 0.22** (135)	**3.74 ± 0.40** (102)	**5.36 ± 0.34** (129)	**2.61 ± 0.18** (56)	**5.34 ± 0.65** (35)

4.2 Alumina-998

Figure 4.2.1 and Table 4.1.1 show the results for the alumina-998, which had a G.P.Avg. of 3.57 MPa √m and a G.P.Std.Dev. of 0.22 MPa √m. The G.P.Avg. and G.P.Std.Dev. are based on the results of all 135 tests accepted. A histogram of all accepted test results in Appendix A2 shows a bell shaped distribution.

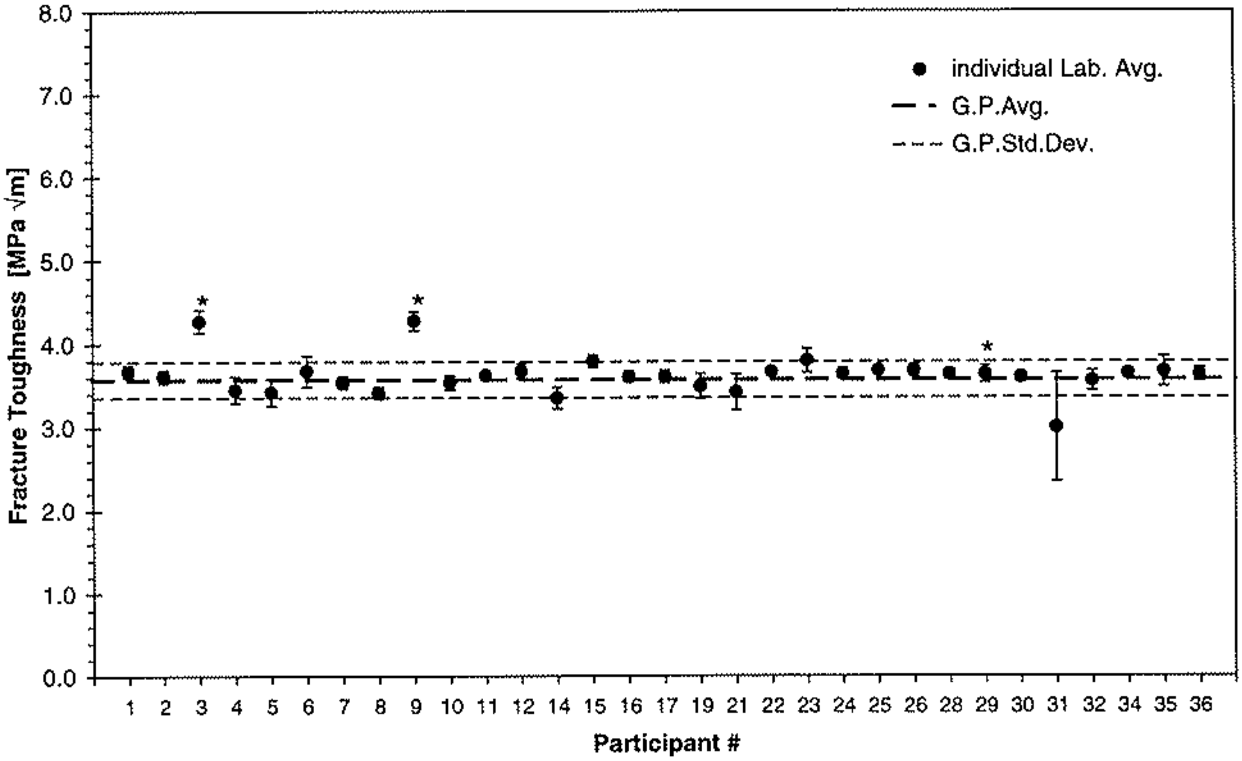


Figure 4.2.1
Master result graph for the alumina-998 showing the individual participant's (Lab.) average with standard deviation. The broad dashed line represents the grand population average (G.P.Avg.) and the narrow outer ones, the grand population standard deviation (G.P.Std.Dev.). Results marked with a * were not used to compute the G.P.Avg. and G.P.Std.Dev.

A notch width of up to at least 30 µm has no influence, as Figure 4.2.2 shows. 28 of the 31 (90 %) participants were able to polish notches with a width ≤ 30 µm. *(Remark: The notch widths used in Figure 4.2.2. are the mean from all notch widths reported by a single participant for a material.)* Because of the wide V-notches, the results from participants #3, #9 and #29 were not accepted (see below and Appendix A3). None of the results from participants #10, #17, #21, #24, #26 and #31 were discarded, even though some or all of their notch depths were outside the limits given in the instructions as $0.2 < a/W < 0.3$.

23 participants reported 5 valid results each (100 %) and 5 reported 4 each (80 %). Based on a total of 140 specimens tested by the 28 participants the overall success rate was 96 %. From the participants reporting 4 successful tests, #7 damaged one specimen during set up and #12 had one where the crack did not start from the V-notch tip.

Participant #31 reported a very low average fracture toughness with a large standard deviation as Figure 4.2.1 shows (the mean fracture toughness is outside the G.P.Avg. scatter bands). A closer look (Appendix A2) revealed that two toughness values were very low (2.2 MPa √m and 2.4 MPa √m).

Also the average fracture toughness of participant #23 was outside the G.P.Avg. scatter bands but only by 0.01 MPa √m. Appendix A2 and A3 reveal that the V-notch geometry was outside the tolerances given in the instructions (see Appendix A1 and Figure 3.1).

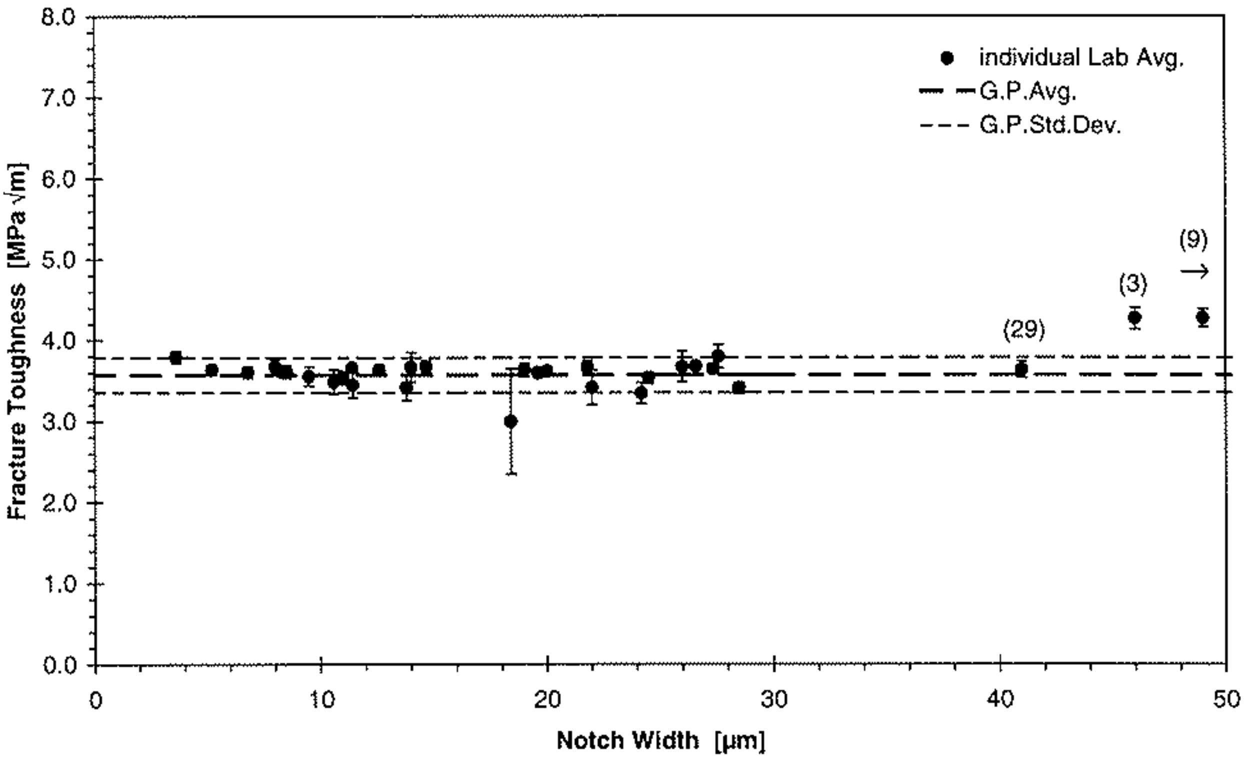


Figure 4.2.2
Notch width dependence of the fracture toughness of the alumina-998. Results not accepted are marked with the participant's code number in parentheses. The result marked with an → represents a V-notch width of 172 µm (!)

The G.P.Avg. was within the scatter bands (mean plus or minus one Std.Dev.) of 16 of the 28 (57 %) participants. On the other hand, the mean of 26 of 28 (93 %) participants was within the G.P.Avg. scatter bands.

Another way of evaluating the consistency of the participants' results is to apply the central limit theorem for the variation of sample means about the G.P.Avg. The sample mean should be distributed about the G.P.Avg. with G.P.Std.Dev.$_x$ / √n (n = # specimens). Since the participants had different numbers of valid tests, their possible deviation from the G.P.Avg. will vary. 18 of the possible 28 (64 %) participants' averages fell within one G.P.Std.Dev.$_x$ / √n, which is very close to the expected 68 % for the normal distribution. 24 of the 28 (86 %) participants' averages' fell within two G.P.Sdt.Dev.$_x$ / √n, which is somewhat less than the expected 95 %.

4.3 Alumina-999

Figure 4.3.1 and Table 4.1.1 show the results for the fine-grained alumina-999, which had a G.P.Avg. of 3.74 MPa √m and a G.P.Std.Dev. of 0.40 MPa √m which is almost twice the deviation computed for the coarser grained alumina (alumina-998). The G.P.Avg. and G.P.Std.Dev are based on the results of all 102 tests accepted. The histogram of all accepted results in Appendix A2 shows a broad and slightly distorted distribution.

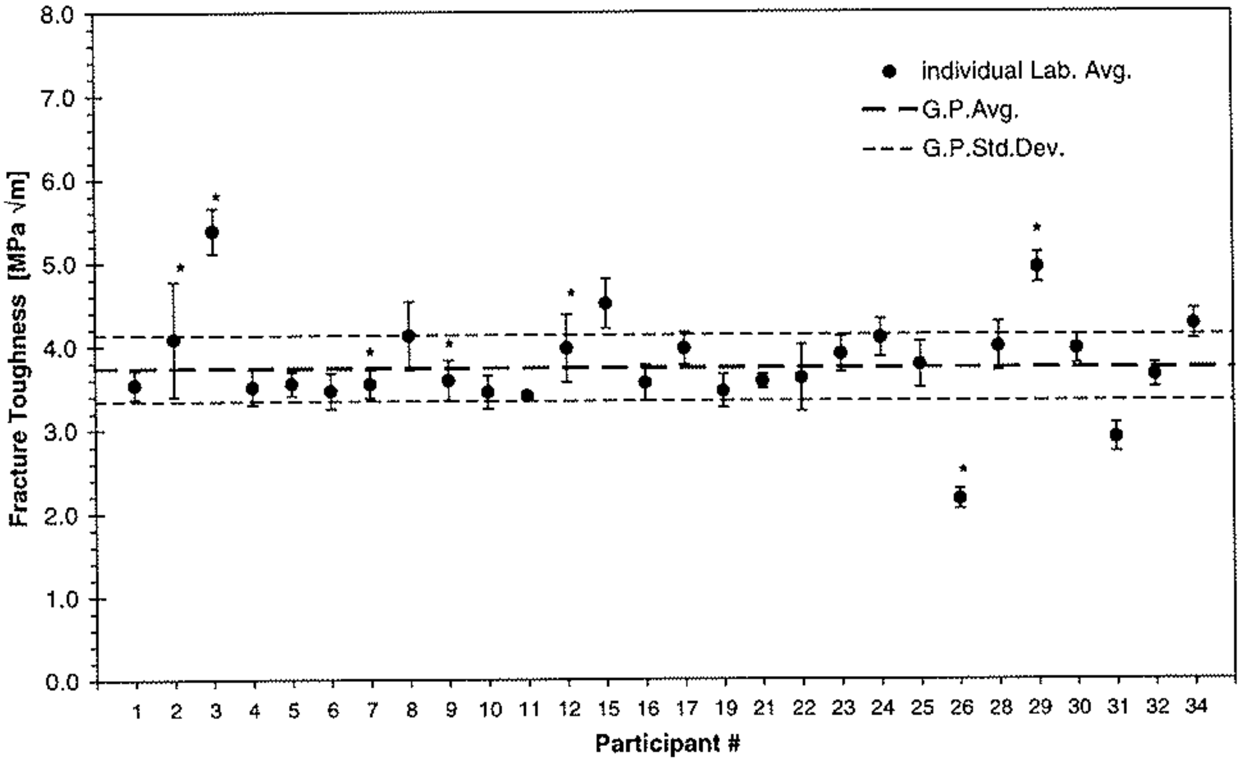


Figure 4.3.1
Master result graph for the alumina-99 showing the individual participants' (Lab.) average with standard deviation. The broad dashed line represents the grand population average (G.P.Avg.) and the narrow outer ones, the grand population standard deviation (G.P.Std.Dev.). Results marked with a * were not used to compute the G.P.Avg. and G.P.Std.Dev.

Participant #26 reported only two and also very low fracture toughness values (2.08 MPa √m and 2.25 MPa √m). His other three specimens failed during handling. The participant was asked to analyse the fractured surfaces fractographically. On both specimens he found large precracks, see Figure 4.3.3. Because of the precracks the values were discarded. Participant #31 also reported a rather low fracture toughness and that two specimens failed during handling. But he could not find any signs of precracking or other damages responsible for the low values measured. Therefore, the results of participant #31 were <u>not</u> discarded.

Up to a notch width of about 30 µm no significant influence of the notch width is visible in Figure 4.3.2. Twenty-one of the 27 (78 %) participants and therefore less than on the coarser grained alumina–998), were able to polish notches with a width ≤ 30 µm. Because of the wide V-notches the results from participant #2, #3, #7, #9, #12 and #29 were not accepted (see below and Appendix A3). On the other hand, none of the results from participants #17, #24, #28 and #31 were discarded, even though the reported notch depths were outside the limits given in the instructions.

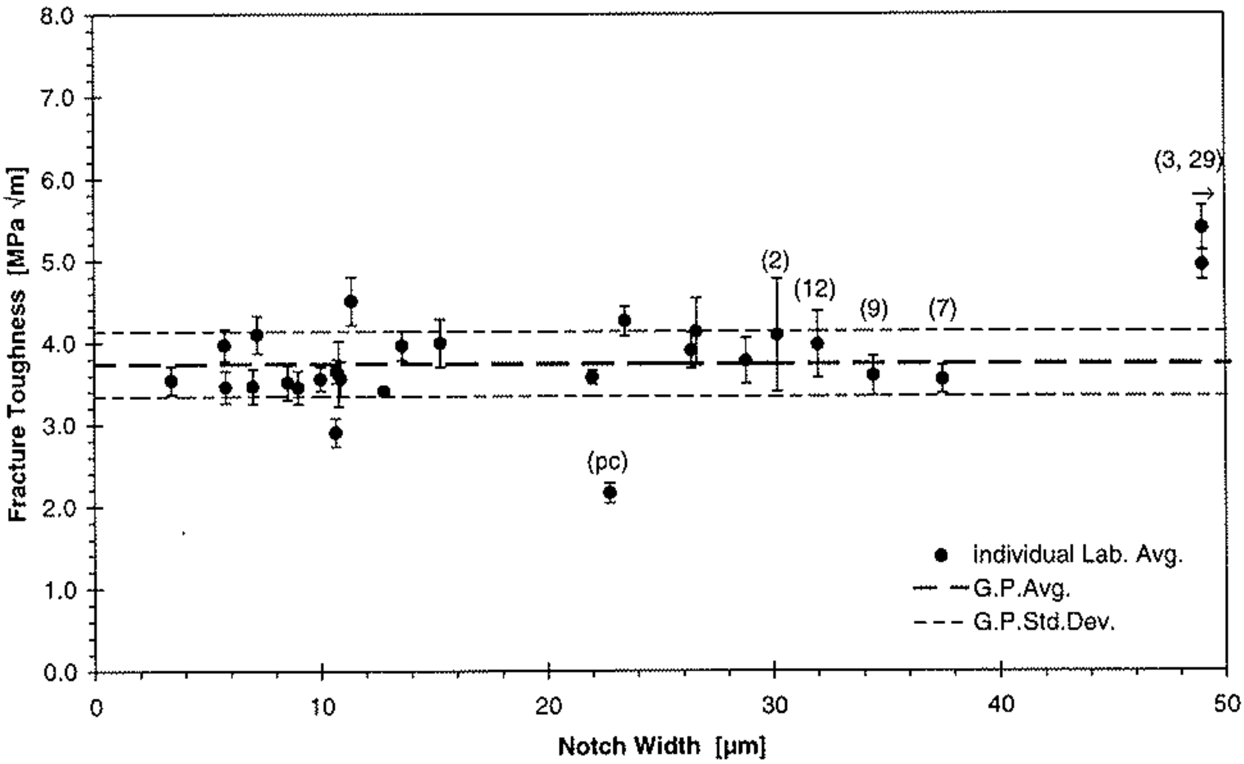


Figure 4.3.2
Notch width dependence of the fracture toughness of the alumina-999. Results not accepted are marked with the participant's code number in parentheses. The results marked with an → represents a V-notch width of 50 µm and 52 µm, respectively. The result marked with (pc) is from participant #26 whose specimens showed large precracks.

19 participants reported 5 valid results each (100 %), one 4 (80 %) and one 3 (60 %). Based on a total of 105 specimens tested by the 21 participants the overall success rate was 97 %. All participants not reporting a 100 % success rate damaged their specimens during preparation.

The G.P.Avg. was within the scatter bands of only 7 of the 21 (33 %) participants and the mean of 18 of the 21 (86 %) participants were within the G.P.Avg. scatter bands.

The central limit theorem for evaluating the consistency of the participants' results was applied on the alumina-999 too. Only 6 of the possible 21 (29 %) participants' averages fell within one G.P.Std.Dev.$_x$ / √n (expected 68 %) and 17 of the 21 (81 %) participants' averages fell within two G.P.Std.Dev.$_x$ / √n (expected 95 %).

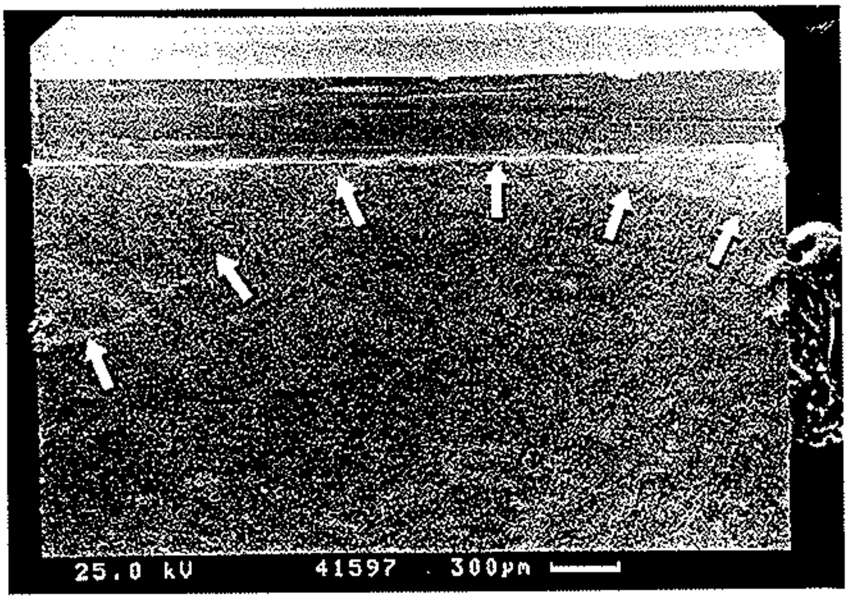


Figure 4.3.3
Fracture surface of the alumina-999 specimen which had a fracture toughness of 2.08 MPa √m and was tested by participant #26. The arrows mark the large precrack.

4.4 Silicon Nitride

Figure 4.4.1 and Table 4.1.1 show the results for the gas pressure sintered silicon nitride, which had a G.P.Avg. of 5.36 MPa √m and a G.P.Std.Dev. of 0.34 MPa √m. The G.P.Avg. and G.P.Std.Dev are based on the results of all 129 tests accepted. The histogram of all accepted results in Appendix A2 shows a well-formed distribution.

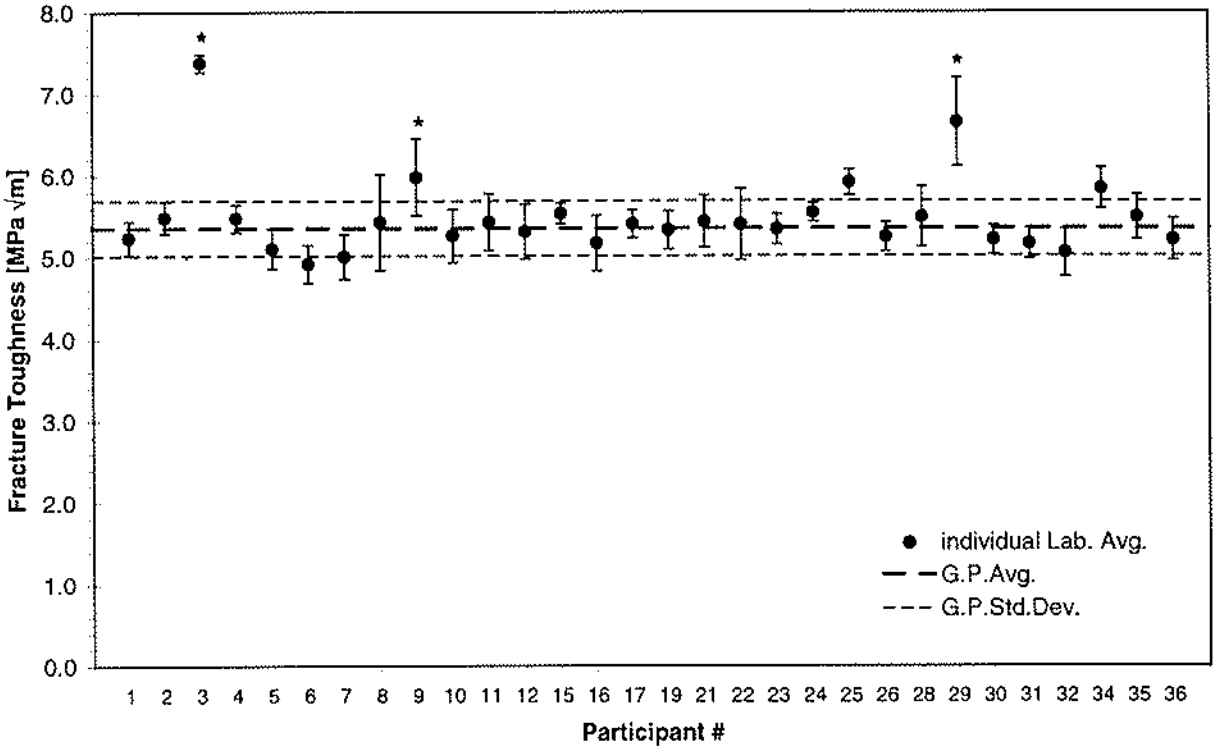


Figure 4.4.1
Master result graph for the GPSSN showing the individual participants' (Lab.) average with standard deviation. The broad dashed line represents the grand population average (G.P.Avg.) and the narrow outer ones, the grand population standard deviation (G.P.Std.Dev.). Results marked with a * were not used to compute the G.P.Avg. and G.P.Std.Dev.

Up to a notch width of 20 - 30 µm no significant influence of the notch width is visible in Figure 4.4.2. Twenty-seven of the thirty participants (90 %) were able to polish notches with a width ≤ 30 µm. Because of the wide V-notches the results from participants #3, #9, and #29 were not accepted (see below and Appendix 3). Like for the alumina materials, none of the results from participants #15, #17, #21, #24, #26, #32 and #35 were discarded because of a notch depth outside the limits.

Twenty-four participants reported 5 valid results each (100 %), one reported 4 (80 %), one 3 (60 %), and one 2 (40 %). Based on a total of 135 specimens tested by the twenty-seven participants, the overall success rate was 96 %. Participant #26 declared one of his five tests not valid, because he found cracks on the notch tip of the specimen. Participant #7 damaged one specimen during polishing the V-notch (he damaged an alumina-998 specimen too). One specimen of participant #7 was rejected by the organiser due to a V-notch width that was more than 7-times the width of the other one reported. Participant #17 declared 3 tests not valid due to the poor V-notch quality (see Appendix A3, participant #17; the V-notch shown is one of the poorest in the round robin).

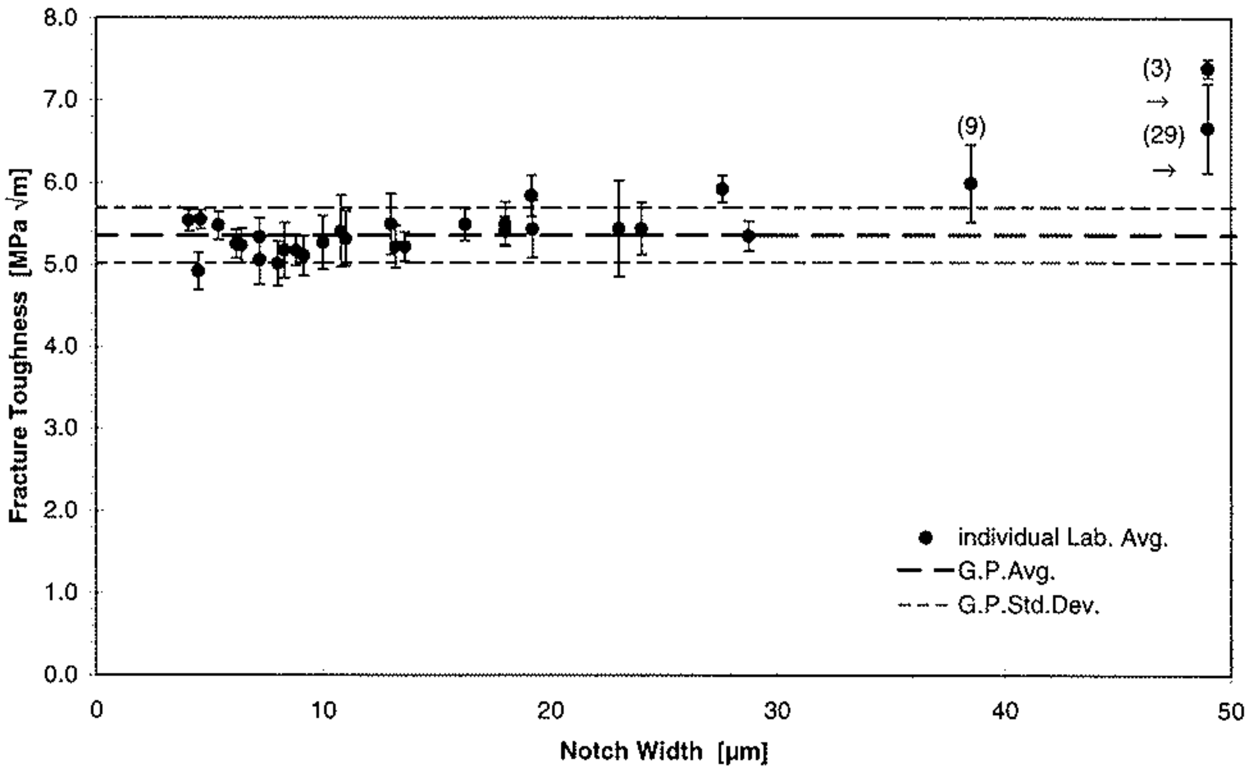


Figure 4.4.2
Notch width dependence of the fracture toughness of the GPSSN. Results not accepted are marked with the participant's code number in parentheses. The results marked with an → represent a V-notch widths of over 50 µm.

The mean fracture toughness measured by participants # 6, #7, #25 and #34 are outside the G.P.Avg. scatter bands as shown in Figure 4.4.1. Again, the specimens and test technique of those participants were analysed more carefully. As mentioned above, participant #25 used a 4-point bend test jig with spans of only 30 / 10 mm and support rollers set into grooves and thus not free to roll. Constrained rollers can lead to an overestimate of the bending strength of 4 % to 7 % and could explain the high values measured [L-4.1]. Of interest might be that the fracture toughness reported by participant #35, who used two fixed rollers, was also a little above the G.P.Avg. for the GPSSN and also for the alumina-998. 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 [L-4.2]. This might lead to an overestimate of the bending strength and could explain the high toughness values. No irregularities in the test technique of participants #6 and #8 could be found and an analysis of the fractured surfaces by the participants did not give any evidence of precracking or other damage, which could explain the low values. *(Remark by the organiser: Detecting scg crack growth on silicon nitride can be difficult and needs experience.)* No results from those four participants were discarded because of the test jig or advancing route of the V-notch.

Participant #8 measured the largest Std.Dev. for the GPSSN as Figure 4.4.1 and Table 4.1.1 show. He analysed the fractured surfaces using a stereo microscope on request from the organiser. The participant reported that the fractured surfaces were completely flat and that no special features could be found. Checking the reported notch depths a_1 to a_3 revealed that the specimen with highest fracture toughness values had a crooked notch. Extrapolating the crookedness to the specimen sides and using then the minimal and maximal depths to compute the fracture toughness would lead to an uncertainty of ± 0.15 MPa √m. But because the specimens fulfilled the criteria $(a_{max}-a_{min})/a \leq 0.1$ given in the instructions, it was not discarded.

The G.P.Avg. was within the scatter bands of twenty of the twenty-seven (74 %) participants and the mean of twenty-three of the twenty-seven (85 %) participants were within the G.P.Avg. scatter bands.

The central limit theorem for evaluating the consistency of the participants' results was applied on the GPSSN too. Seventeen of the possible 27 (63 %) participants averages fell within one G.P.Std.Dev.$_x$ / $\sqrt{n}$ (expected 68 %) and 24 of the 27 (89 %) participants averages fell within two G.P.Std.Dev.$_x$ / $\sqrt{n}$ (expected 95 %).

4.5 Sintered Silicon Carbide

Figure 4.5.1 and Table 4.1.1 show the results measured for the SSiC, which had a G.P.Avg. of 2.61 MPa √m and a G.P.Std.Dev. of 0.18 MPa √m. The G.P.Avg. and G.P.Std.Dev are based on the results of all 56 tests accepted. The histogram of all accepted results in Appendix A2 shows that the graph has a well-formed shape with a peak at about 2.7 MPa √m.

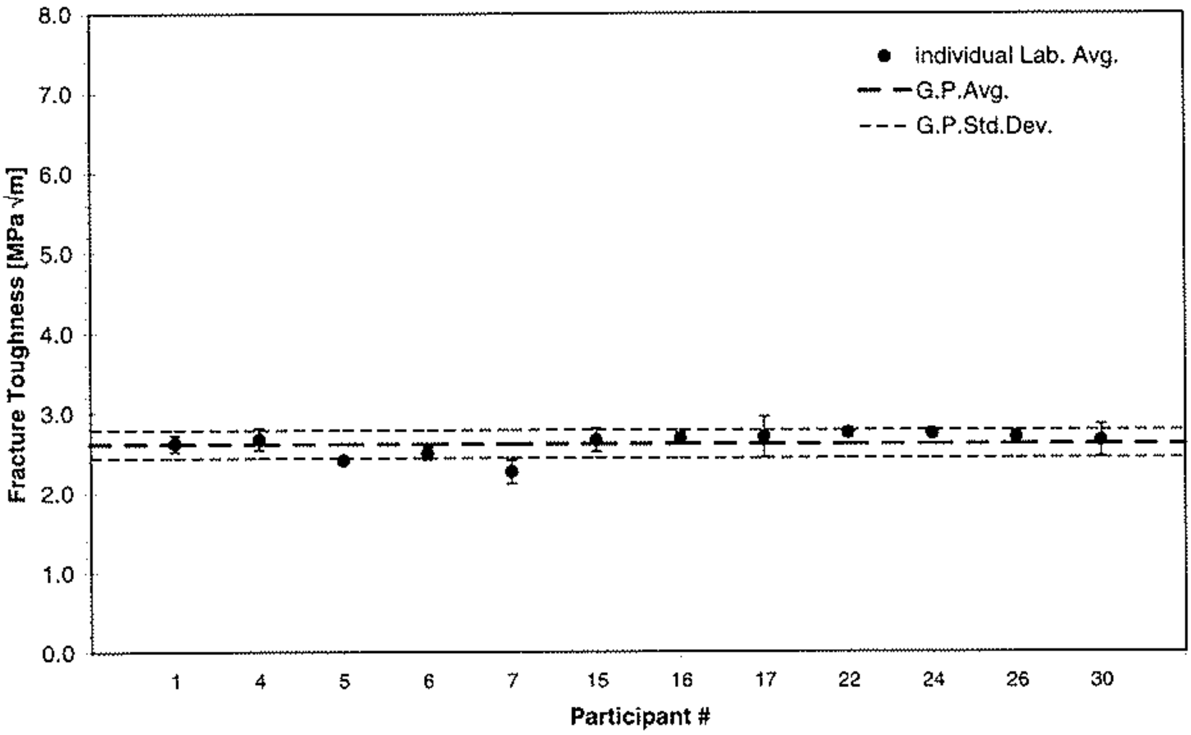


Figure 4.5.1
Master result graph for the SSiC showing the individual participants' (Lab.) average with standard deviation. The broad dashed line represents the grand population average (G.P.Avg.) and the narrow outer lines the grand population standard deviation (G.P.Std.Dev.).

Participant #17 initially reported a fracture toughness of 3.29 ± 0.20 MPa √m which was significantly higher than the others. No explanation could be found for the high values. Because he was the only participant who used a wire saw to cut the starter notches (see Appendix A3) and his notches were also shorter than asked for in the instructions, he was asked to repeat the fracture toughness measurements on the SSiC. The average fracture toughness measured on the second set was 2.30 MPa √m but this time with a large standard deviation of 0.58 MPa √m. A closer look at the results showed that the fracture toughness measured on two of the five specimens was very low. On request, the participant supplied digital images of those two fractured surfaces. On both surfaces large precracks (pop-in) could be recognised. Therefore, only three specimens from the second test set were used in the analysis of the round robin.

The second test result of interest is from participant #7. He reported a fracture toughness of 2.65 MPa √m with a large standard deviation of 0.42 MPa √m. The high fracture toughness measured on a single specimen was responsible for the large standard deviation. If only the four other specimens could be taken into account a standard deviation of only 0.08 MPa √m would have resulted. The participant reported that using fractography no anomalies were found. Therefore, he was asked to repeat the tests. For the second set of five specimens he reported four valid tests with an average fracture toughness of 2.19 MPa √m and a standard deviation of 0.08 MPa √m. For the analysis, the second set of test specimens of participant #7 were used.

Of interest might be, that no participant reported problems polishing V-notches into the SSiC. Qualitatively good V-notches were polished for example by the participants #5 and #6 (see Appendix A3). Participant #26 polished small V-notches but with tilted notch tips.

No influence of the notch width is visible in Figure 4.5.2. All twelve participants (100 %) were able to polish notches with a width ≤ 30 µm. As mentioned above, the test results from the round robin organiser (participant #1) were taken out of the previous mini-round robin.

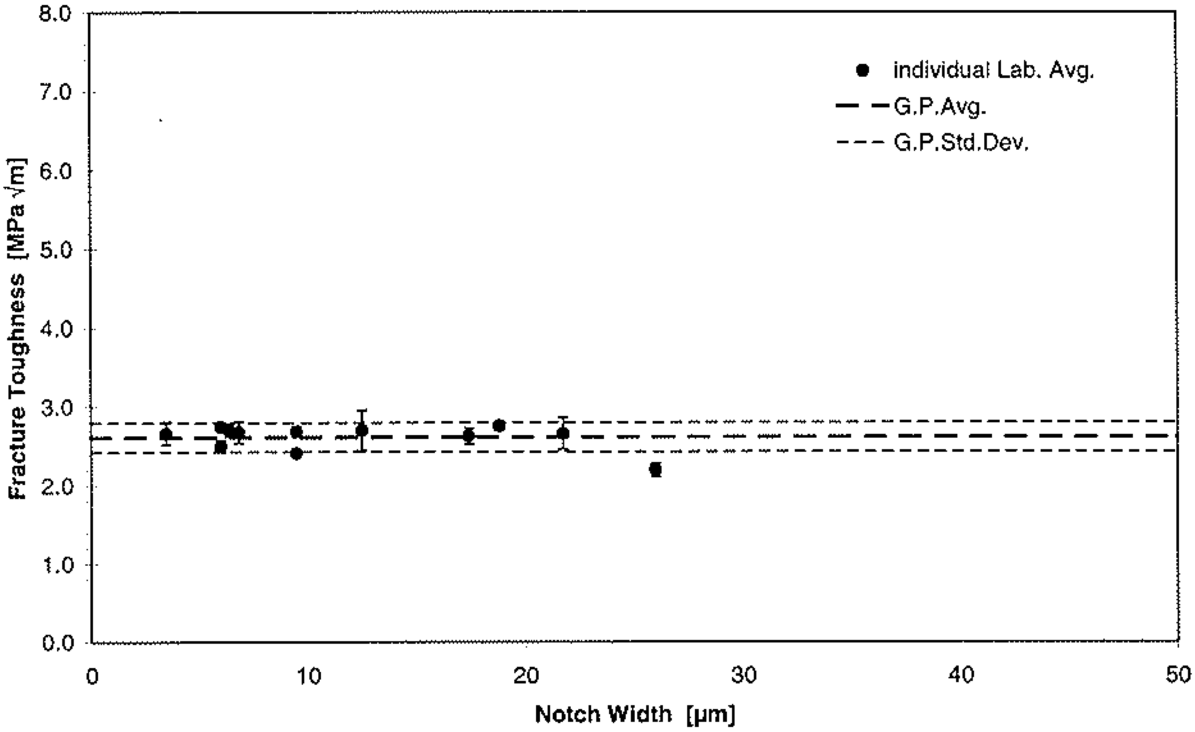


Figure 4.5.2
Notch width dependence of the fracture toughness SSiC. All results were accepted.

Nine participants reported 5 valid results each (100 %), two reported 4 (80 %) and one only three (60 %). Participant #30, who had only 4 successful tests, reports that he made a mistake preparing the starter notch on one specimen. Based on a total of 60 specimens tested by the twelve participants, the overall success rate was 93 %.

The G.P.Avg. was within the scatter bands of six of the twelve (50 %) participants and the mean of ten of the twelve (83 %) participants were within the G.P.Avg. scatter bands.

Applying the central limit theorem for evaluating the consistency of the participants' results, it was found that six of the possible twelve (50 %) participants' averages fell within one G.P.Std.Dev.$_{,x}$ / √n (expected 68 %) and ten of the twelve (83 %) participants' averages fell within two G.P.Std.Dev.$_{,x}$ / √n (expected 95 %).

4.6 Yttria-Stabilised Tetragonal Zirconia Polycrystal

Figure 4.6.1 and Table 4.1.1 show the results for the Y-TZP, which had a G.P.Avg. of 5.34 MPa √m and a G.P.Std.Dev. of 0.65 MPa √m. The G.P.Avg. and G.P.Std.Dev are based on the results of all 35 tests conducted. A histogram of all results in Appendix A2 shows that the results have a very flat distribution without reaching a real peak.

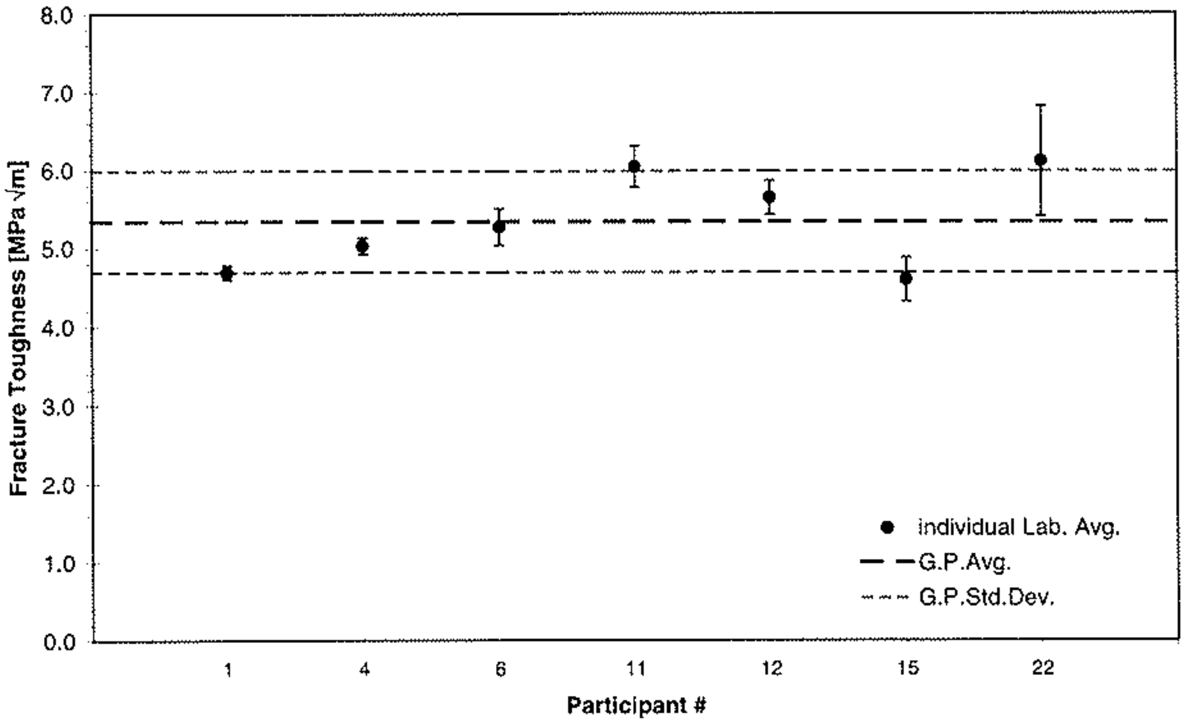


Figure 4.6.1
Master result graph for the Y-TZP showing the individual participants' (Lab.) average with standard deviation. The broad dashed line represents the grand population average (G.P.Avg.) and the narrow outer lines the grand population standard deviation (G.P.Std.Dev.).

The smallest notches polished into the Y-TZP were about 2 µm and the widest 16 µm, respectively. No significant influence of the large notch width distribution is visible in Figure 4.6.2. Therefore, no results were discarded due to the notch width. Next to the five specimens with a notch width of about 2 µm, the round robin organiser (participant #1) tested five specimens with a notch width of about 16 µm. Taking only those two test sets into account, a clear increase of the measured fracture toughness from 4.69 MPa √m to 5.48 MPa √m was seen.

The most noticeable result in Figure 4.6.1 is from participant #22 due to the large standard deviation. From the five specimens tested, one fractured at a substantially lower load than the others (see Appendix A2). Taking only the four others into account a fracture toughness of 6.42 MPa √m with a standard deviation of only 0.15 MPa √m would result. The participant could not find a reason for the outlier even using fractography. Therefore, the value was not discarded.

All seven participants who tested the Y-TZP reported 5 valid results each. Therefore, based on a total of 35 tests, the overall success rate was 100 %.

The G.P.Avg. was within the scatter band of only one (14 %) participant and the mean of four of the seven (57 %) participants were within the G.P.Avg. scatter bands.

Applying also the central limit theorem for evaluating the consistency of the participants' results it was found that only one of the possible seven (14 %) participants' averages fell within one G.P.Std.Dev.$_x$ / $\sqrt{n}$ (expected 68 %) and only three of the seven (43 %) participants' averages fell within two G.P.Std.Dev.$_x$ / $\sqrt{n}$ (expected 95 %).

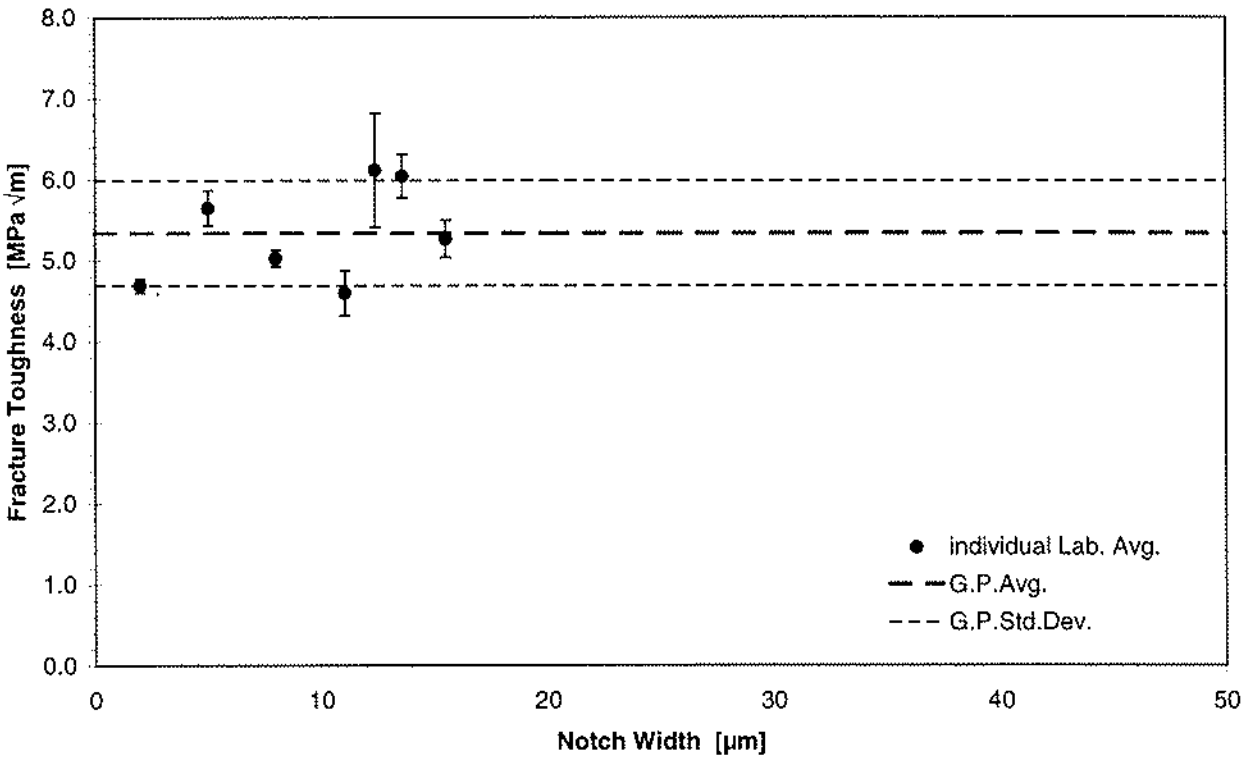


Figure 4.6.2
Notch width dependence of the fracture toughness of the yttria-stabilised tetragonal zirconia polycrystal (Y-TZP).

5 Discussion

5.1 Notch quality

Twelve participants (#3, #8, #9, #11, #14, #19, #25, #29, #30, #34, #35, and #36) polished the notches by hand as described in the instructions (Appendix A1) and 19 participants used a machine. The participants usually built the machines themselves or modified an existing one, for example participant #10 used a modified polishing machine. Typical V-notch polishing times for five specimens reported were 0.5 h for the aluminas and 1 to 1.5 hours for the GPSSN and SSiC. Some participants using a notch-polishing machine found that it was not necessary to saw a notch (slot) as a guide for the razor blade when a coarse-grained diamond paste was used at the beginning to reduce the polishing time.

To polish the notches, some participants used 250 µm thick (razor) blades normally used to scrape paint from window glass. Others used real razor blades with thicknesses ranging from 100 µm to 200 µm. The polishing stroke length varied from about 4 mm to 20 mm. The polishing itself was done with frequencies ranging from 1 Hz (hand) up to 15 Hz (machine) and 0.5 to 3 µm diamond paste. Participants reported that they used loads on the razor blades between 1 N and 5 N. A first analysis of all this information furnished by the participants did not give a clear picture on the influence of the parameters. The only thing noticeable was that the participants #3, #9, and #29, whose results were not used for the statistical analysis due to the large notch width, polished the notches by hand. On the other hand, participant #19 seemed not to have any problems to polish notches by hand as small as the smallest ones polished by machine. The fracture toughness he measured for the alumina-998, alumina-999 and GPSSN compared very well with the G.P.Avg. of the individual materials.

An overview of the V-notch and V-notch tip quality produced by the participants is shown in Appendix A3. On the upper end of a subjective notch quality scale are the notches of participants #5, #6, and #32 and on the lower end, those of participants #17, #22, and #23. Interestingly, no influence of the notch quality on the average fracture toughness measured can be seen but the standard deviations reported by participant #17, #22, and #23 are larger than those of participant #5, #6, and #32.

The general impression is that it is easy to polish V-notches with a width between 20 µm and 30 µm, but that it needs somewhat optimised parameters for each combination of polishing machine or hand, razor blade, stroke length, frequency, and load for notches smaller 10 µm.

5.2 Notch width

The fracture toughness measured on edge notched bend bars can be influenced by the notch width as is well known and mentioned in many publications. From a practical point of view, this had been demonstrated for example by Primas and Gstrein in an ESIS round robin [L-1.5, L-1.6]. Munz and Fett found that the measured fracture toughness K_I rises above a critical notch width S_{crit} [L-5.1, L-5.2]. Munz, Bubsy and Shannon 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 not wider than 70 µm [L-5.3]. Kübler found empirically that fracture toughness values which are comparable with other methods, could be measured on three different silicon nitrides and also on an alumina, Y-TZP, SSiC, and CMC if the notch width was less than about twice the size of a major microstructural feature, for example the average grain size [L-1.10]. Interesting theoretical work by Fett and Munz [L-5.4, L-5.5], a study by Damani, Gstrein and Danzer [L-1.7] and a new yet unpublished analysis by Fett [L-5.6, L-5.7] seem to confirm the $S \leq 2 \cdot a_{mfs}$ (S: acceptable notch width; a_{mfs}: major microstructural feature size) criteria to estimate an acceptable notch width for a material. In Appendix A5: Notch width – Theory and Model this is discussed in more detail. *(Remark: Hence forth, the expressions "edge notch model" or "semi-elliptical model" will be used if data is fitted in accordance with the Fett-Munz model or Fett model, respectively, as discussed in Appendix A5.)*

Acceptable notch width: For a first validity check of the round robin test results simply the Figures 4.2 to 4.6 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 or not to accept a result. (*Remark: All notches reported for the SSiC and Y-TZP were smaller than 30 µm*).

To calculate the acceptable notch width with respect to the criteria $S \leq 2 \cdot a_{mfs}$ as discussed before and in Appendix A5, the major microstructural feature sizes for the ceramics had to be defined first and used thereafter. Table 5.2 shows the sizes, which were defined, based on the micrographs in chapter 2.

Table 5.2: Acceptable notch width

Material	average grain size, Table 2.1 [µm]	major microstructural feature sizes a_{mfs} [µm]	acceptable notch width S [µm]	Remarks
Alumina-998	> 10	~ 17	34	length of larger grains
Alumina-999	~ 1.7	~ 4.5	9	length of larger grains
GPSSN	< 1	~ 3.5	7	length of elongated grains
SSiC	7	~ 10	20	length of larger grains
Y-TZP	0.45	~ 0.8	1.6	diameter of larger grains

Alumina-998: Using a major microstructural feature size a_{mfs} = 17 µm (Table 5.2) and therefore an acceptable notch width of S = 34 µm a good agreement is found to the visually defined 30 µm (Figure 4.3.2).

Alumina-999: For the alumina-999 the major microstructural feature size is 4.5 µm (Table 5.2) and therefore the acceptable notch width 9 µm. Only participants #1, #4, #6, #10, #17, #19, and #24 were able to polish such small notches. Using only the results from these participants a fracture toughness of 3.63 ± 0.32 MPa √m had been calculated. Interestingly, this value does not significantly differ from the one based on all accepted results (3.74 ± 0.40 MPa √m). Also Figure 5.2.1 reveals no significant influence of the notch width up to 30 µm. In order to get an idea about the influence of the notch width under exactly the same test conditions – notch polishing, equipment and procedure – the round robin organiser (participant #1) tested an additional set of five bend bars with a notch width of 25 µm. As shown by the data points marked 1-1 and 1-2 in Figure 5.2.1, the fracture toughness increased only by 0.13 MPa √m. Figure 5.2.1 shows further that the edge crack and even more the semi-elliptical crack models overestimate the influence of the notch width.

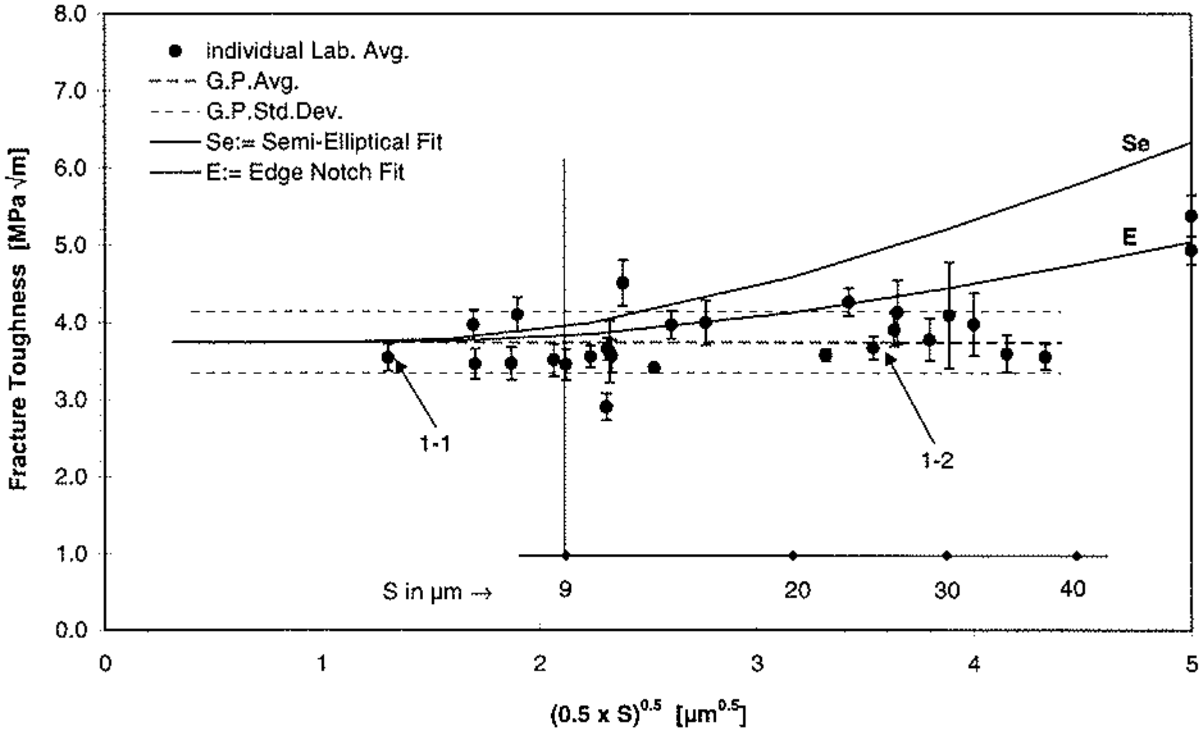


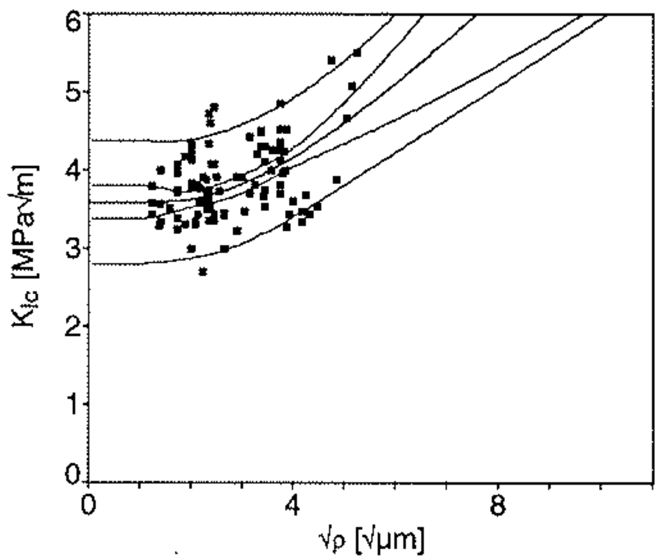
Figure 5.2.1
Influence of the notch width for the alumina-999. The data points marked 1-1 and 1-2 were tested under exactly the same test conditions. (S = notch width)

Besides the organiser, Rudolph investigated the notch width influence but used only individual test results for which the notch width was known [L-5.30]. For the analysis itself, she used also the edge notch model but fitted the crack size in front of the notch and also the Y-factor and found therefore a very good agreement as Figure 5.2.2 shows. The following estimated values were computed:

fracture toughness	$K_{Ic} \approx 3.6\ \{3.4;\ 3.8\}_{90\%}$ MPa √m
Y-factor	$Y \approx 0.96$
crack size	$a_{mfs} \approx 7.4$ µm
critical notch width	$S \approx 16$ µm ($S = 2\,\rho$; computed for $K_{measured}/K_{Ic} = 0.95$)

As Figure 5.2.2 shows, the measured fracture toughness for the "critical" notch width of 16 µm (√ρ = 2.8 √µm) as computed by Rudolph is slightly influenced by the notch width whereas the fracture toughness for the "acceptable" notch width of 9 µm (√ρ = 2.1 √µm; Table 5.2) is not, at least it is not noticeable.

So the maximum acceptable notch width of 9 µm is stringent or in other words, the relation $S \leq 2 \cdot a_{mfs}$ is very helpful to estimate the acceptable notch width.



ρ: notch root radius
outer lines: 90 % confidential intervals

Figure 5.2.2
Influence of the notch width for the alumina-999, if only individual test results are used for which the notch width is known. The analysis had been conducted in accordance with edge notch model but next to the crack size in front of the notch also the Y-factor was fitted.
Source: Rudolph [L-5.30]

GPSSN: The relation $S \leq 2 \cdot a_{mfs}$ delivers an acceptable notch width of only 7 μm (about 4 times smaller than the one visually defined) for the GPSSN. Similar to the fine-grained alumina only a few participants were able to fulfil this stringent assumption (participants #1, #4, #6, #15, #24 and #26). Using again only the results from these participants, a fracture toughness of 5.33 ± 0.28 MPa √m was calculated. These values do not significantly differ from those based on the tests with notches of up to 30 μm (5.36 ± 0.34 MPa √m).

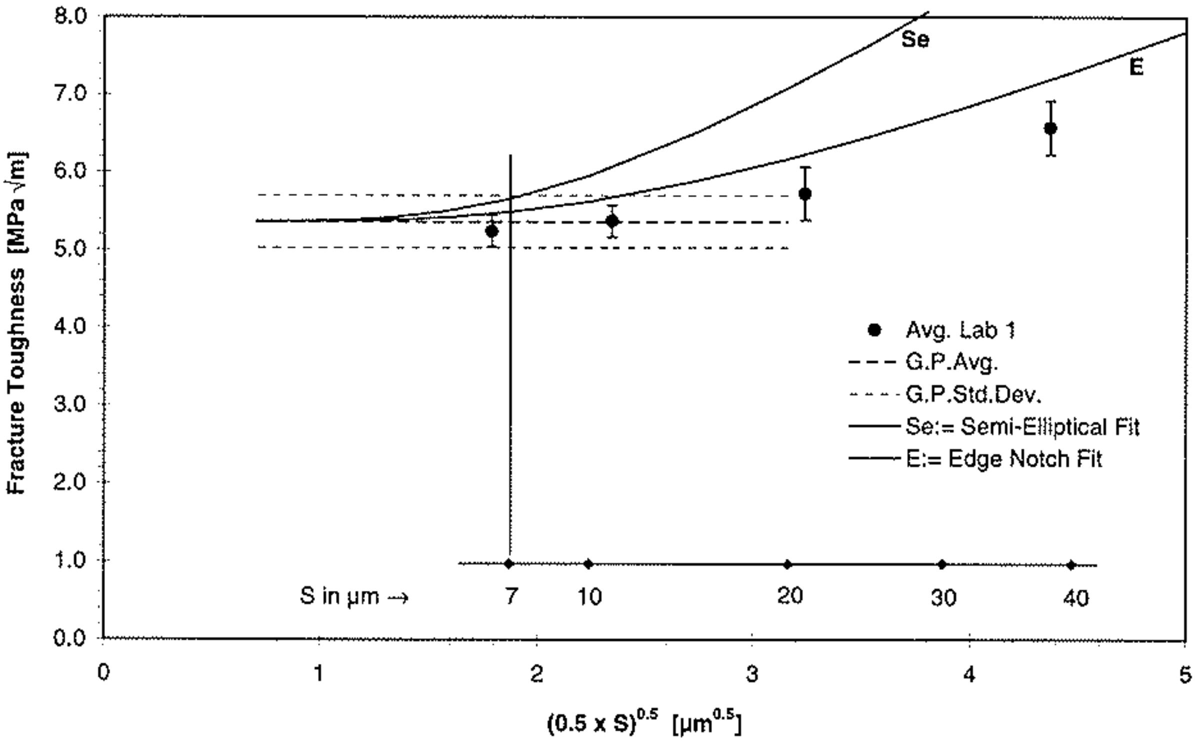


Figure 5.2.3
Influence of the notch width for the GPSSN under exactly the same test conditions.
(S: notch width)

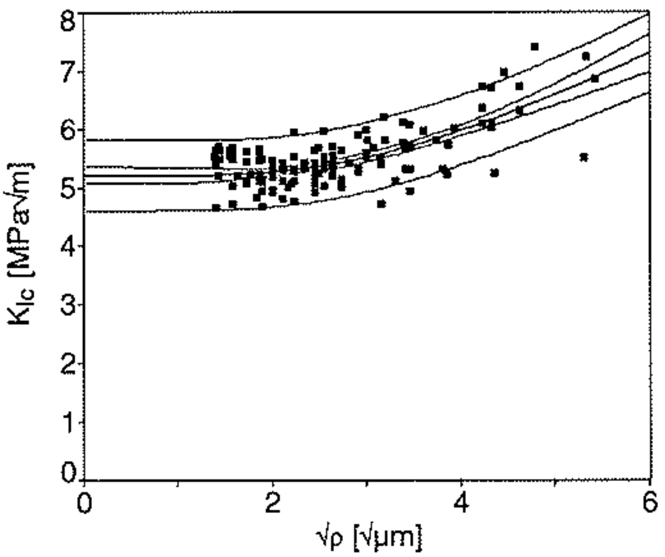
The round robin organiser (participant #1) tested additional bend bars again with varying notch widths to get an idea about the notch width influence on the GPSSN under exactly the same test conditions. Already at very small notches an increase of the measured fracture toughness was noticeable. Figure 5.2.3 shows that the acceptable notch width of 7 µm as calculated from the relation $S \leq 2 \cdot a_{mfs}$ is in good comparison with the test data. The measured average fracture toughness does not drop out of the grand population standard deviation up to a notch width S = 20 µm.

As described before, Rudolph investigated the notch width influence also for the GPSSN using only individual test results for which the notch width was known [L-5.30]. The following estimated values were computed:

fracture toughness	$K_{Ic} \approx 5.2 \{5.1; 5.3\}_{90\%}$ MPa √m
Y-factor	Y ≈ 0.8
crack size	$a_{mfs} \approx$ 11 µm
critical notch width	S ≈ 16 µm (S = 2 ρ ; computed for $K_{measured}/K_{Ic} = 0.95$)

As Figure 5.2.4 shows the measured fracture toughness for the "critical" notch width of 16 µm (√ρ = 2.8 √µm) as computed Rudolph is already influenced by the notch width whereas the fracture toughness for the "acceptable" notch width of 7 µm (√ρ = 1.9 √µm; Table 5.2) is not, at least it is not noticeable.

Therefore, also for the GPSSN the relation $S \leq 2 \cdot a_{mfs}$ is very helpful to estimate the acceptable notch width.



ρ: notch root radius
outer lines: 90 % confidential intervals

Figure 5.2.4
Influence of the notch width for the GPSSN, if only individual test results are used for which the notch width is known. The analysis had been conducted in accordance with the notch edge model but next to the crack size in front of the notch also the Y-factor was fitted.
Source: Rudolph [L-5.30]

SSiC: From the twelve participants who tested the SSiC only two had polished notches slightly outside the acceptable notch width S = 20 µm (Table 5.2). Also Figure 4.5.2 shows no problem measuring the fracture toughness of the SSiC with the SEVNB method.

Y-TZP: The very fine grained Y-TZP had been chosen for the round robin because it was assumed that the relation $S \leq 2 \cdot a_{mfs}$ could not be fulfilled, or would be at least the most difficult [L-1.10]. Figure 5.2.5 reveals that actually no participant was able to polish notches with an acceptable notch width of 1.6 µm or less (see Table 5.2).

In Figure 5.2.5 an influence of the notch width is recognisable. Therefore and to get an idea of the notch width influence under exactly the same test conditions, the round robin organiser (participant #1) tested two sets of 5 specimens, marked 1-1 and 1-2 in Figure 5.2.5. Also incorporated in the Figure is the SCF fracture toughness measured in an earlier VAMAS round robin [L-1.1]. The SCF method works with real cracks or in other words with a notch or defect width of ≈ 0 µm. It is interesting to notice that the SEVNB fracture toughness measured on the bend bars with the smallest notches (marked 1-1) is within the SCF standard deviation. As before, the edge crack and the semi-elliptical crack models are shown, here based on the SCF values. Taking the SCF fracture toughness value it seems to be clear that the SEVNB method if used with notches wider than 1.6 µm delivers too high fracture toughness values for the fine-grained Y-TZP.

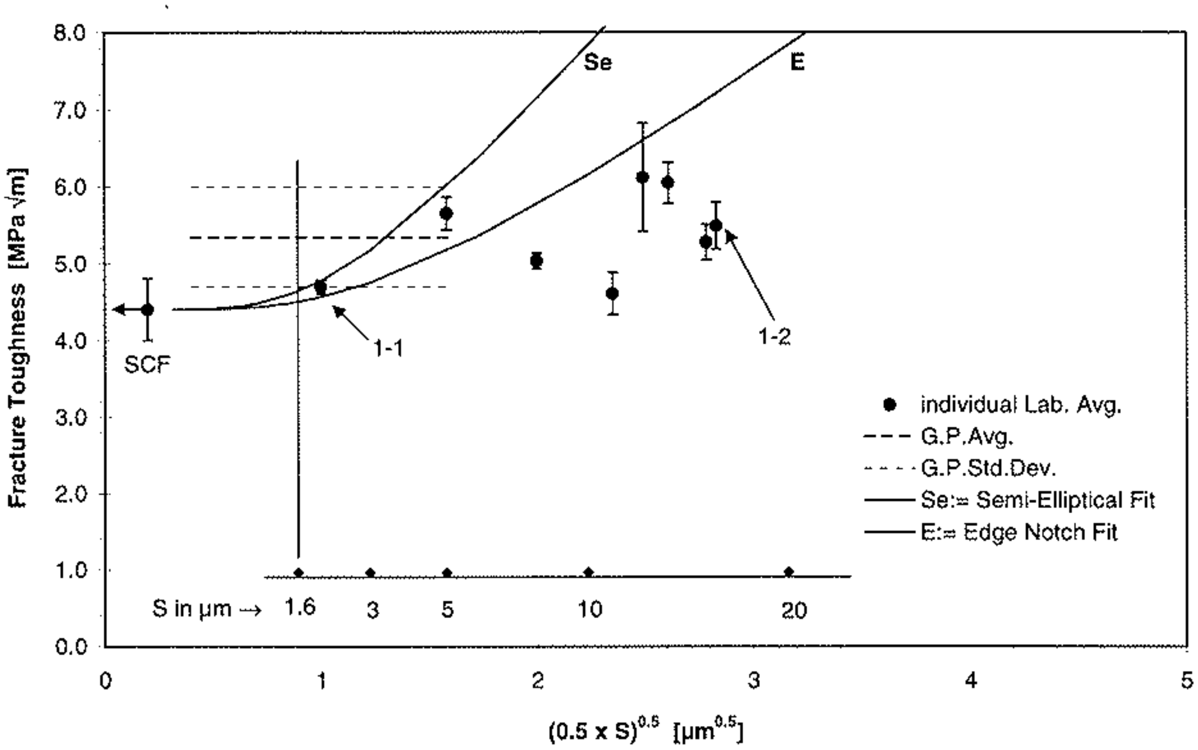


Figure 5.2.5
Influence of the notch width on the measured Y-TZP fracture toughness. The data points marked 1-1 and 1-2 were tested under exactly the same test conditions. Results from an earlier VAMAS round robin are marked "SCF" (Surface Crack in Flexure) [L-1.1].
(S: notch width)

So in summary the results of Figures 4.2.2, 5.2.1, 5,2.2, 5.2.3, 5.2.4 and materials alumina-998, alumina-999, GPSSN, SSiC leads to the following conclusion:
Rule of thumb: Notches smaller 10 µm deliver "good" fracture toughness values for ceramics with an average grain size greater about 1 µm.

5.3 Notch depth

The participants were asked to polish their notches to a depth between 0.8 mm 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, if there would be any influence at all. The small a/W-ratio, if compared with those demanded for the SEPB fracture toughness test method by the ASTM and JIS standards, had been chosen due to the following reasons [L-5.31, L-4.2]:

- A DIN draft standard for the determination of the fracture toughness asks for a crack (notch) depth of $0.2 < a/W \leq 0.3$ [L-3.2].
- Schindler showed that the measured fracture toughness obtained from SENB-specimens can increase with an increasing initial crack (notch) length a/W [L-5.32].
- If no saw cut is used for polishing the notches, shorter ones are finished faster.

Not all participants were able to polish notches between an a/W-ratio of 0.2 and 0.3, as Figure 5.3.1 reveals for the GPSSN. Additional 4-point bending tests marked + in the figure and additional 3-point bending test marked ++ were conducted by the round robin organiser and #30, respectively. All these SEVNB tests cover an a/W-ratio range between 0.1 and 0.55. As shown in Figure 5.3.1, no significant influence of the notch depth is recognisable for the GPSSN.

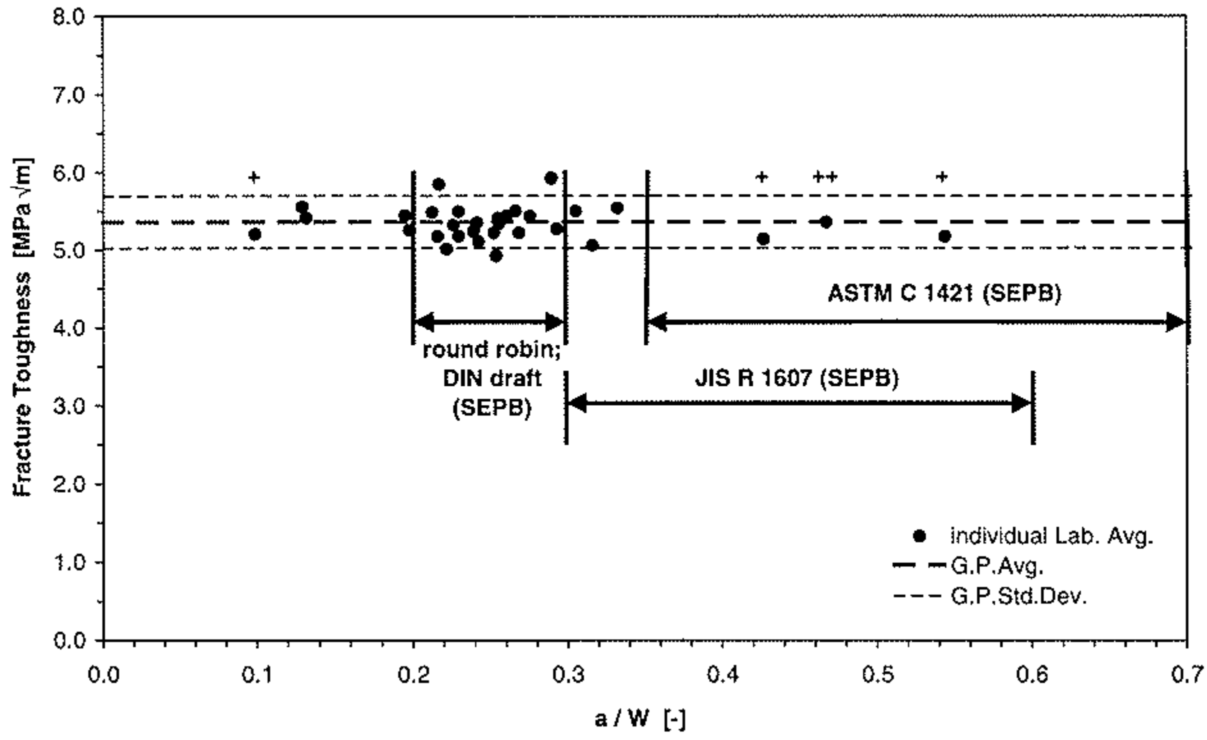


Figure 5.3.1
Notch depth dependence of measured fracture toughness. Additional 4-point bending tests are marked + and additional 3-point bending test ++, respectively.

Also for the coarse and fine-grained aluminas, no significant notch depth dependence is recognisable, as Table 5.3 reveals. Based on the discussed three ceramics, 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 exist. (Remark: No data is available to analyse the notch depth influence on the SSiC and Y-TZP fracture toughness.)

Table 5.3: Notch depth dependence

Alumina-998			Alumina-999		
Participant	K_{Ic} [MPa √m]	a/W [-]	Participant	K_{Ic} [MPa √m]	a/W [-]
#21	3.42 ± 0.22	0.15	#17	3.97 ± 0.19	0.16
#17	3.61 ± 0.06	0.16	#24	4.10 ± 0.22	0.16
#26	3.68 ± 0.07	0.16	#30 [1)]	3.86 ± 0.16	0.48
#24	3.64 ± 0.06	0.17			
#30 [1)]	3.53 ± 0.05	0.45			
#1	3.62 ± 0.07	0.51			
G.P.Avg	3.57 ± 0.22		G.P.Avg	3.74 ± 0.40	

[1)] 3-point bending

5.4 Repeatability and Reproducibility

To determine the repeatability (within–laboratory) and reproducibility (between–laboratories) of the SEVNB fracture toughness measurement method, ISO Standard 5725-2 "Accuracy (trueness and precision) of Measurement Methods and Results" was used. The statistical results are shown in Table 5.4 and are compared with results from an earlier VAMAS round robin with the SCF method [L-1.1]. 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 an identical manner. *(Remark: In ISO 5725-2 the criterion for an outlier is 1 % whereas in ASTM 691 it is 0.5 %.)*

Table 5.4: Repeatability and reproducibility coefficient of variance and standard deviation of the SEVNB method compared with the SCF method measured on hot pressed Si_3N_4 (earlier VAMAS round robin [L-1.1]). *(Remark: Only accepted data were used for the statistical analysis.)*

		Total		**Repeatability (within-lab)**		**Reproducibility (between-lab)**	
Material	Method	# Particip.	# Spec.	Std.Dev. MPa √m	CV %	Std.Dev. MPa √m	CV %
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	19	102	0.24	5.4	0.31	6.8
hot iso-pressed Si_3N_4	SCF	15	100	0.38	7.7	0.45	8.9

In accordance with Table 5.4, the repeatability of the SEVNB method for all ceramics tested in the round robin compares very well (or is even better) than those from the SCF method. The reproducibility of the SEVNB method measured for the alumina-998, GPSSN, and SSiC is in the range or lower than that measured for two different silicon nitrides with the SCF method. The between-laboratory coefficient of variation for the Y-TZP is the largest of all five ceramics tested but this is not surprising due to the strong notch width dependence found and discussed earlier. Possible reasons for the rather large between-laboratory coefficient of variation computed for the alumina-999 will be discussed later.

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 with 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: Only the within- and between-laboratory consistencies of participant #31 were so poor that they were rated outliers (see also Figure 4.2.1). As mentioned earlier, an analysis showed that two toughness values reported were very low (2.2 MPa √m and 2.4 MPa √m). On request of the organiser, the participant analysed the tested specimens fractographically, but could not find any signs of crack growth or other defects responsible for the low values. (*Remark: Detecting subcritical or stable crack growth on coarse-grained alumina can be difficult and needs experience.*)

Alumina-999: 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 but it might be of interest that

- participant #8 reported that he had problems at the beginning resetting the razor blade properly into the notch and therefore polished some small additional notches at the notch root, see Appendix A4.
- participant #22 had the same problems than participant #8, see Appendix A3.
- participant #31 explained that two specimens failed while polishing the V-notches. 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, Table 5.4. A reason for the high coefficients of variation could be local grain size variations in rods from which the specimens had been machined. The manufacturer of the rods mentioned that he had seen local grain size variations before, if this alumina-999 with MgO additions as a grain refiner had been post hipped. He supposes that the reason for such local grain growth are voids or areas with a different density, which allow neighbouring grains to grow into them. No significant grain size discrepancies could be found in the notch tip areas of randomly selected specimens from three different test conditions by the organiser, as Figure 5.4.1 shows. On the other hand, clusters of large grains with sizes up to 6 µm can be seen for example in Figure 5.4.1c near the notch tip.

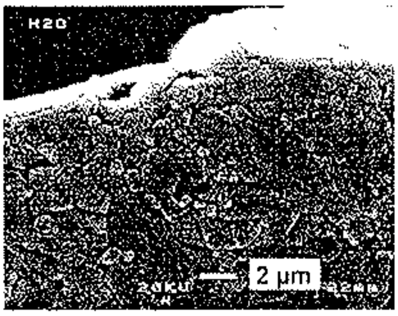


Figure 5.4.1a Frem2414-V2

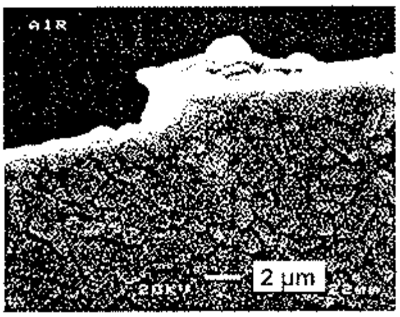


Figure 5.4.1b Frem2418-V2

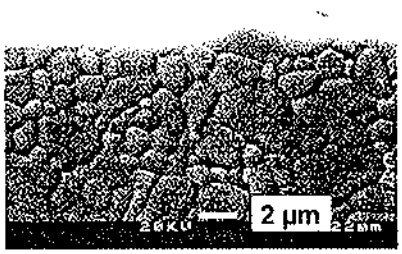


Figure 5.4.1c Fem3917-V3

Microstructure of alumina-999 bend bars in the area of the notch tip. Bend bar tested in

a) water K_{Ic} = 4.21 MPa √m (specimen 3)
b) air K_{Ic} = 3.33 MPa √m (specimen B5-1)
c) nitrogen atmosphere K_{Ic} = 4.02 MPa √m (specimen 2B)

(Remark: Surface preparation: etched at 1410 °C / 2h)

Another reason for the high coefficients of variation could be an environmental influence, for example scg. Therefore, the organiser tested additional sets of bend bars in nitrogen atmosphere and also water to suppress scg and support scg, respectively. Interestingly, the measured fracture toughnesses were not ranked as expected $K_{water} < K_{air} < K_{nitrogen}$ but $K_{air} < K_{nitrogen} \approx K_{water}$ (3.54 ± 0.17 MPa √m < 4.00 ± 0.22 MPa √m ≈ 3.97 ± 0.20 MPa √m). Therefore, if scg influences the measured fracture toughness the notch tips of the specimens tested in water had to be protected for example by polishing paste remains or H_2O-water molecules had more difficulties to reach the notch tip than H_2O-air ones. (Remark: Before testing in water, the specimens were cleaned in acetone, dried at 120° / 2h and watered for > 60h in distilled water.)

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 within-laboratory consistency of participant #22 and 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. *(Remark: No influence of the test jig used by participant #25 on the measured K_{Ic}-values could be seen for the two aluminas, which had a ~ 30 % lower fracture strength than the GPSSN.)* 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 and an analysis of the fractured surfaces by the participants did not give any evidence of precracking or other damage, which would explain the low values. Finally, participant #22 had again at least in some cases, the same difficulties resetting the razor blade properly into the V-notch and therefore, polished additional notches at the notch root, see Appendix A3. Because the reasons for the outliers and stragglers could not be absolutely clarified, none of the results of those participants were eliminated.

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, as Figure 4.5.1 and Table 5.4 show. The reasons for the stragglers and the outlier are not clear yet, but it might be of interest that participants #7 and #30 reported specimens which failed before the actual testing and that as mentioned before precracks (pop-in) were recognised on the fractured surfaces of two not accepted specimens from participant #17 (see Table 4.1.1, and chapter 4.5).

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 strong notch width dependence found and 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 the mentioned outlier.

5.5 Comparison of results with other methods

One of the important goals of the round robin was to see how well fracture toughness values measured with the SEVNB method compare with values determined with other methods. Results measured with methods like SCF and SEPB on a voluntary basis by some participants, are shown and compared with the outcome of the round robin.

Alumina-998: The comparison shows a strong discrepancy between the fracture toughness values measured by the SEPB, CN and SCF methods compared with the SEVNB method, Figure 5.5.1. Additional SEVNB tests by participant #16 and the round robin organiser 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).

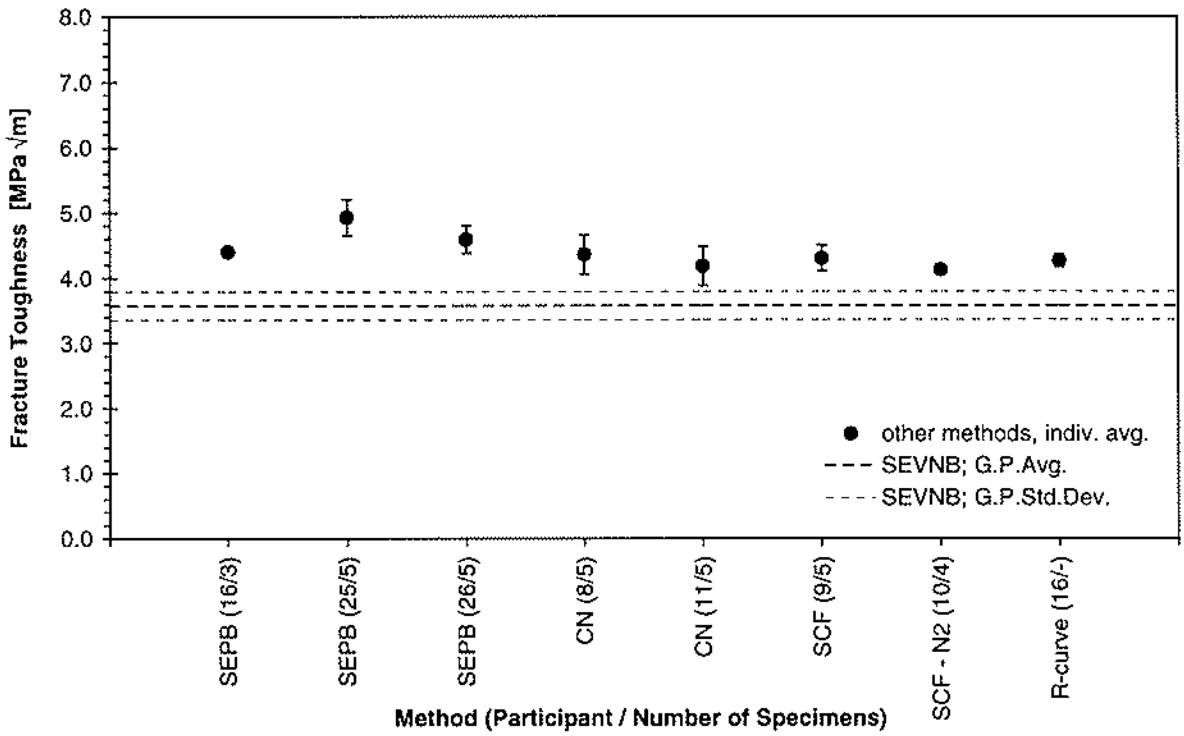


Figure 5.5.1
Comparison of fracture toughness values measured with test methods preferred by participants with values from the SEVNB method. *(The R-curve values from participant #16 were calculated with a crack size of 60 μm.)*

Participant #16 also measured the indentation strength and indentation crack length to calculate the R-curve behaviour in accordance with a procedure described by Krause [L-5.50] and determined a value of $K_R = 3.77\ [c]^{0.03}$ MPa √m (c = crack length in μm). If the fracture toughness values from SEPB ($K_{SEPB} \approx 4.6$ MPa √m; crack length: a ≈ 1500 μm), CN ($K_{CN} \approx 4.3$ MPa √m; crack length: a ≈ 1000 μm), SCF ($K_{SCF} \approx 4.2$ MPa √m; semi-elliptical crack: a ≈ 90 μm, 2c ≈ 230 μm) and the R-curve are compared with respect to the crack size, good agreement can be found. Further, using the smallest crack size observed in the R-curve determination (semi-elliptical crack: a ≈ ?, 2c ≈ 120 μm), the calculated fracture toughness K_R of 4.26 MPa √m also compares very well with the values measured, especially with the SCF ones.

The large discrepancy between the fracture toughness values measured with the SEVNB and the other methods, especially the SCF method having a rather small crack, might be explained by a study from Nishida et al. [L-5.51] 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) monolithic alumina with a microstructure similar to the one seen in this round robin, they detected and quantified the increase in the K_R value as 1.5 MPa √m within the first 10 to 20 grains just beyond the notch tip. On the other hand, they did not find an increase in the K_R value in a fine-grained (1 µm) alumina as Figures 5.5.2a show. Further, in a recently published study Pezzotti et al. measured on an alumina with a grain size of 25 µm a nearly linear R-curve with a slope of ≈ 2 MPa √m mm^{-1} and a critical stress intensity for crack propagation of only 1.8 MPa √m [L-5.52] using the same technique as described above. Therefore, the ≈ 0.6 MPa √m higher SCF fracture toughness values compared with the SEVNB ones of the alumina-998 (grain size 10 µm) could be explained or at least part of it.

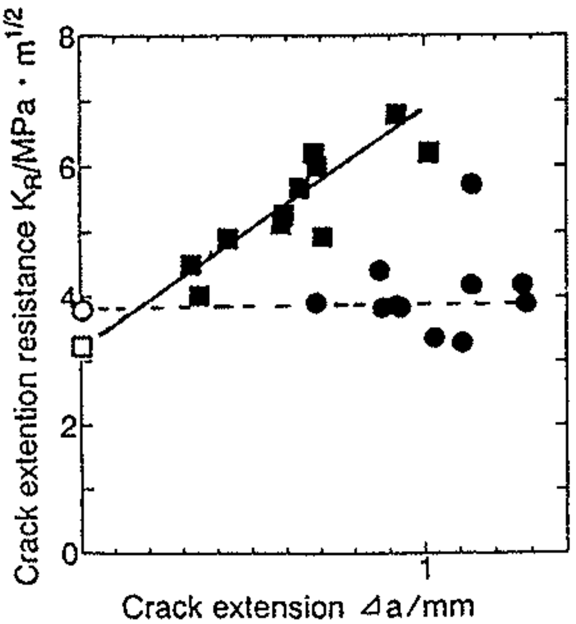


Figure 5.5.2a
R-curve of a ●; fine- (1 µm) and ■; coarse-grained (18 µm) alumina. The lowest value of the stress intensity factor (open marks) was calculated from the initial V-notch depth and the onset load value for non-linearity – in the load – deflection curve, P_i shown in Figure 5.5.2b. (Lines drawn by round robin organiser.)

Source: Nishida et al. [L-5.51]

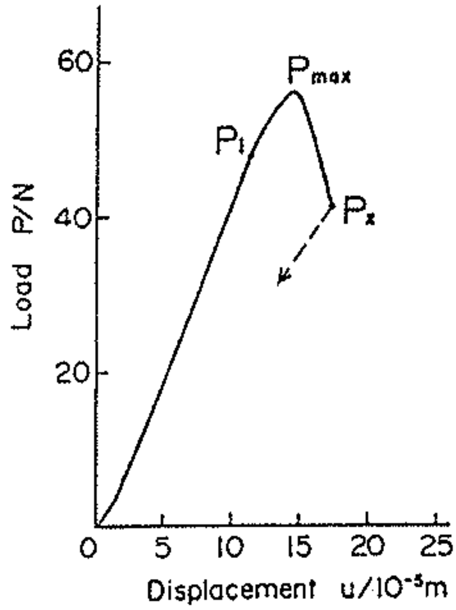


Figure 5.5.2b
Typical load – load-point displacement curve of a silicon nitride. The curve looses its linearity at the load value P_i. Then, during stable crack propagation, the load quickly released at the value P_x to check the crack extension length.

Source: Nishida et al. [L-5.51]

Another possibility for the large discrepancy could be stable crack growth or a pop-in during all SEVNB tests. For example, undetected crack growth of ≈ 250 µm would increase the fracture toughness from 3.6 to 4.2 MPa √m and therefore to the value measured with the SCF method. Only participant #10, an experienced fractographer, saw evidence of a 50 µm to 150 µm "initiation" region at the root of the notches. He thinks that the steps or little jogs found at the notch root are suggesting that small cracks had popped in on slightly different planes and orientations before they converged to one main crack. Therefore, the round robin organiser took a closer look at his digitally recorded load-time curves and found evidence of stable crack growth, assuming no plastic deformation in the alumina-998. As can be seen in Figure 5.5.3a, the load-time curves do not have a linear behaviour in the final part before the failure of the specimen. It seems as if this behaviour does not depend on the a/W-ratio, crosshead speed or notch width, Figure 5.5.3b.

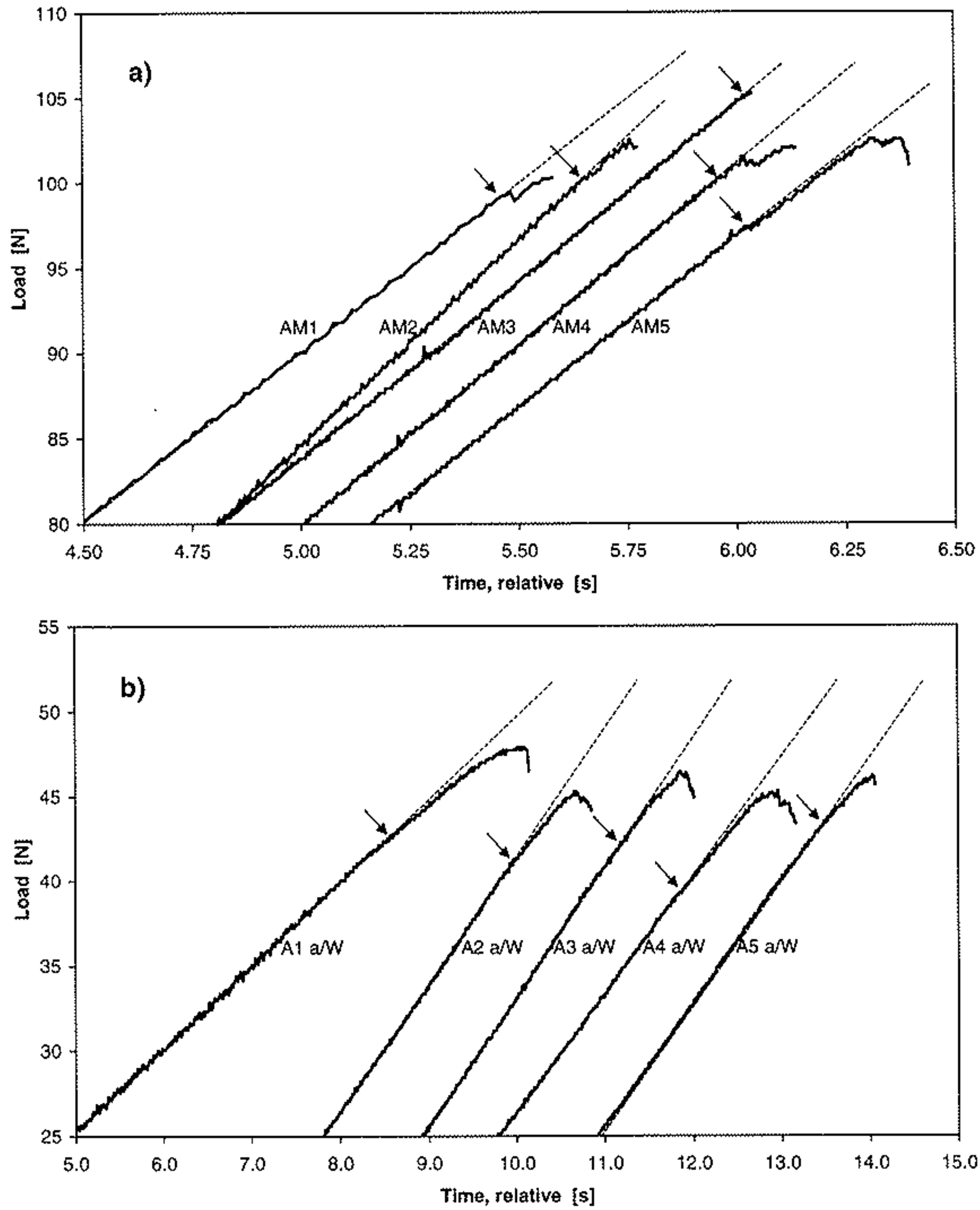


Figure 5.5.3a and b
Digitally recorded load-time curves measured on the alumina-998 by the round robin organiser (participant #1). The dashed lines demonstrate the slope of the curves in the last 20 % and 50 %, respectively, and the arrows mark the area where a first deviation from the straight line is recognisable. The following test conditions were used:

a) a/W ≈ 0.22 mm, crosshead speed ≈ 0.3 mm/min, notch width ≈ 20 µm, K_{Ic} = 3.67 MPa √m (specimens from the round robin)
b) a/W ≈ 0.51 mm, crosshead speed ≈ 0.15 mm/min, notch width ≈ 4 µm, K_{Ic} = 3.62 MPa √m (additional specimens used to evaluate the notch width influence)

The arrows in Figure 5.5.3 mark the areas where a first deviation from the linear behaviour of the load-time curves is noticeable. From all five curves shown in Figure 5.5.3a this deviation starts last on specimen AM3 on which the highest fracture toughness (3.80 MPa √m) had been measured. On the other four specimens of this batch an average fracture toughness of

3.64 MPa √m with a surprising small standard deviation of only 0.02 MPa √m had been measured. The discrepancy in fracture toughness between specimen AM3 and the average value of the others of 0.16 MPa √m could represent a crack growth of 70 µm and therefore in the order of the crack "initiation" region found by participant #10.

Combining the effects provoked by a crack "initiation" region with the K_R increase within the first grains beyond the notch tip, as found by Nishida et all., the discrepancy in fracture toughness between the SEVNB and the other methods might be explained.

Alumina-999: Five participants did additional fracture toughness tests as Figure 5.5.4 shows. The fracture toughness measured with the SEPB (participant #25 and #26), CN (participant #8) and one with the SCF method (participant #10) is on the lower end of the SEVNB spread band (G.P.Avg. minus Std.Dev.) whereas the second SCF value (participant #9) is on the higher end (G.P.Avg. plus Std.Dev.). A fractographic analysis of the fractured SCF surfaces by participant #10 revealed "halos" around the precracks (rings that are darker or brighter than the precrack). Swab and Quinn found such "halos" before in alumina [L-5.53]. Therefore, participant #10 concluded that environmentally assisted scg was the cause for the "halos" and that the "real" fracture toughness is 4.12 MPa √m. Additional SCF and SEVNB tests by participant #10 and the round robin organiser in a nitrogen atmosphere confirmed the scg suspicion (see also Figure 5.5.4). On the other hand, the organiser measured also a high fracture toughness in distilled water but as described in chapter 5.4 "protected" notch tips might be responsible for that.

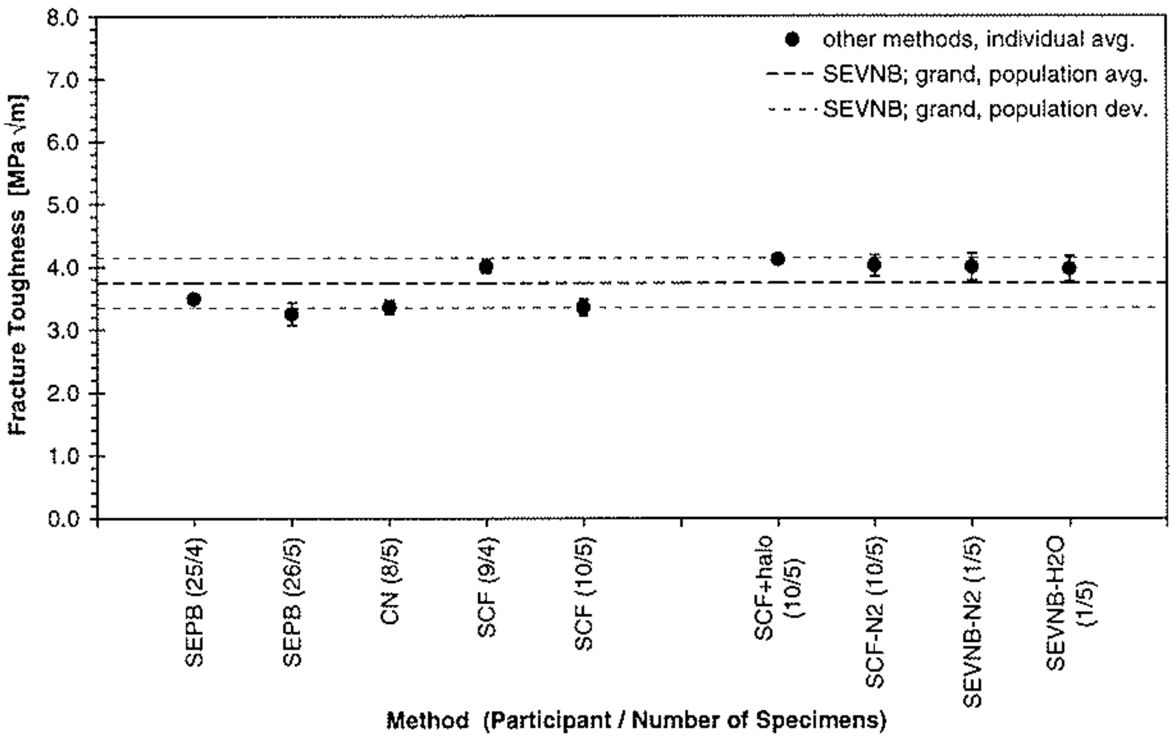


Figure 5.5.4
Comparison of fracture toughness values measured with test methods preferred by participants with values from the SEVNB method. The fracture toughness values SCF(10/5) and SCF+halo(10/5) are calculated from the same test set. N2: tested in nitrogen atmosphere; H2O: tested in distilled water.

A further analysis of Figure 5.5.4 reveals two groups of results. One group contains fracture toughness values at the lower end of the SEVNB standard deviation band:

- SEPB: participant #25 and #26

- CN: participant #8
- SCF: participant #10.

and another group at the higher end, respectively:

- SCF: participant #9, the "halo"-corrected ones and those tested in a nitrogen atmosphere from participant #10
- SEVNB: tested in nitrogen atmosphere and those tested in water from participant #10.

Figure 5.5.5 shows all accepted fracture toughness values measured with the SEVNB method for alumina-999 grouped by participants. It is interesting to see that also here exist primarily the above-described groups. Into the group on the lower end belong the fracture toughness values from participants #1, #4, #5, #6, #10, #11, #16, #19, #21, #31 and into the one at the higher end those from participants #8, #15, #17, #23, #24, #28, #30, #34. Only the results from participants #22, #25 and #34 do not clearly belong to one group or the other. It is very interesting to see that the computed fracture toughness for the lower group is 3.47 ± 0.22 MPa √m and 4.11 ± 0.30 for the higher one, respectively. In conclusion, a very good agreement is found with the two groups seen in Figure 5.5.4 and therefore a strong suspicion of subcritical or slow crack growth (scg) as being responsible for the large scatter of results. The only point unclear at present is what causes an alumina-999 SEVNB test result to be in the scg influenced or not-influenced group, respectively. *(Hypothesis: Not removed oil based polishing paste can protect a notch tip and therefore suppress scg. Whereas notches polished with non-protecting paste (or well removed oil based polishing paste) are exposed to scg.)*

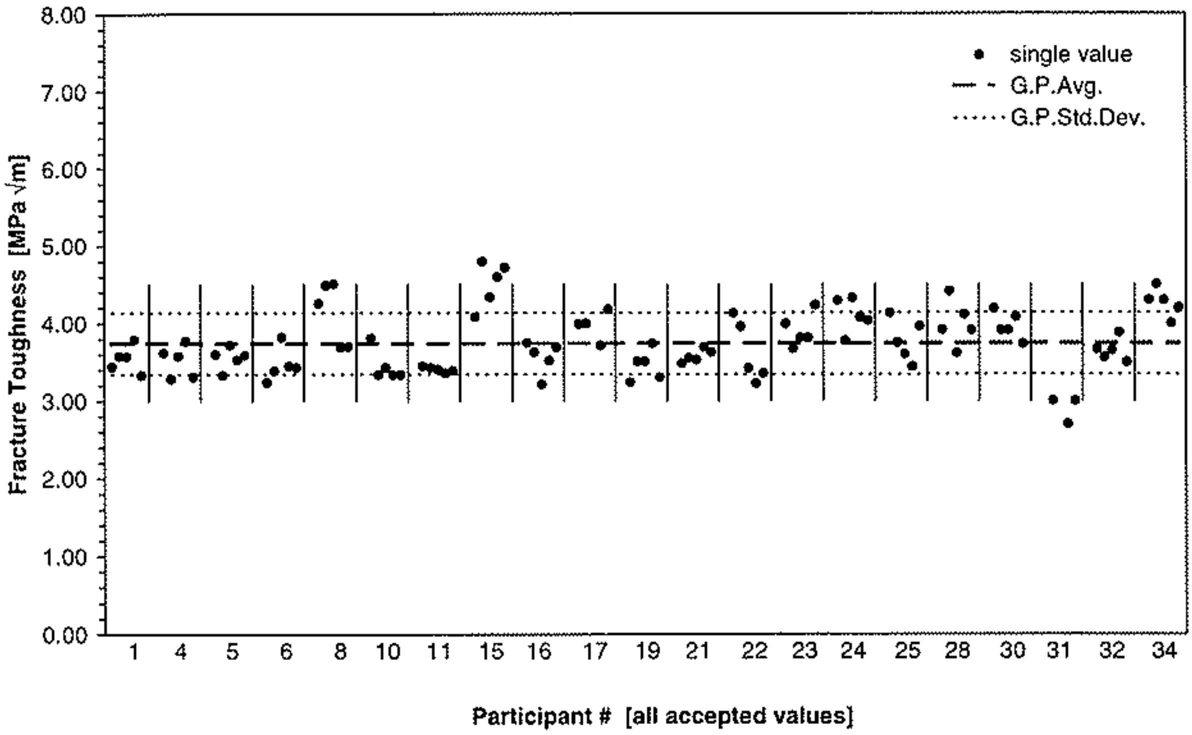


Figure 5.5.5
All accepted SEVNB fracture toughness values measured on the alumina-999, grouped by participants.

Finally, the round robin organiser took again a closer look at his digitally recorded load-time curves from the tests in air, nitrogen atmosphere and also distilled water. As can be seen in Figure 5.5.6, all load-time curves do have a linear behaviour in the final part before the failure of the specimen. Therefore, the curves do not give any evidence of stable crack growth or a pop-in as before those of the alumina-998.

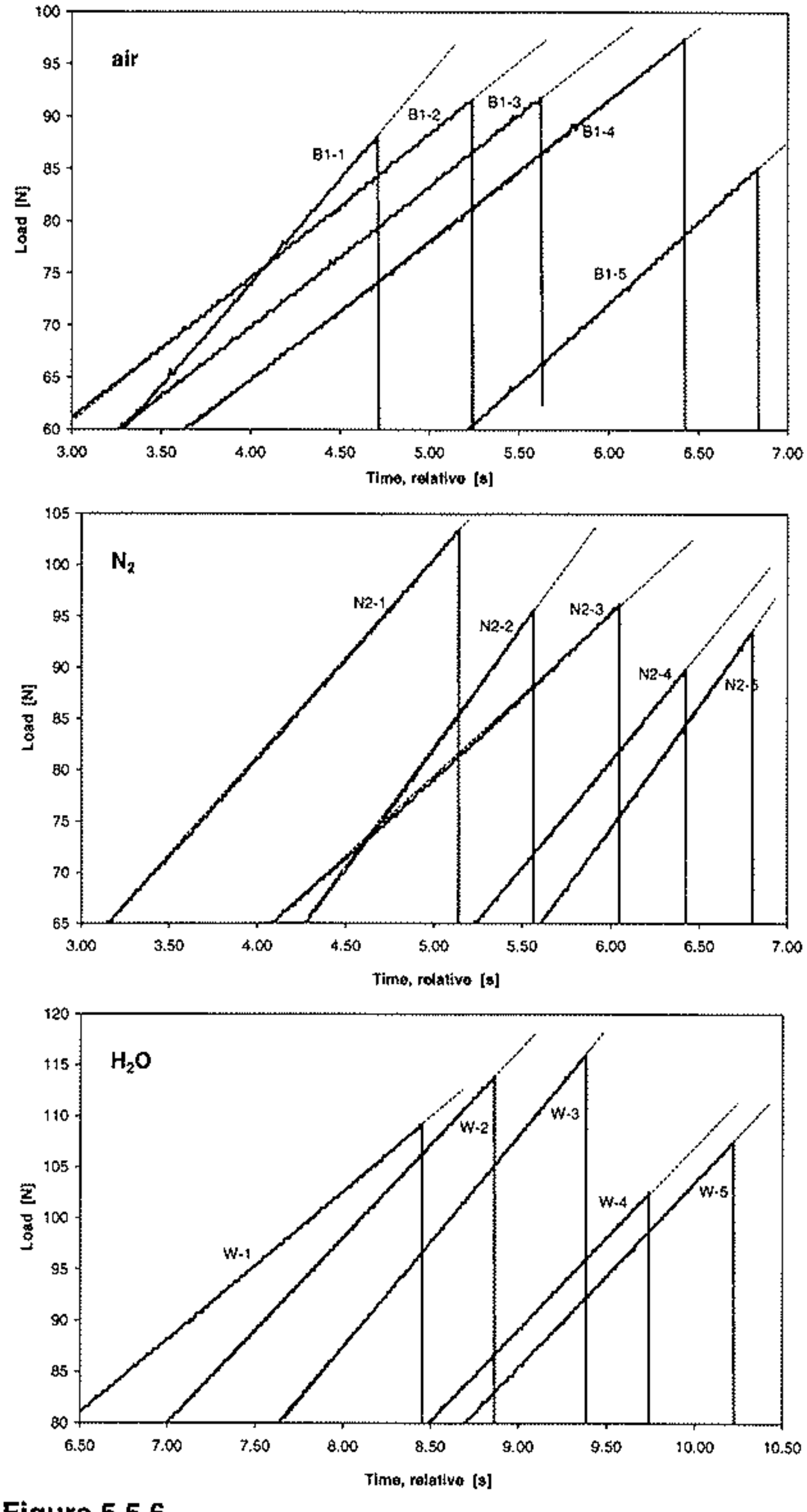


Figure 5.5.6

Digitally recorded load-time curves measured on the alumina-999 in different test media by the round robin organiser. The dashed lines demonstrate the slope of the curves in the last 30 % to 40 %. The following test conditions were used:

air a/W ≈ 0.26 mm, crosshead speed ≈ 0.20 mm/min, notch width ≈ 4 µm, K_{Ic} = 3.54 MPa √m (specimens from the round robin)

N2 a/W ≈ 0.29 mm, crosshead speed ≈ 0.30 mm/min, notch width ≈ 4 µm, K_{Ic} = 4.00 MPa √m (additional specimens used to evaluate the influence of scg)

H_2O a/W ≈ 0.23 mm, crosshead speed ≈ 0.25 mm/min, notch width ≈ 3 µm, K_{Ic} = 3.97 MPa √m (additional specimens used to evaluate the influence of scg)

GPSSN: The fracture toughness measured with the SEVNB method compared very well with values from other methods, as shown in Figure 5.5.7. *(Remark: Participant #3 is not taken into account because he had some obvious problems with the SCF method.)* The toughness measured with the SEPB method compared well with those from the SEVNB method, especially when all specimens of participant #6 are considered and not only the two that were declared valid.

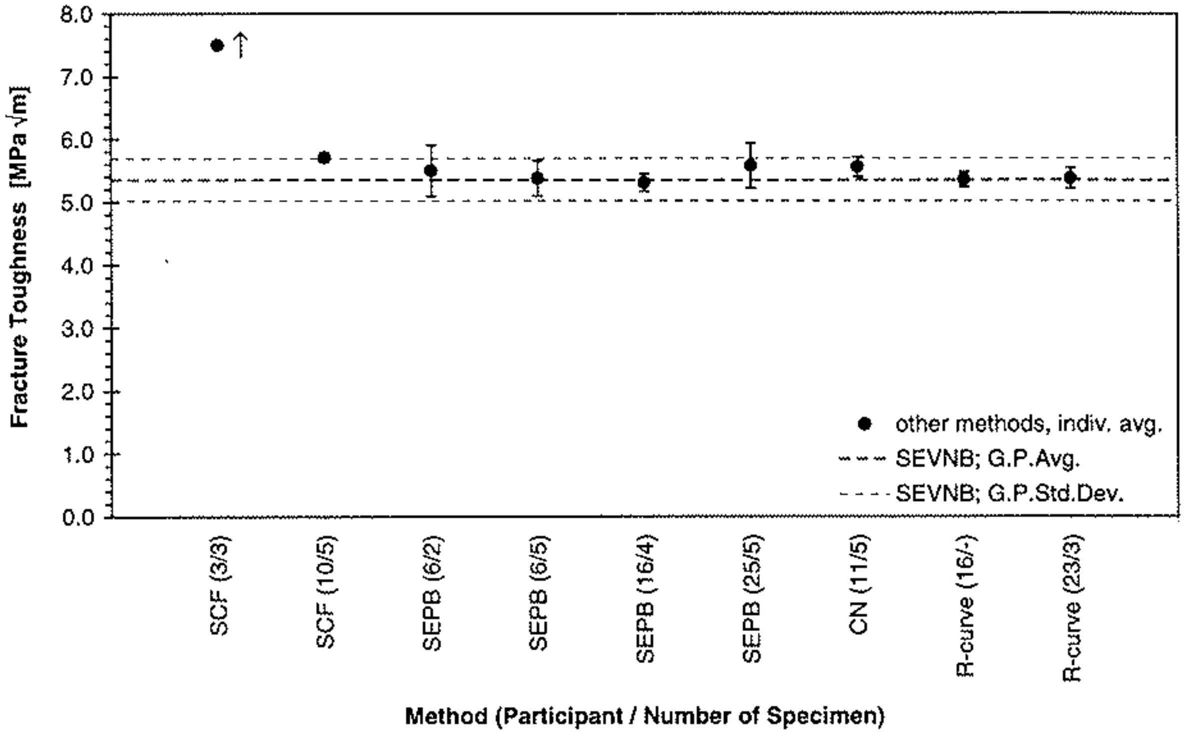


Figure 5.5.7
Comparison of fracture toughness values measured with test methods preferred by participants with values from the SEVNB method. (The R-curve values from participant #16 for the GPSSN were calculated with a crack size 50 μm. The one from participant #23 for the GPSSN is the average calculated for three specimens with a crack size between 75 μm and 105 μm.)

Participant #16 also determined the R-curve for this material (method as described above under "alumina-998") and calculated $K_R = 5.30\ [c]^{0.003}$ MPa √m. Using the smallest crack size observed while determining the R-curve (c ~ 50 μm), the calculated fracture toughness K_R = 5.36 MPa √m compares perfectly with the values from all other methods, including SEVNB. Participant #23 measured the 4-point bend strength on seven bend bars with different indentation sizes to calculate the R-curve. Using only the three specimens with the smallest indentation crack sizes (between 75 μm and 105 μm) an average fracture toughness of 5.38 MPa √m can be computed using the formula $K_{Ic} = 0.88\ (\sigma\ P^{1/3})^{3/4}$ MPa √m (fracture load σ in MPa and indentation load P in N).

As before, the round robin organiser took a closer look at his digitally recorded load-time curves. As can be seen in Figure 5.5.8, the load-time curves do have a linear behaviour in the final part before the failure of the specimen. Therefore, the curves do not give any suspicion of stable crack growth or a pop-in.

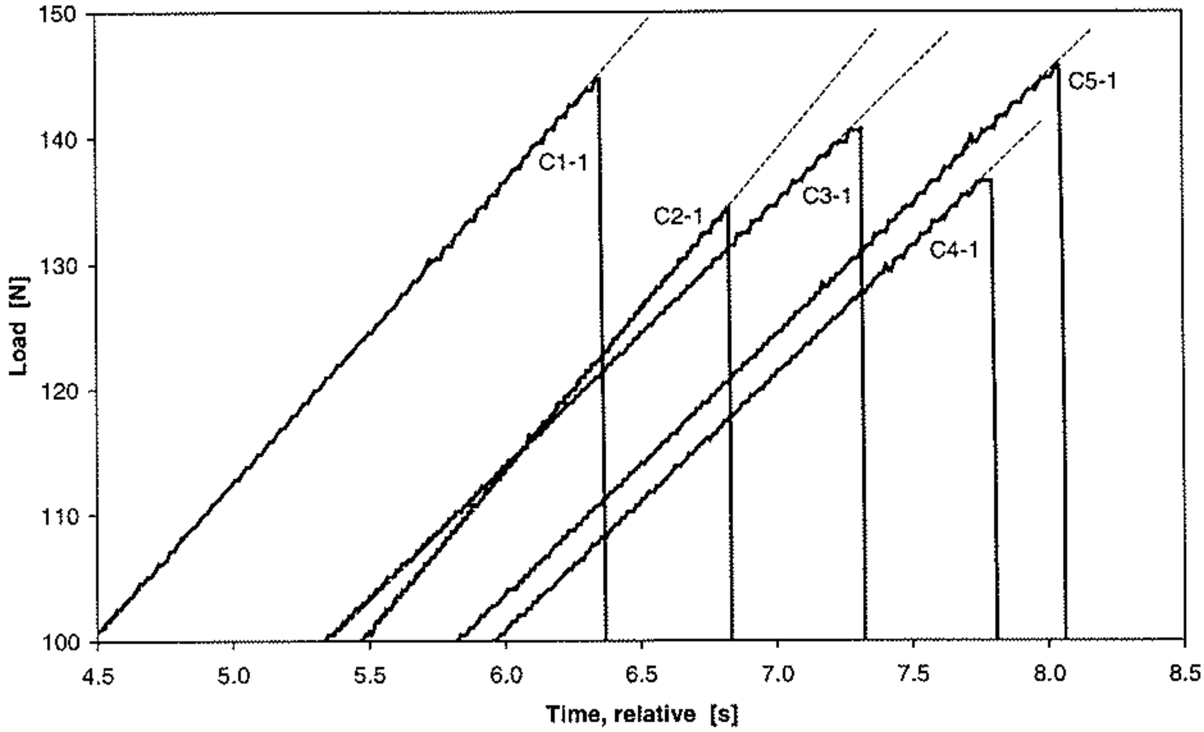


Figure 5.5.8
Digitally recorded load-time curves measured on the GPSSN by the round robin organiser (participant #1). The dashed lines demonstrate the slope of the curves in the last ≈ 30 %. The following test conditions were used: a/W ≈ 0.24 mm, crosshead speed ≈ 0.35 mm/min, notch width ≈ 7 µm, K_{Ic} = 5.23 MPa √m (specimens from the round robin)

SSiC: This SSiC was used before in an ESIS fracture toughness round robin and also in a preliminary study [L-1.5, L-1.10], as mentioned earlier. Therefore, and because only a few specimens were still available for the present round robin, no participant had been asked to conduct additional fracture toughness test with his preferred method. In Figure 5.5.9 all available data from the earlier activities are shown and compared with the results from the SEVNB fracture toughness round robin.

The first three values in Figure 5.5.9 were measured with the well-accepted CN and SEPB test methods. The average from the CN tests is only slightly higher than the SEVNB one, but all tests had been declared invalid in the ESIS round robin because no stable crack growth had been observed. The two SEPB results shown in the figure do not differ too much from each other, but both are lower than the SEVNB one. It might be of interest that the slightly lower SEPB-JFCC value had been measured by a laboratory, which is very familiar with the SEPB method.

The results from the two indentation methods (IF and IS) are higher and lower, respectively, than the SEVNB one. This is not surprising because those two semi-empirical methods should be calibrated for each material.

The SENB-ESIS result had been computed from tests on specimens with a notch width between 60 and 180 µm. Such wide notches will clearly rise the measured fracture toughness values and therefore overestimate the toughness of a material.

Finally, the SEVNB-Lb result was only put into Figure 5.5.9 to demonstrate that the fracture toughness of a ceramic can be measured very reproducible with the SEVNB method, even after years.

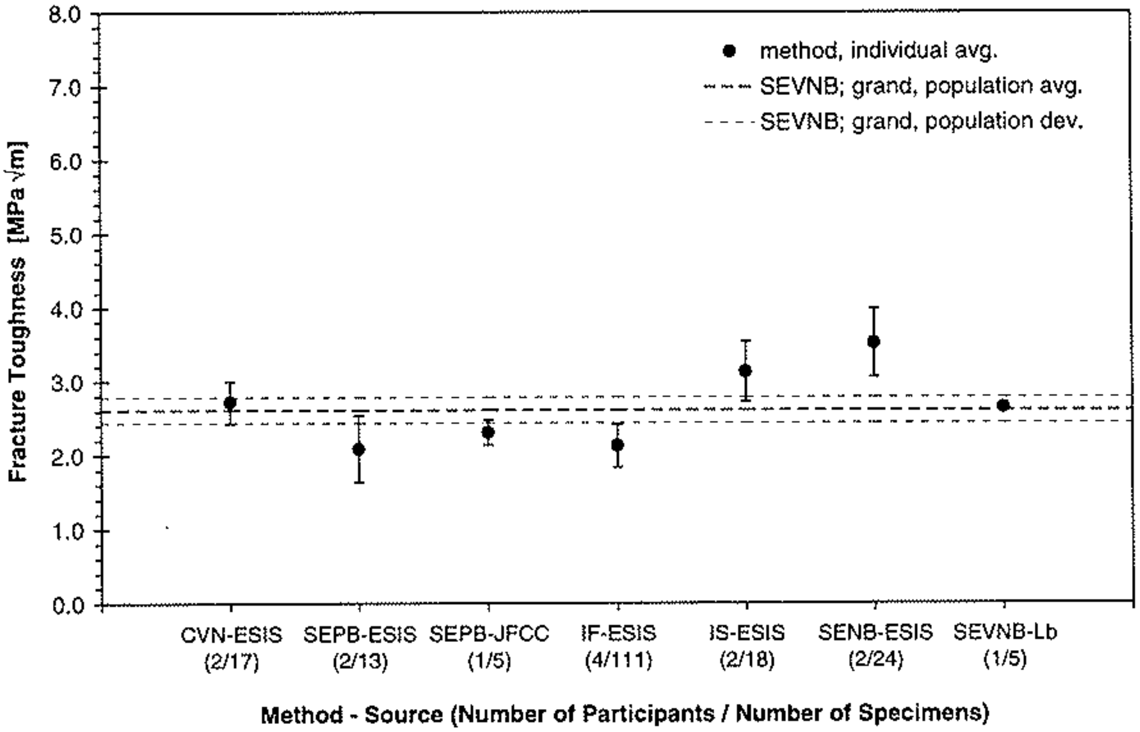


Figure 5.5.9
Comparison of fracture toughness values measured with different test methods on exactly the same SSiC in an earlier ESIS round robin and values from literature.
Source: "ESIS" [L-1.5] ; "SEPB-JFCC" and "SEVNB-Lb" [L-1.10]

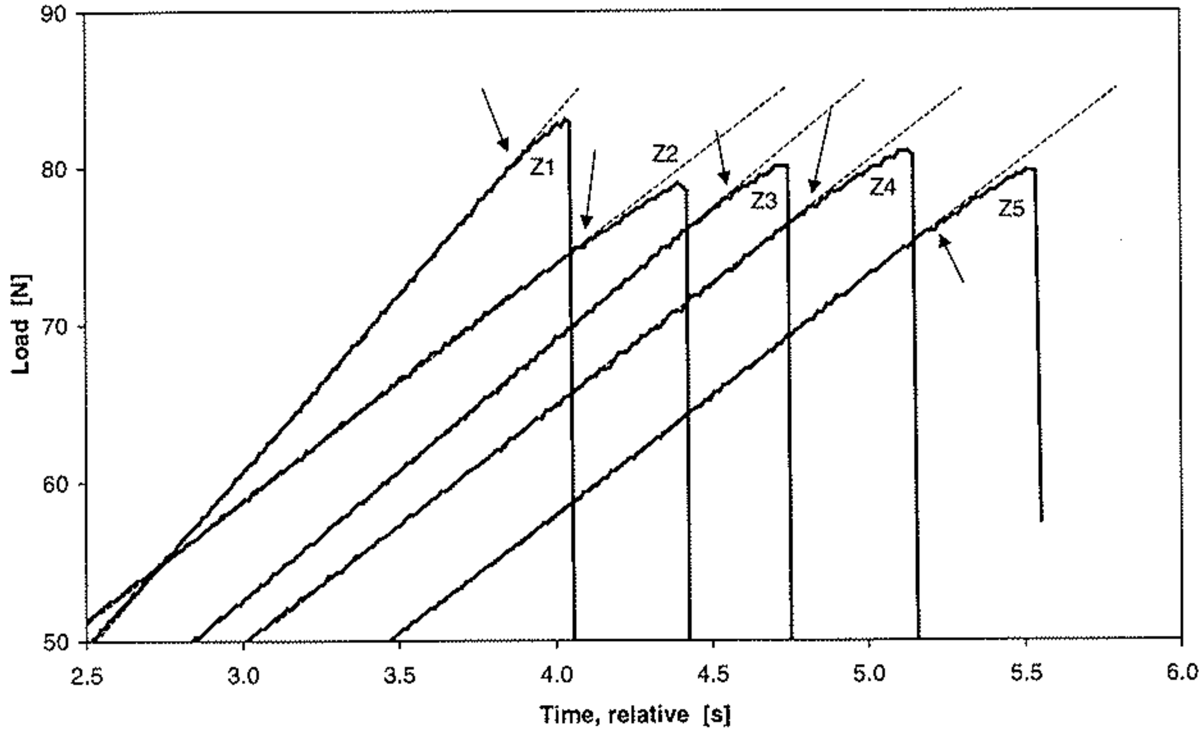


Figure 5.5.10
Digitally recorded load-time curves measured on the Y-TZP by the round robin organiser (participant #1). The dashed lines demonstrate the slope of the curves in the last ≈ 40 % and the arrows mark the area where a first deviation from the straight line is recognisable. The following test conditions were used: a/W ≈ 0.25 mm, crosshead speed ≈ 0.3 mm/min, notch width ≈ 2 μm, K_{Ic} = 4.69 MPa √m (specimens from the round robin)

Y-TZP: The Y-TZP had been used before in a VAMAS fracture toughness round robin and also in a preliminary study [L-1.1, L-1.10], as mentioned earlier. Therefore, and because only a few specimens were still available for the present round robin no participant could be asked to conduct additional fracture toughness test with his preferred method. Why the SCF fracture toughness measured in the earlier round robin was significantly lower than the SEVNB one is discussed under "Y-TZP" in chapter 5.2.

The round robin organiser (participant #1) took again a closer look at his digitally recorded load-time curves and found some indications of stable crack growth, assuming no plastic deformation in the Y-TZP. As can be seen in Figure 5.5.10, the load-time curves do not have a linear behaviour in the final part before the failure of the specimen.

5.6 Comments by participants

All participants were asked to supply comments and opinions to the SEVNB method. Following are fragments from their answers grouped by subject. It might be of interest to notice that some comments and opinions are contradictory. Most questions raised have been discussed above and are not commented further. The abbreviations p#n will help to find the detailed comments and opinions of the participants in Appendix A4.

User-friendliness of the SEVNB method

- quite friendly ... p#3
- without problems ... p#6
- with more experience this would be a relatively fast test method ... p#8
- quite easy and shorter in time than other standard methods ... p#9
- fairly easy and straightforward ... p#10
- reasonably simple ... p#11
- easy to use ... p#12
- most useful method ... p#14
- really nice method and very cheap ... p#15
- play a crucial role ... p#16
- promising, user-friendly technique but needs too much time in specimen polishing.. p#16
- very user-friendly technique ... p#17
- simple, easy, fast, cheap ... p#26
- relatively easy to perform and friendly in using it ... p#28
- very time and cost effective ... p#29
- advantages in its simplicity ... p#32
- promising new method ... simple and relatively fast process ... p#36

V-Notch preparation

- easily be applied with a machine ... p#2
- very good experiences with an equipment similar to that suggested ... p#5
- resetting the razor blade can lead to a small additional notch at the notch root ... p#8
- improvement over the previous SEVNB technique that used a V-shaped diamond wheel ... // ... takes too much time, therefore a parametric study on polishing time as a function of several variables ... p#16
- time consuming, but polishing by machine will reduce the time ... p#19
- poor quality of the razor blades ... p#24
- can be carried out at any laboratory ... p#29
- notch geometry obtained for the last material already appears very good ... // ... long time and high cost to polish by hand, therefore use a machine... p#35
- improvement possible by machine polishing ... p#36

Reproducibility and reliability of the SEVNB method

- own pre-tests show a good reproducibility ... p#2
- reliability is quite good ... p#3
- very reliable ... #12
- reliable ... p#15
- most accurate fracture toughness test method ... p#16
- less Std.Dev. than SEPB ...//... believable values with small Std.Dev. ... p#26
- mean value seems to be reliable ...//... Std.Dev. is very low ... p#28
- reliable data with a small scatter ... p#29
- reliable for any class of materials ... p#30
- lower scatter than SEPB method ... p#32
- reliable determination of the fracture toughness and low Std.Dev. ... p#36

Further use and standardisation of the SEVNB method

- will apply this method also in the future ... p#2
- could be taken into account as possible standard ... p#11
- we are using it as a standard ...//... hope for wide standardisation ... p#15
- excellent technique and will become a standard ...//... in our laboratory we will use the SEVNB for K_{IC} measurement exclusively ... p#17
- method is now standard in our company ... p#24
- would prefer to use this method, but it must be well known by the industry and recommended by standardisation organisations like ISO or CEN ... p#26
- the role of the size and shape of the defects on the toughness values is elucidated before proceeding to claim the method a standard ... p#29
- can become a standard test method and can be recommended for inclusion into the drafts of national and international standards ... p#30
- still unsure whether I would use this method if it became a standard as it would be difficult to compare results between various workers due to notch tip variations ... p#32
- recommend for the standardisation ... p#35

Various

- evidence of an 50-150 µm "initiation" at the root of the notch ... // ... method may not strictly satisfy the strict (idealistic?) requirements of a true fracture toughness test. On the other hand, the results are credible, easily obtained, and the method may be the beneficiary of offsetting errors at the notch root ... // ...scg will affect both SEVNB and SCF, as well as SEPB or other methods ... p#10
- wider range of notch depth for more flexibility ... // ... notch widths of > $10x10^{-3}$mm is almost equivalent to a sharp precrack ... // ... does the SEVNB technique tend to give some underestimated fracture toughness? ... p#16
- applicability to nearly every ceramic, but not to super-fine grained ceramics ... p#17
- testing time should not exceed 1 s with respect to scg ... p#22
- quite useful to obtain the initial fracture toughness ... p#25
- lower K_{IC} as it was expected for small V-notch tips ... p#26
- grain pullouts or inherent defects below the surface where the fracture origins ... p#29
- results are clearly above crack tip toughness ... p#32

6 Conclusions

1. Very consistent results were obtained for the **coarse-grained alumina-998**. The fracture toughness for the 135 tests accepted from 28 participants was 3.57 ± 0.22 MPa √m (mean, standard deviation). The coefficients of variation for the repeatability (within-lab) and reproducibility (between-lab) are only 4.6 % and 6.1 %, respectively. The mean is significantly lower than results from other credible test methods. A combination of a high sensitivity to sub critical crack growth near the V-notch tip, stable crack growth and a pop-in of small cracks to form a crack "initiation" region might be responsible for the discrepancy.

2. Reasonably consistent results were obtained for the **fine-grained alumina-999**. The fracture toughness for the 102 tests accepted from 21 participants was 3.74 ± 0.40 MPa √m. The coefficients of variation for the repeatability and reproducibility are 6.2 % and 10.7 %, respectively, and are comparable with coefficients typically computed for the SEPB method. The reason for the rather high coefficient of variation for the reproducibility seems to be a combination of material inhomogeneity (clusters of large grains) and the acceptance of results from specimens having a too wide notch in relation to the grain size. Subcritical crack growth might have influenced the repeatability and reproducibility too. The mean is comparable with results from other credible test methods.

3. Consistent results were obtained for the **gas pressure sintered silicon nitride**. The fracture toughness for the 129 tests accepted from 27 participants was 5.36 ± 0.34 MPa √m. The coefficients of variation for the repeatability and reproducibility are 5.3 % and 6.3 %, respectively, and are equal or better than those computed for the SCF method on two comparable silicon nitrides in an earlier round robin. The mean compares very well with results from other credible test methods.

4. Very consistent results were obtained for the **sintered silicon carbide**. The fracture toughness for the 56 tests accepted from 12 participants was 2.61 ± 0.18 MPa √m. The coefficients of variation for the repeatability and reproducibility are only 4.5 % and 6.8 %. The mean compares very well with results from an earlier round robin and values from the literature.

5. As predicted, less consistent results were obtained for the **yttria–stabilised tetragonal zirconia polycrystal** due to its grain size in the submicron range. Therefore, the very fine grained ceramic showed the limitations of the method with respect to the grain size or major microstructural feature size, respectively. The fracture toughness for the 35 tests accepted from 7 participants was 5.34 ± 0.65 MPa √m. The coefficients of variation for the repeatability and reproducibility are 6.2 % and 12.7 %, respectively.

6. If the **V-notch width** is less than 10 µm "good" fracture toughness values can be measured for ceramics with an average grain size or major microstructural feature size of greater about 1 µm. This rule of thumb is based on the two aluminas, the GPSSN, SSiC and Y-TZP tested.

7. The **V-notch depth** has no influence on the measured fracture toughness over a very wide range. Therefore, the same depths stipulated for the SEPB method by ASTM and JIS, respectively, can be used with the SEVNB method.

8. Participants who were unfamiliar with the method had in general **no difficulty**. Some participants who polished the V-notches by hand felt that the method required to much time, therefore the use of a machine is recommended.

9. The SEVNB method proved to be **forgiving and robust** with respect to the notch preparation, notch depth or optical notch quality.

10. Participants rated the method **user-friendly,** easy and cheap to conduct, reliable and accurate. Many participants will continue to use the SEVNB method and would therefore be interested in a SEVNB standard.

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- Mr. George Quinn, Chairman VAMAS TWA# 3, NIST, USA-Gaithersburg
- Dr. Wolfhart Rieger, Metoxit, CH-Thayngen
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- Dr. Edith Rudolph, BAM Berlin, D-Berlin
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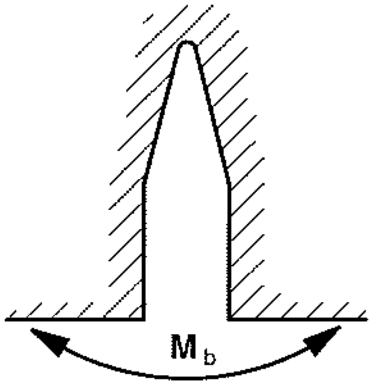
VAMAS TWP#3 / ESIS TC6 ROUND ROBIN
ON FRACTURE TOUGHNESS OF CERAMICS USING THE

SEVNB METHOD

(SINGLE-EDGE-V-NOTCHED BEAM)



INSTRUCTIONS

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Content

Remark:
The following parts of the original INSTRUCTIONS dated May 20, 1997 were improved on August 28, 1998 and September 8, 1999:
- *Page 1:* *Address*
- *Page 4:* *Table 2.1, Table 2.2*
- *Page 5:* *Table 2.4, Table 2.5*
- *Page 12:* *Figure 9*

1. INTRODUCTION

Many methods are currently used to measure the fracture toughness (K_{Ic}) of ceramic materials. Methods based on a widely accepted theory 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 demonstrated in a Versailles Project of Advanced Materials and Standards (VAMAS) round robin test report [1] that accurate K_{Ic}-values can be measured with the SCF method. However, they also showed that making the necessary small cracks and finding their crack front after the test is performed can be challenging, if not impossible. The CN test is simple to conduct, but Himsolt, Munz and Fett stated that the generation of a sharp crack cannot be ensured in all tests [2]. Salem, Shannon and Jenkins mentioned that K_{Ic}-values measured with the CN method depend on the loading rate [3]. Further, they reported that for materials with a rising R-curve behaviour, the specimen proportions and notch geometry can also influence the result. Nishida, Hanaki and Pezzotti concluded that practical problems with the SEPB method make the K_{Ic}-determination difficult to apply and even unsuitable for some ceramics [4]. The simple and inexpensive SENB method, on the other hand, can be influenced by the notch width, as for example Primas and Gstrein recently reported in a European Structural Integrity Society (ESIS) round robin test report [5]. From the report it seems that for most of today's ceramics, a notch width of 20 µm or less is necessary to conduct valid tests. Until recently, it was almost impossible to cut such fine notches. Nishida, Hanaki and Pezzotti introduced an interesting technique to taper a saw cut to a sharp V-Notch using a razor blade sprinkled with diamond paste [4]. This method which is known as the SEVNB method, in order to distinguish it from the SENB method, relates to basic work conducted by Awaji and Sakaida [6].

The aim of this round robin is to examine whether the method is user-friendly, reliable and most importantly, how well the results compare with other recognised methods and if the SEVNB method is a potential standard test method.

Remark:
Just recently an old East German patent [7] explaining how to polish sharp V-Notches into bend bars with a razor blade and diamond past was brought to the attention of the round robin organiser.

2. MATERIALS

In the round robin, five ceramic materials with varying difficulties for measuring the fracture toughness will be used. Each participant is required to test the alumina-998 (Material A: Al_2O_3 - 998) and the silicon nitride (Material C: Si_3N_4). The second alumina (Material B: Al_2O_3 - 999), the silicon carbide (Material D: SSiC) and the yttria-stabilised tetragonal zirconia (Material E: Y-TZP) are optional. Only participants who respond to in the QUESTIONNAIRE that they would like to test additional ceramics will receive specimens made from material B, D and E.

2.1 MATERIAL A: Al_2O_3 - 998

Powder purity	> 99.8 %
Processing	Metoxit, CH-Thayngen
cold pressed	isostatic
sintered	oxidising atmosphere
Form and size	bar, ~ 4 x 5 x 45 mm
Avg. grain size	>10 µm
Density	3.86 g/cm³
Strength	342 MPa in 4-point bend testing at room temperature

2.2 MATERIAL B: Al_2O_3 - 999

Powder purity	> 99.9 %
Processing	Metoxit, CH-Thayngen
cold pressed	isostatic
sintered	oxidising atmosphere
post hipped	gas-pressure
Form and size	rod, ~ dia. 40 mm and length ~ 50 mm
Avg. grain size	1.3 µm
Density	3.97 g/cm³
Strength	350 MPa in 4-point bend testing at room temperature

2.3 MATERIAL C: Si_3N_4

Powder type	N 3208
Processing	Bayer-CFI, D-Rödental
Form and size	plate ~ 53 x 47 x 5 mm
Density	3.23 g/cm³
Porosity	< 1 %
Avg. grain size	< 1 µm with elongated grains
Strength	> 920 MPa in 4-point bend testing at room temperature

2.4 MATERIAL D: SSiC

Powder	unknown
Processing	Hutschenreuther, Germany
Avg. grain size	7 µm
Density	3.15 g/cm^3

Material D was previously used in an ESIS TC6 round robin on fracture toughness. The SSiC is described in detail in a ESIS TC6 test report [5].

2.5 MATERIAL E: Y-TZP

Powder	TOSOH TZ-3Y (3 mole-% yttria-stabilised tetragonal zirconia)
Processing	Metoxit, CH-Thayngen
cold pressed	isostatic
sintered	oxidising atmosphere
post hipped	gas-pressure
Form and size	rod, dia. ~ 50 mm and length ~ 200 mm
Avg. grain size	0.45 µm
Density	6.03 g/cm^3
Strength	> 750 MPa in 4-point bend testing at room temperature

Material E was previously used in a VAMAS TWA#3 round robin on fracture toughness (SCF-method). The Y-TZP is described in detail in a VAMAS test report [1].

3. SPECIMENS

3.1 PACKAGE

Each participant will receive a package containing:
- 5 specimens each from Material A and C on which the fracture toughness is to be measured using the SEVNB method.
- 5 specimens each from the optional ceramics (Materials B, D, and E) on which the fracture toughness is to be measured using the SEVNB method.
- additional 5 specimens each from Material A and C (and B, if chosen) on which the fracture toughness is to be measured using the participants preferred method (SCF, SEPB, CN, ...).
- 2 extra specimens of each material (or a similar material) which will be tested using the SEVNB method. These extra specimens are required only for sample preparation and will not be tested. These extra specimens are hereafter called "dummies" or "dummy specimens". The dummies may vary in length from the test specimens.
- razor blade sample. This razor blade should not be used for preparing the specimens. Participants should supply there own razor blades.
- report form (PROTOCOL: SEVNB Method).

Not included in the package but required for specimen preparation:
- holder (participants are free to choose the material, design and dimension).
- diamond paste.
- glue or resin.
- heavy tape or razor blade holder.

3.2 SHAPE AND DIMENSIONS

The specimens that will be distributed are of prism shape with a rectangular cross section. Figure 1 shows the shape and dimension of the specimens.

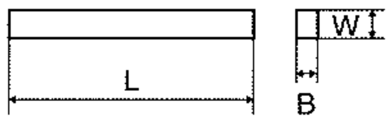
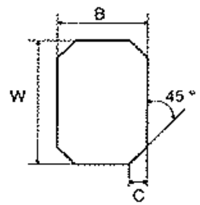


Figure 1:

Dimensions of Specimen
Length L = 45 ... 50 mm
Width W = 4 mm
Thickness B = 3 mm



Enlarged cross section.

Chamfer C = 0.1 ... 0.2 mm

Note:
- *Some specimens prepared of Material A and all prepared of Material D are not chamfered.*

3.3 SPECIMEN PREPARATION

If you have no experience polishing sharp V-Notches used with the SEVNB method **it is highly recommended that you first try out the technique and equipment** with surplus specimens from your lab.

3.3.1 Polishing by Hand

Step H1: Prepare a holder for your diamond saw to mount 5 specimens and 2 dummy specimens, as shown in Figure 2. The dummies are used only to protect the specimens during sawing and polishing the notch.

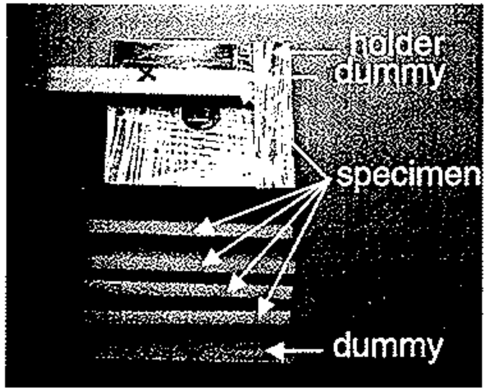


Figure 2:
Holder, specimens and dummy specimens (marked with an X).

Step H2: Mount 5 specimens and 2 dummy specimens with glue or resin on the holder as shown in Figure 3a and 3b. One 3 mm wide side of the specimens must face the holder. (This side will be in compression during the fracture test). Mount specimens and dummies as close together as possible. Draw a pencil line across the middle of the specimens, as shown in Figure 3a. The notch will be sawed and polished along this pencil line.

Note:
- *Avoid bending the specimen while mounting to the holder.*

Step H3: Mount the holder on a diamond saw. Saw a starter notch along the pencil line. The notch should have the same depth of about 0.5 to 0.6 mm over its entire length. Figure 4 shows the sawing set-up. After sawing, clean the holder, specimens and especially the notch.

Note:
- *The width of the diamond saw blade should be about the same or only a little larger than the width of the razor blade used. Otherwise the razor blade might skate over the surface of the pre-sawn notch and it might be difficult to start polishing the V-Notch. V-shaping the saw blade might help if only saw blades with a to large width are available.*

- *Razor blades thinner than about 0.2 mm might not be stiff enough by themselves for this task. If the razor blades are not stiff enough glue or screw the blades between two steel plates.*
- *Use a razor blade with a tip angle $\beta < 30°$, or as small as possible (see Figure 9).*

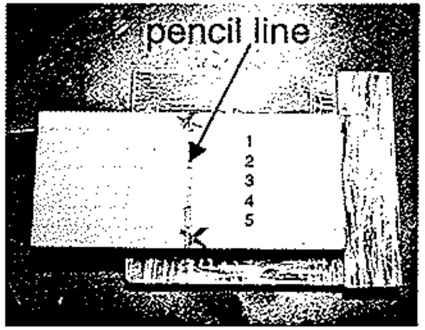


Figure 3a
Holder with specimens and dummy specimens.

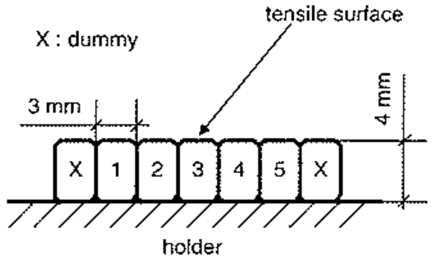


Figure 3b
Cross section along pencil line.

Step H4: Fix the holder in a vice. Fill the starter notch with a diamond paste as shown in Figure 5. The diamond past should have a maximum grain size between 3 and 6 µm.

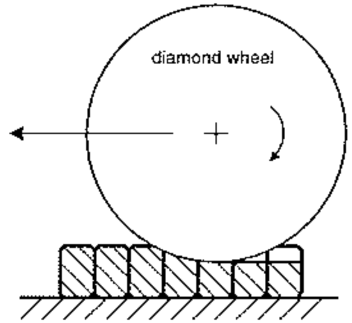


Figure 4:
Sawing the starter notch.

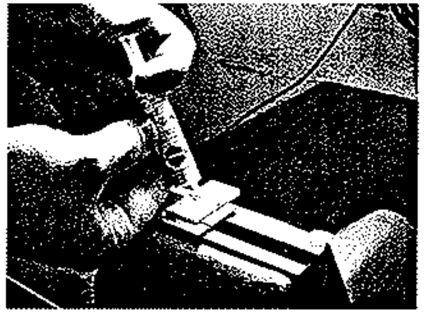


Figure 5:
Filling the notch with diamond paste.

Step H5: To protect your fingers, put the razor blade into a holder or apply a heavy self-adhesive tape as shown in Figure 6. Put the razor blade into the starter notch and apply a light force. Polish the V-Notch with the razor blade with a gentle back-and-forth motion (white arrows in Figure 6). Move the razor blade as straight as possible. Control the depth of the V-Notch periodically with an optical microscope (approx. every 10 min.) on both ends of the V-Notch. Do not remove the samples from the specimen holder, if possible. If not, remount the specimens on the holder with a razor blade inserted in the V-Notch. If the total V-Notch depth is less than 0.8 mm continue to polish. The total final V-Notch depth should be between 0.8 mm and 1.2 mm.

Note:
- *Polishing the V-Notch by hand into alumina specimens (for example Material A) will take about 30 minutes, and into silicon nitride specimens (for example Material C) about 4 hours. If your razor blade jerks while polishing the V-Notch, put an oil drop into the notch.*
- *Polishing by hand with a 4 μm diamond past produced a V-Notch width S of about 20 μm (see Figure 10). Using a diamond past finer than 4 μm for example a 1 μm diamond past did not decrease the width further in the coarse grained alumina (Material A). If the V-Notch width S is lager than about 30 μm use a new razor blade or different razor blade brand.*
- *Cleaning of the V-Notch might be difficult. For example, if a finer diamond past is used at the end of the polishing (3 μm diamond past after starting with 6 μm diamond past) in order to get a smaller V-Notch, different steps might be necessary to flush and scrape out the diamonds.*
- *In coarse grained materials large grains might pop out into the V-Notch during polishing and limit the ability to get a sharp V-Notch.*
- *Putting a force of about 5 N to 10 N on the razor blade while polishing will be sufficient.*

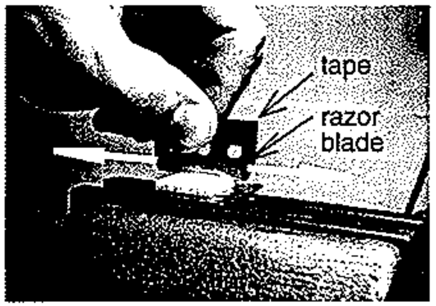


Figure 6:
Polishing out the V-Notch.

Step H6: Remove the specimens from the holder. Clean the specimens with acetone in a small powerful ultrasonic bath. Be careful not to damage the specimens while cleaning. Dry the specimens.

Note:
- *Avoid bending the specimen while removing from the holder (respectively remounting to the holder, if necessary).*

3.3.2 Polishing by Machine

If you have experience polishing V-Notches and also a machine to do it, then you should use your own procedure. An example of a polishing machine is shown in Figure 7a and 7b. Report your procedure in detail.

Note:
- *Polishing by machine with a 1 μm diamond past produced a V-Notch width S (see Figure 10) of about 2 μm in the fine grained Y-TZP (Material E). Using a diamond past finer than 1 μm for example a 0.5 μm diamond past did not decrease the width further.*

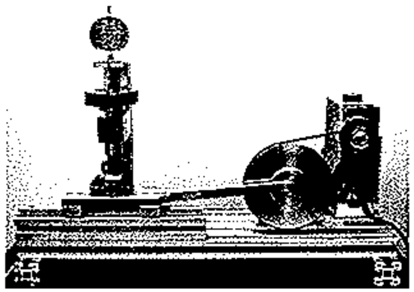


Figure 7a:
Simple machine for polishing out the V-Notches.

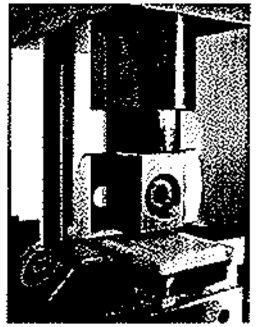


Figure 7b:
Vice with specimens and razor blade.

3.3.3 Preferred Method of Participant

Prepare the specimens as required for your preferred method (SCF, SEPB, CN, ...). Report the standard used for the preparation. If no standard was used, describe the preparation. Report all necessary information.

4. TEST PROCEDURE

4.1 TEST MACHINE

A testing machine capable of keeping a uniform crosshead speed should be used. Further, the machine should be capable of measuring the true load at rupture of the specimen to better than ± 1 %. Report the accuracy of your testing machine and the date of the last load calibration.

4.2 TEST JIG (FOUR-POINT FLEXURE)

The specimen should be supported on two bearing edges perpendicular to its length. The outer support bearing edges shall be parallel rollers of diameter 5 mm ± 0.2 mm and shall be capable of rolling outward on flat support surfaces. One of the rollers shall additionally be capable of rotating about an axis parallel to the length of the specimen such that torsional loading is minimised. The two rollers shall be positioned initially with their centres 40 mm ± 0.5 mm apart with their axes parallel to within 1 °. The separation of the centres of the rollers in their starting positions shall be measured to the nearest 0.1 mm with a travelling microscope. The rollers shall be made from hardened steel or other hard material with a hardness greater than 40 HRC (Rockwell C-scale). The rollers shall have a smooth burr-free surface finish with roughness less than 0.5 µm Ra, and shall have a diameter uniform to ± 0.02 mm.

The two loading rollers are located at the quarter points, with an inner span of 20 mm ± 0.2 mm and are free to roll inwards. The rollers are free to rotate separately about an axis parallel to the length of the specimen to allow alignment. The distances between the rollers shall be measured to the nearest 0.1 mm along the length of the specimen perpendicular to the direction of loading, using a travelling microscope or other suitable device. The loading rollers shall be symmetrically positioned to within ± 0.1 mm. The arrangement for loading shall ensure that equal forces are applied to the two loading rollers.

The above definition is copied from EN 843-1: Determination of flexural strength. Figure 8 shows a four-point flexure test jig. Any deviation from this test jig configuration must be reported.

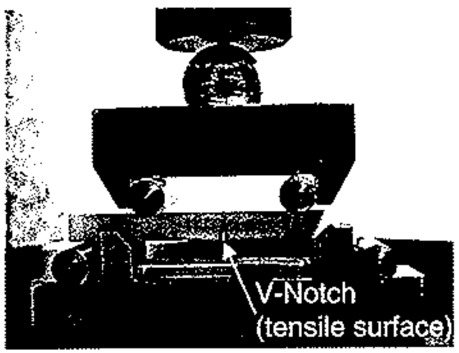


Figure 8:
Four-point flexure test jig in accordance with EN 843-1: Determination of flexural strength.

4.3 TEST PROCEDURE SEVNB METHOD

Test all 5 specimen for each material supplied.

Step 1: Calibrate the magnification of the microscope and photo equipment with a stage micrometer. Select 2 of the 5 specimens for an optical check. Photograph the V-Notch on one side of each of the selected specimens using a magnification ≥ 50 X. Control the V-Notch geometry with the help of the photograph. Report any deviation from the geometry shown in Figure 9. Return copies of the photographs.

Photograph the V-Notch tip on the same two specimens with a magnification ≥ 300 X (better ≥ 1000 X). Measure the V-Notch angle and width in accordance with Figures 9 and 10. Report the V-Notch angle and width. Return copies of the two photographs.

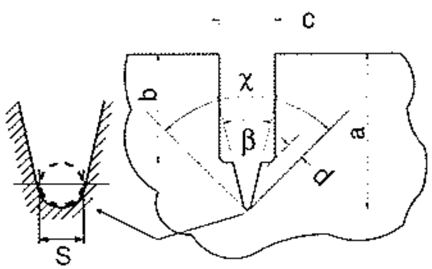


Figure 9:
a = 0.8 mm ... 1.2 mm
b = 0.5 mm ... 0.6 mm
c ≥ width of razor blade
d ≥ 0 mm
χ ≤ 90°
β ~ 30°, or as small as possible
S = V-notch width

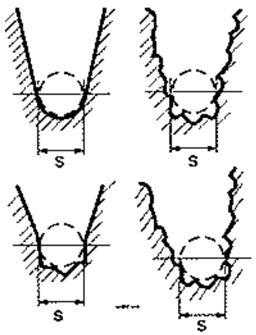


Figure 10:
Schematic examples of V-Notch tips and how to measure the V-Notch width S.

Step 2: Measure thickness B and width W (refer to Figure 1) of each specimen using micrometer callipers. Read each value to an accuracy of three decimal places.

Step 3: Place the 3 mm width face with the V-Notch down as shown in Figure 8. Load the specimen with a crosshead speed of 0.5 mm/min. Record the fracture load (maximum load) to three significant figures. The tests should be performed in air at room temperature. Report the temperature and relative humidity (%-RH) during the tests.

Step 4: The depth of the V-Notches is measured by observing the fracture surface (Figure 11) using the calibrated microscope with a magnification ≥ 50 X. Read the depths a1, a2 and a3 to three significant figures. Check if the fracture started at the bottom of the V-Notch over its entire length, if not report. Return both halves of the two specimens photographed in step 1.

Note:

- *With white material it might be difficult to measure the V-Notch depth. To highlight the broken edges different techniques could be used. For example:*
  - *fibre optic light-source (grazing incidence illumination) almost in the same plane than the broken surface.*
  - *dye the V-Notch before fracture .*

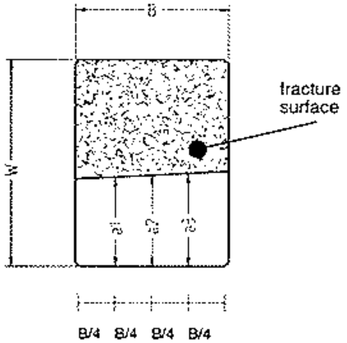


Figure 11:
Measurement of V-Notch depth a_1, a_2, a_3.

4.4 PREFERRED METHOD OF PARTICIPANT

Report the testing standard you used for your preferred method. If no standard was used, report the machine, equipment and explain the procedure. Report all measurements (dimension, load at fracture, etc.) used to compute the fracture toughness. Explain the measurements with diagrams and schematic drawings.

5. ANALYSIS

5.1 SEVNB METHOD

The average depth of the V-Notch is calculated using equation 1. The V-Notch depth (a) shall be between 0.8 mm and 1.2 mm and satisfy equation 2. The relative initial V-Notch depth is calculated using equation 3.

$$a = (a_1 + a_2 + a_3) / 3 \qquad (1)$$

$$(a_{max} - a_{min}) / a \le 0.1 \qquad (2)$$

$$\alpha = a / W \qquad (3)$$

In the case of **4-point flexure with spans of 40 and 20 mm,** compute the fracture toughness K_{Ic} using the formulas (3), (4) and (5) [8], [9]:

$$K_{Ic} = \sigma\sqrt{a}Y = \frac{F}{B\sqrt{W}} \cdot \frac{S_1 - S_2}{W} \cdot \frac{3\sqrt{\alpha}}{2(1-\alpha)^{1.5}} Y \qquad (4)$$

with: $$Y = 1.9887 - 1.326\alpha - \frac{(3.49 - 0.68\alpha + 1.35\alpha^2)\alpha(1-\alpha)}{(1+\alpha)^2} \qquad (5)$$

If for any reason you tested the specimens in **3-point flexure with a span of 30 mm,** compute the fracture toughness K_{Ic} using the formulas (3), (6) and (7) [10]:

$$K_{Ic} = \frac{F \cdot S}{B \cdot W^{1.5}} \cdot \frac{3\sqrt{\alpha}}{2} Y^* \qquad (6)$$

with: $$Y^* = 1.964 - 2.837\alpha + 13.71\alpha^2 - 23.25\alpha^3 + 24.13\alpha^4 \qquad (7)$$

If for any reason you tested the specimens in **3-point flexure with a span of 40 mm,** compute the fracture toughness K_{Ic} using the formula (3), (6) and (8) [11]:

with: $$Y^* = 1.972 - 2.746\alpha + 13.44\alpha^2 - 22.84\alpha^3 + 23.86\alpha^4 \qquad (8)$$

where:

K_{Ic}	fracture toughness	(MPa √m)
σ	fracture strength	
F	fracture load	(MN)
B	specimen thickness	(m)
W	specimen width	(m)
S, S_1, S_2	support span ($S_1 > S_2$)	(m)
a	average V-Notch length	(m)
a_{max}	maximum among a_1, a_2, a_3	(m)
a_{min}	minimum among a_1, a_2, a_3	(m)

α	relative V-Notch depth	(-)
Y, Y*	stress intensity shape factors	(-)

Perform all calculations to three significant figures. Calculate the average K_{Ic} value, and round the result to two decimal places.

Note:
If using equations (3), (4) and (5) to compute the fracture toughness K_{Ic}, control the calculation with the following values:

$F = 100 \cdot 10^{-6}$ MN
$B = 3 \cdot 10^{-3}$ m $W = 4 \cdot 10^{-3}$ m $a = 1 \cdot 10^{-3}$ m
$S_1 = 40 \cdot 10^{-3}$ m $S_2 = 20 \cdot 10^{-3}$ m
Result: $K_{Ic} = 3.80$ MPa $\sqrt{m}$

5.2 PREFERRED METHOD OF PARTICIPANT

Report the standard you used to compute the fracture toughness. If no standard was used, report the procedure of analysis.

6. REPORT

For each material you test with the **SEVNB method** use a separate data sheet. Copy the supplied sheet as many times as needed. After finishing the tests and computing the fracture toughness return the following for each material tested;
- report form (PROTOCOL);
- a detailed description of the procedure used to notch the SEVNB specimens if different to that described in "3.3 Specimen Preparation" or;
- a detailed description of the machine and procedure used to polish the V-Notches.
- the photographs <u>and</u> specimens used to control the V-Notch geometry and measure the V-Notch width (see "4.3 Test Procedure");
- a schematic drawing showing the dimensions and function of the test jig if different from the one described in "4.2 Test Jig (Four-Point Flexure)";
- a separate sheet with remarks, comments, recommendations, tips & tricks, etc. to the SEVNB method and round robin exercise;
- your opinion about the user-friendliness and reliability of the SEVNB method and whether you would use the SEVNB method if it became a standard test method.

For each material you tested with **your preferred method**, fill out a separate data sheet (use your own data sheet or test protocol). After finishing the tests and computing the fracture toughness return the following for each material tested;
- report form (PROTOCOL);
- a detailed description of the procedure used to prepare the specimens;
- a description of all equipment used for testing, with schematic drawings and photos.

Return everything by December 31, 1997 to the organiser.

REFERENCES

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11. F. Wakai, S. Sakaguchi, Y. Matsuno, "Calculation of Stress Intensity Factors for SENB Specimens by Boundary Collocation Procedure", Yogyo-kyokai-shi, Vol. 93, No.8, pp 479-80, 1985

Appendix A2: Individual Fracture Toughness Results

The following table lists the fracture toughness values from the individual participants that were accepted and used to compute the grand population average and standard deviation. Included are also the corresponding notch widths (the participants had to measure the notch width on at least two specimens from each material they tested). In the table the notch width of the specimens were rounded to zero decimal places. The following abbreviations are used:

- no specimens distributed to the participant
n.a. result not accepted by the round robin organiser
** declared "not valid" by participant or specimen broken during preparation

Following the table Figures A2.1 to 5 show frequency histograms for all accepted results including stragglers and outliers. The same horizontal axis is used and the interval size is 0.1 MPa √m.

Table A2.1: All accepted fracture toughness values

	Material A Al_2O_3 - 998		**Material B** Al_2O_3 - 999		**Material C** Si_3N_4		**Material D** SSiC		**Material E** Y-TZP	
Participant	Notch width µm	K_{IC} MPa √m	Notch width µm	K_{IC} MPa √m	Notch width µm	K_{IC} MPa √m	Notch width µm	K_{IC} MPa √m	Notch width µm	K_{IC} MPa √m
1	23	3.66	3	3.44	8	5.46	20	2.55	2	4.83
	23	3.65	3	3.58	5	5.02	18	2.50	2	4.59
	23	3.80	4	3.57	7	5.21	14	2.60	2	4.68
	20	3.61	3	3.79	6	5.05	18	2.74	2	4.70
	20	3.64	4	3.33	6	5.43	17	2.71	2	4.64
2		3.64	35	n.a.	17	5.31		-		-
		3.61	28	n.a.	14	5.70		-		-
	8	3.63	28	n.a.		5.34		-		-
		3.49	30	n.a.	20	5.38		-		-
	9	3.70	30	n.a.	14	5.70		-		-
3	46	n.a.	45	n.a.	57	n.a.		-		-
		n.a.		n.a.		n.a.		-		-
		n.a.		n.a.		n.a.		-		-
	46	n.a.	55	n.a.	46	n.a.		-		-
		n.a.		n.a.		n.a.		-		-
4	13	3.49	11	3.62	7	5.45	4	2.80	8	4.98
	14	3.19	4	3.28	4	5.20	9	2.48	9	4.99
	9	3.61	9	3.58	5	5.62	10	2.81	7	5.21
	11	3.44	10	3.77	5	5.52	5	2.67	8	5.01
	9	3.52	9	3.31	6	5.60	7	2.61	7	4.95
5	14	3.33		3.60	9	5.00		2.38		-
		3.69	9	3.33	9	5.29		2.44		-
		3.27		3.72		4.76	7	2.41		-
	13	3.41	11	3.53		5.10		2.41		-
		3.40		3.59		5.38	12	2.39		-
6	30	3.70		3.24	5	4.72	6	2.57		5.59
	28	3.95	6	3.39		5.17	6	2.50	15	5.15
	23	3.43	8	3.82	4	4.65		2.45		5.35
	23	3.71		3.45		4.95		2.56		5.29
	26	3.59		3.43		5.11		2.39	16	4.97

7		3.44	35	n.a.	58	n.a.	19	**		-
	28	3.61		n.a.		**		2.26		-
		3.56		n.a.	8	4.94		2.25		-
		**	40	n.a.		5.31	33	2.09		-
	21	3.53		n.a.		4.78		2.45		-
8	24	3.36	26	4.26	24	6.06		-		-
		**	23	4.49	24	4.92		-		-
	32	3.49	28	4.51	20	4.71		-		-
	28	3.35	28	3.69	23	5.77		-		-
	30	3.45	28	3.70	24	5.71		-		-
9	160	n.a.	38	n.a.	24	n.a.		-		-
	165	n.a.	33	n.a.	38	n.a.		-		-
	160	n.a.	47	n.a.	56	n.a.		-		-
	170	n.a.	19	n.a.		**		-		-
	207	n.a.		**	38	n.a.		-		-
10	11	3.56	9	3.81		5.11		-		-
		3.52		3.34	9	4.79		-		-
		3.40	9	3.43		5.64		-		-
	11	3.59		3.34		5.39		-		-
		3.62		3.34	11	5.40		-		-
11	24	3.59	14	3.45	18	5.97		-	12	6.12
	20	3.68	12	3.43	18	5.58		-	12	6.22
	20	3.58	14	3.41	14	5.20		-	14	6.13
	22	3.66	12	3.36	22	5.10		-	18	6.17
	14	3.61	12	3.39	24	5.30		-	12	5.57
12	8	3.72	36	n.a.	10	5.52		-	5	5.35
		**	28	n.a.		5.53		-		5.50
	8	3.56		**	12	5.18		-	5	5.69
		3.65		**		4.79		-		5.87
		3.77		**		5.57		-		5.82
14	20	3.22		-		-		-		-
	26	3.46		-		-		-		-
	26	3.26		-		-		-		-
	22	3.30		-		-		-		-
	27	3.52		-		-		-		-
15	4	3.81	12	4.08	4	5.62	4	2.59	10	5.02
	4	3.77	12	4.80	4	5.70	4	2.71	12	4.58
	4	3.87	11	4.34	4	5.37	4	2.86	11	4.30
	3	3.68	11	4.60	4	5.47	3	2.67	11	4.42
	3	3.83	11	4.72	4	5.52	3	2.47	11	4.69
16		3.51	11	3.75	7	4.83	9	2.66		-
	8	3.57		3.63	10	5.06	10	2.60		-
	9	3.65		3.21		5.43		2.69		-
		3.63		3.52		4.93		2.79		-
		3.67	11	3.69		5.62		2.66		-
17	6	3.63	6	3.99		**	12	2.98		-
	6	3.58	4	4.00		**	13	n.a.		-
	8	3.53		**		**	12	n.a.		-
	6	3.72	6	3.71	18	5.53	14	2.48		-
	8	3.58	7	4.18	18	5.29	12	2.63		-
19	12	3.55	6	3.24	8	5.18		-		-
	11	3.60	5	3.51	7	5.22		-		-
	10	3.24	5	3.51	7	5.66		-		-
	10	3.47	6	3.74	7	5.11		-		-
	10	3.59	7	3.30	7	5.50		-		-

21		3.53		3.48	24	5.70		-		-
		3.55		3.55		4.92		-		-
	24	3.53	24	3.53		5.41		-		-
		3.45	20	3.69	24	5.68		-		-
	20	3.04		3.63		5.48		-		-
22	18	3.63	8	4.13	11	5.32	18	2.77	12	6.36
	12	3.69	6	3.96	13	5.96	15	2.82	10	6.34
	7	3.65	12	3.42	10	4.75	13	2.71	11	6.34
	10	3.65	17	3.23	7	5.53	26	2.77	15	6.65
	10	3.71	11	3.36	13	5.46	22	2.69	14	4.88
23	29	3.91	30	3.99	30	5.21		-		-
	30	3.86	24	3.67	23	5.31		-		-
	28	3.61	21	3.81	23	5.67		-		-
	30	3.93	28	3.81	29	5.31		-		-
	21	3.69	29	4.23	38	5.23		-		-
24	5	3.56	8	4.29	4	5.39	8	2.65		-
	5	3.62	8	3.77	5	5.48	6	2.72		-
	5	3.63	8	4.32	5	5.59	5	2.76		-
	5	3.67	6	4.07	4	5.62	6	2.77		-
	6	3.72	6	4.03	5	5.67	5	2.79		-
25	31	3.70	28	4.13	30	5.71		-		-
	24	3.71	24	3.75	31	6.01		-		-
	25	3.64	32	3.60	26	5.96		-		-
	24	3.66	31	3.44	28	5.80		-		-
	29	3.70	29	3.96	23	6.11		-		-
26		**	28	n.a.	7	5.51	6	2.66		-
	13	3.60		**		**	5	2.71		-
	14	3.74	18	n.a.	6	5.10	5	2.75		-
		3.64		**	6	5.21	8	2.79		-
	17	3.74		**	5	5.19	8	2.58		-
28	10	3.54	13	3.91	10	5.94		-		-
	9	3.65	20	4.41	15	5.00		-		-
	15	3.62	10	3.62	13	5.67		-		-
	15	3.68	24	4.11	15	5.62		-		-
	14	3.69	10	3.91	13	5.24		-		-
29	40	n.a.	53	n.a.		n.a.		-		-
		**	51	n.a.	59	n.a.		-		-
		n.a.		n.a.	43	n.a.		-		-
		n.a.		**		n.a.		-		-
		n.a.		n.a.		n.a.		-		-
30	20	3.61	8	4.19	13	5.41	25	2.86		-
	20	3.57	18	3.91	12	5.09	23	2.72		-
	18	3.52	17	3.91	15	5.13	20	2.38		-
	20	3.62	12	4.08	12	5.05		**		-
	20	3.69	13	3.73	16	5.40	19	2.66		-
31	19	3.55		**	8	4.90		-		-
	15	3.55	14	3.00	8	5.20		-		-
	17	2.40		**	9	5.10		-		-
	25	3.30	10	2.70	10	5.25		-		-
	16	2.20	8	3.00	9	5.40		-		-
32	10	3.48	11	3.66	7	4.93		-		-
	10	3.65	11	3.56	7	5.09		-		-
	10	3.72	11	3.65	7	5.13		-		-
	10	3.47	11	3.88	7	4.66		-		-
	10	3.47	11	3.50	7	5.47		-		-

34	31	3.70	23	4.30	18	5.80		-		-
	26	3.60	23	4.50	21	5.80		-		-
		**	24	4.30	20	6.20		-		-
	28	3.70	26	4.00	17	5.90		-		-
	25	3.60	22	4.20	20	5.50		-		-
35	15	3.94		**	19	5.67		-		-
	13	3.53		**	17	5.25		-		-
		3.75		**		5.20		-		-
		3.64		**		5.83		-		-
		3.49		**		5.51		-		-
36	16	3.64		-	14	5.55		-		-
	16	3.51		-	13	5.31		-		-
	16	3.62		-	12	4.91		-		-
	22	3.67		-	14	5.31		-		-
	25	3.73		-	13	5.01		-		-

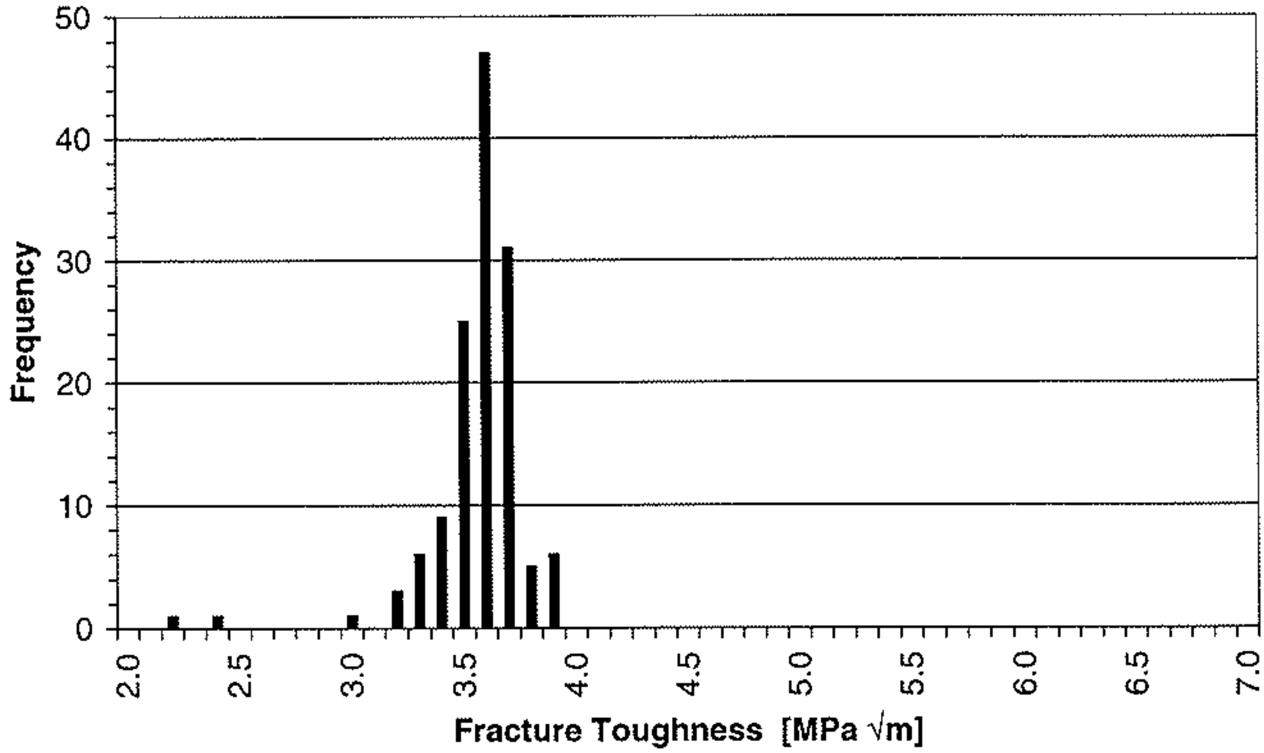


Figure A2.1
Frequency distribution graph for material A (alumina–998).

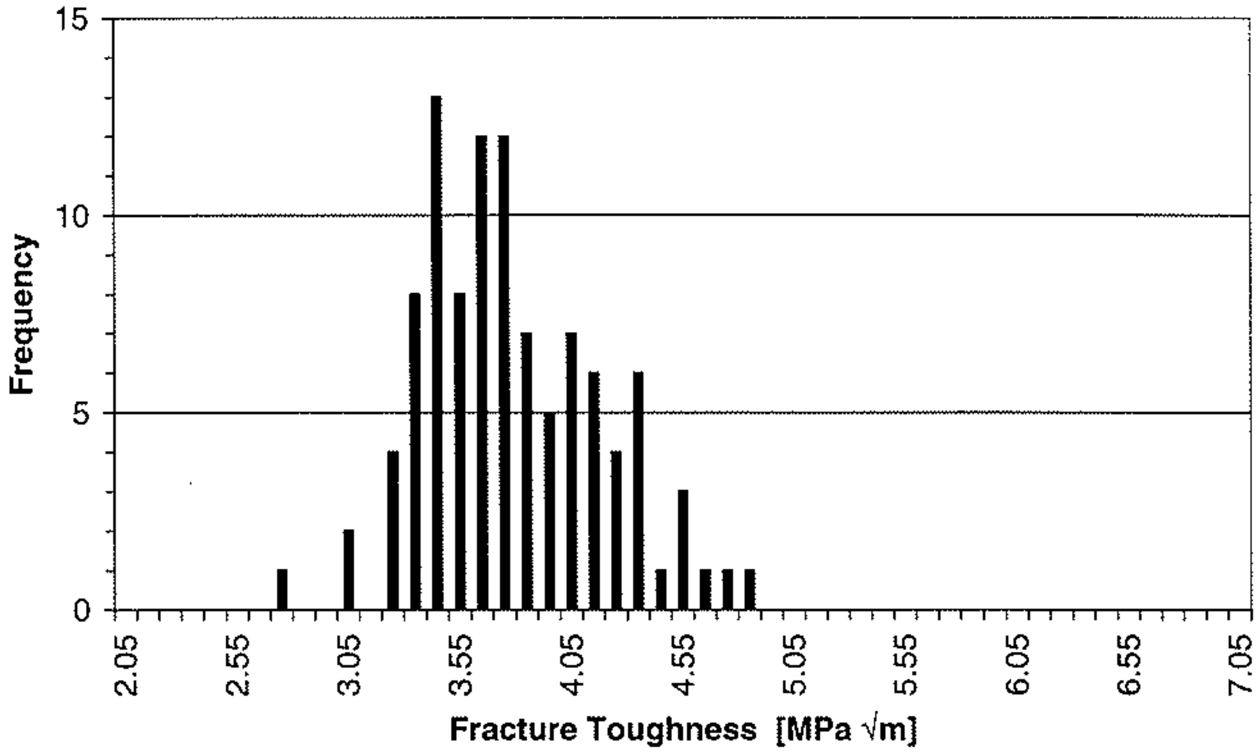


Figure A2.2
Frequency distribution graph for material B (alumina–999).

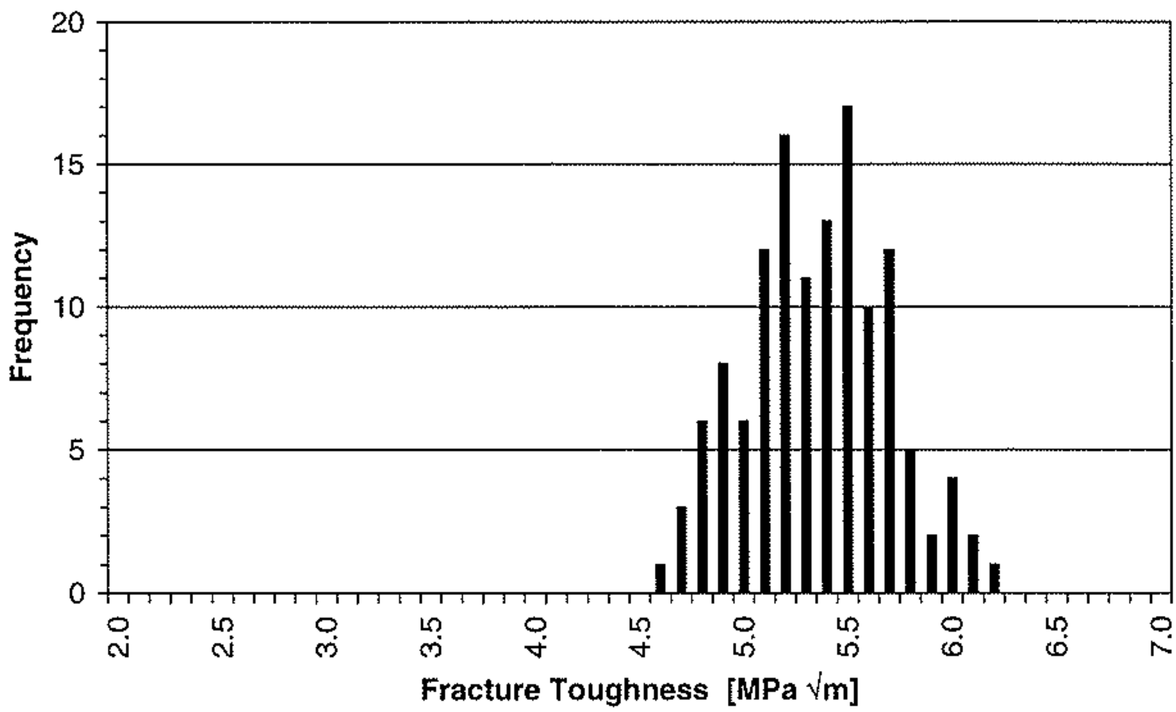


Figure A2.3
Frequency distribution graph for material C (GPSSN).

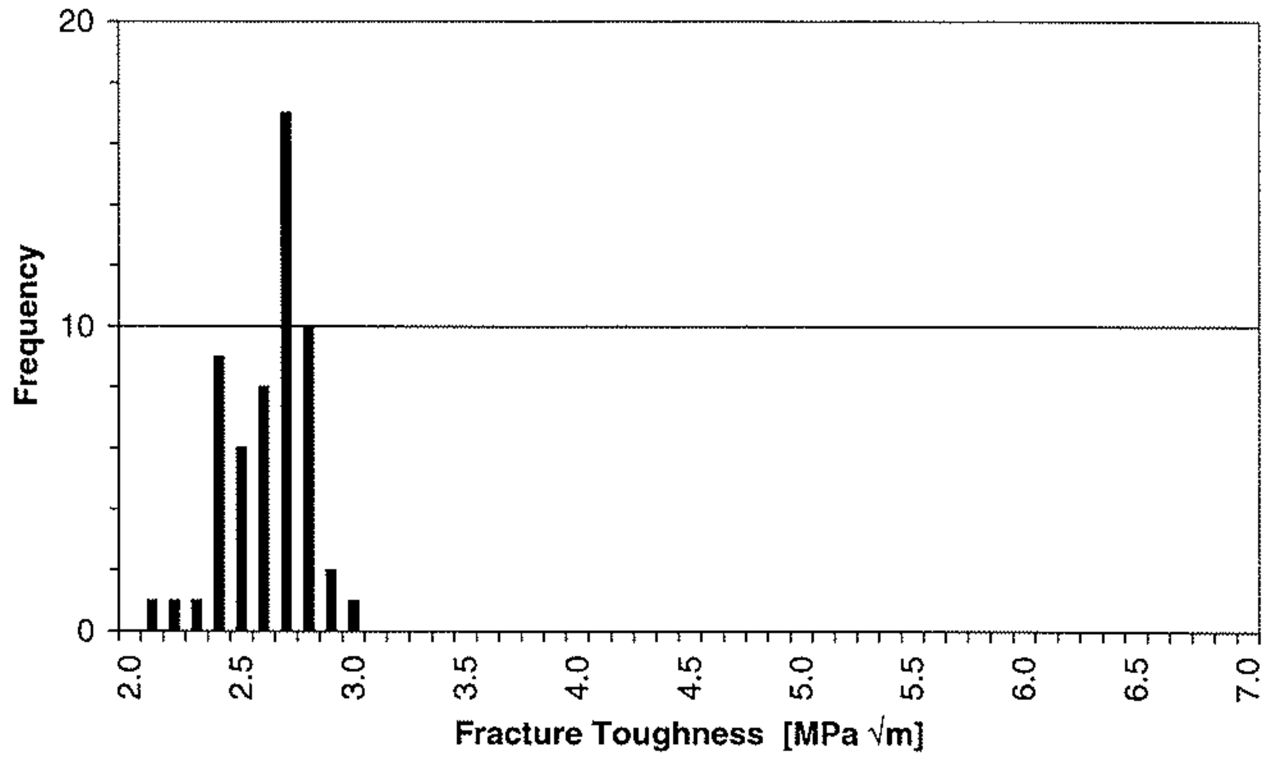


Figure A2.4
Frequency distribution graph for material D (SSiC).

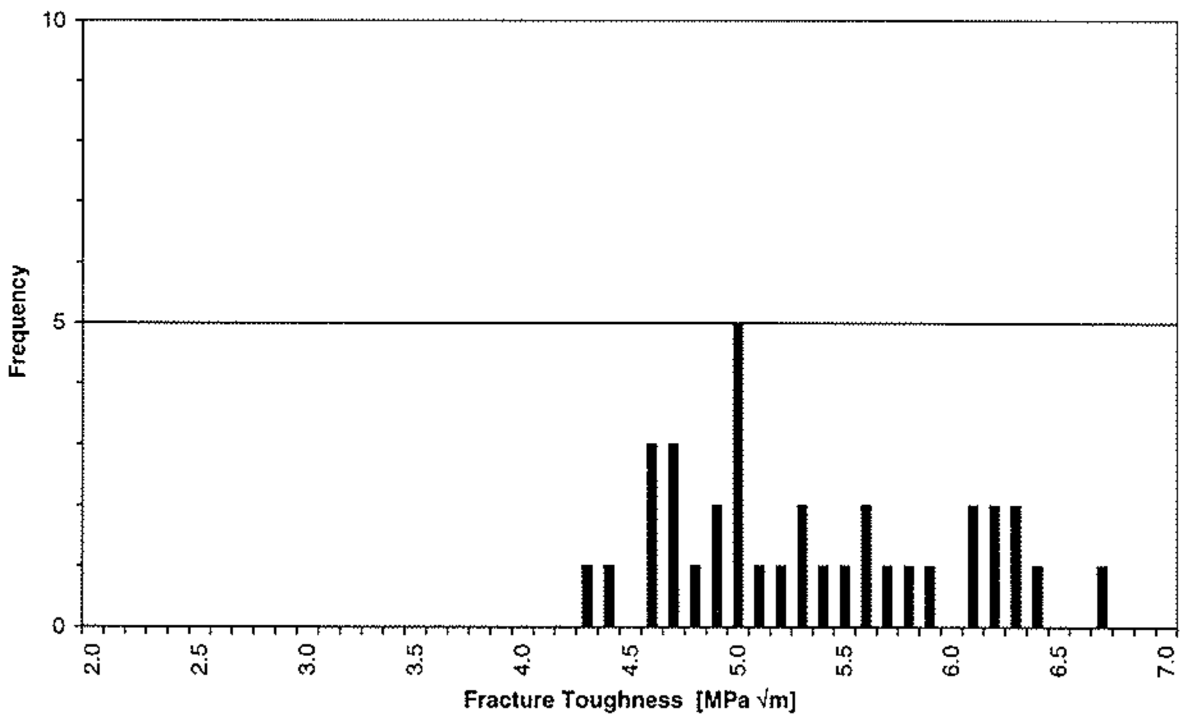
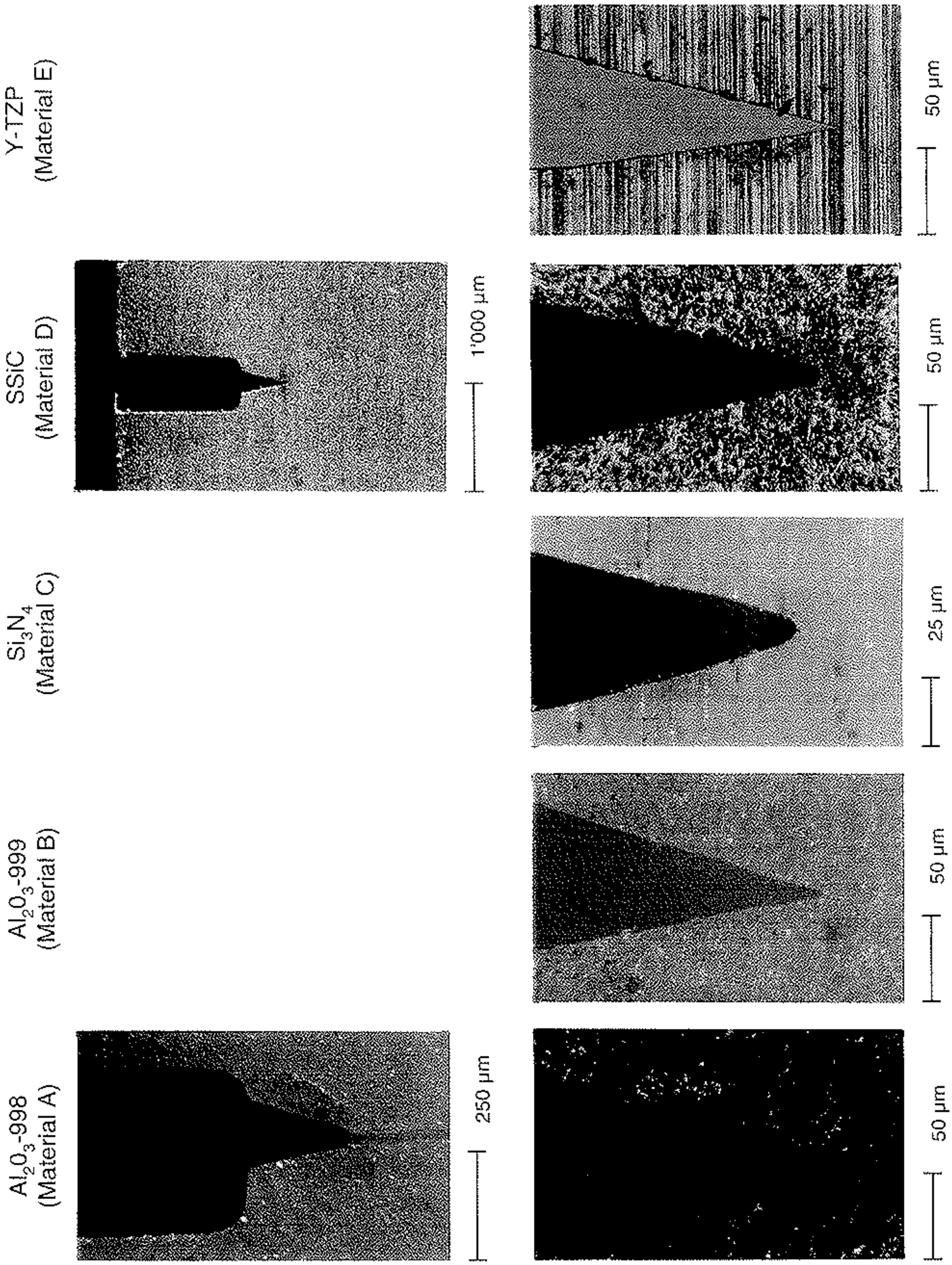


Figure A2.5
Frequency distribution graph for material E (Y-TZP).

Appendix A3: Notch and Notch Tip Geometry Participant # 1



Remarks:

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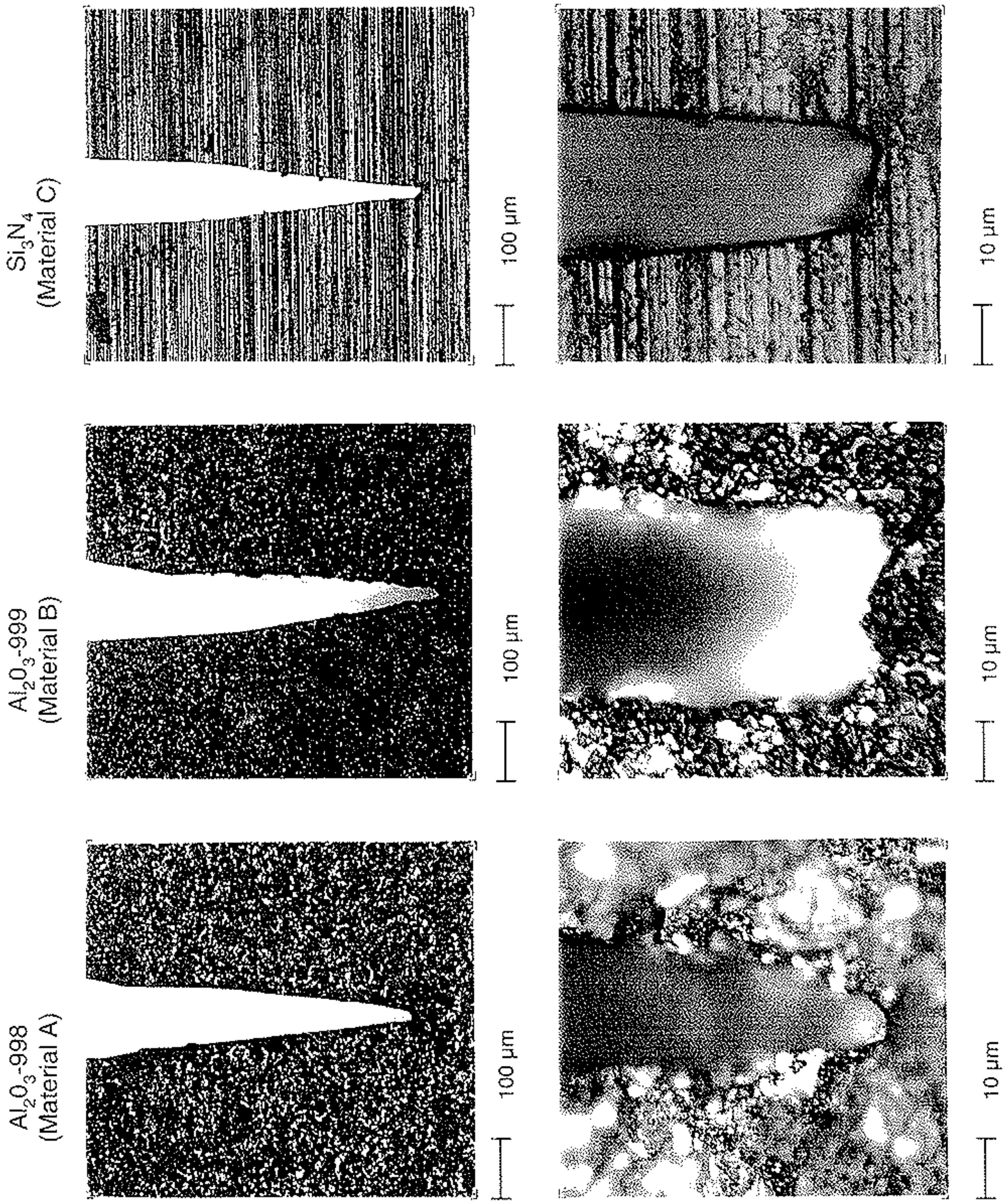
Participant # 2



Remarks:

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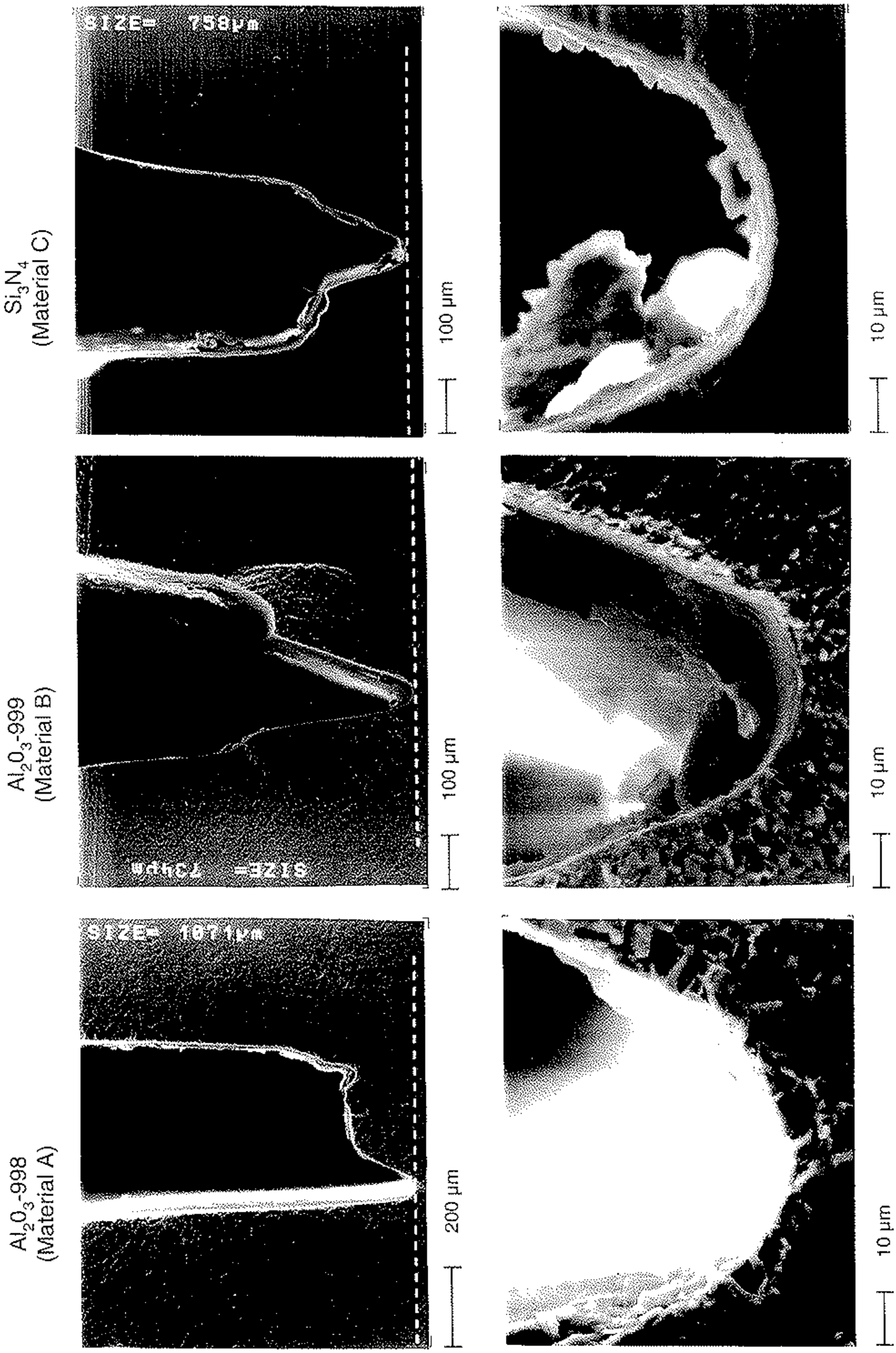
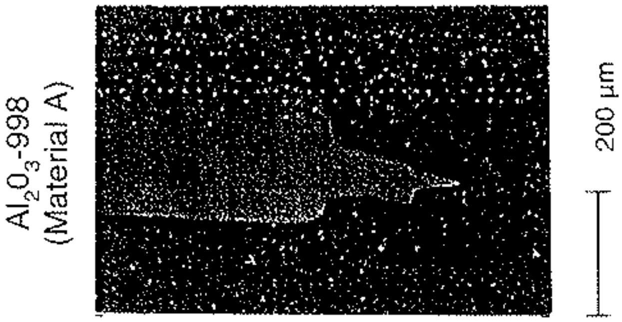
Participant # 3

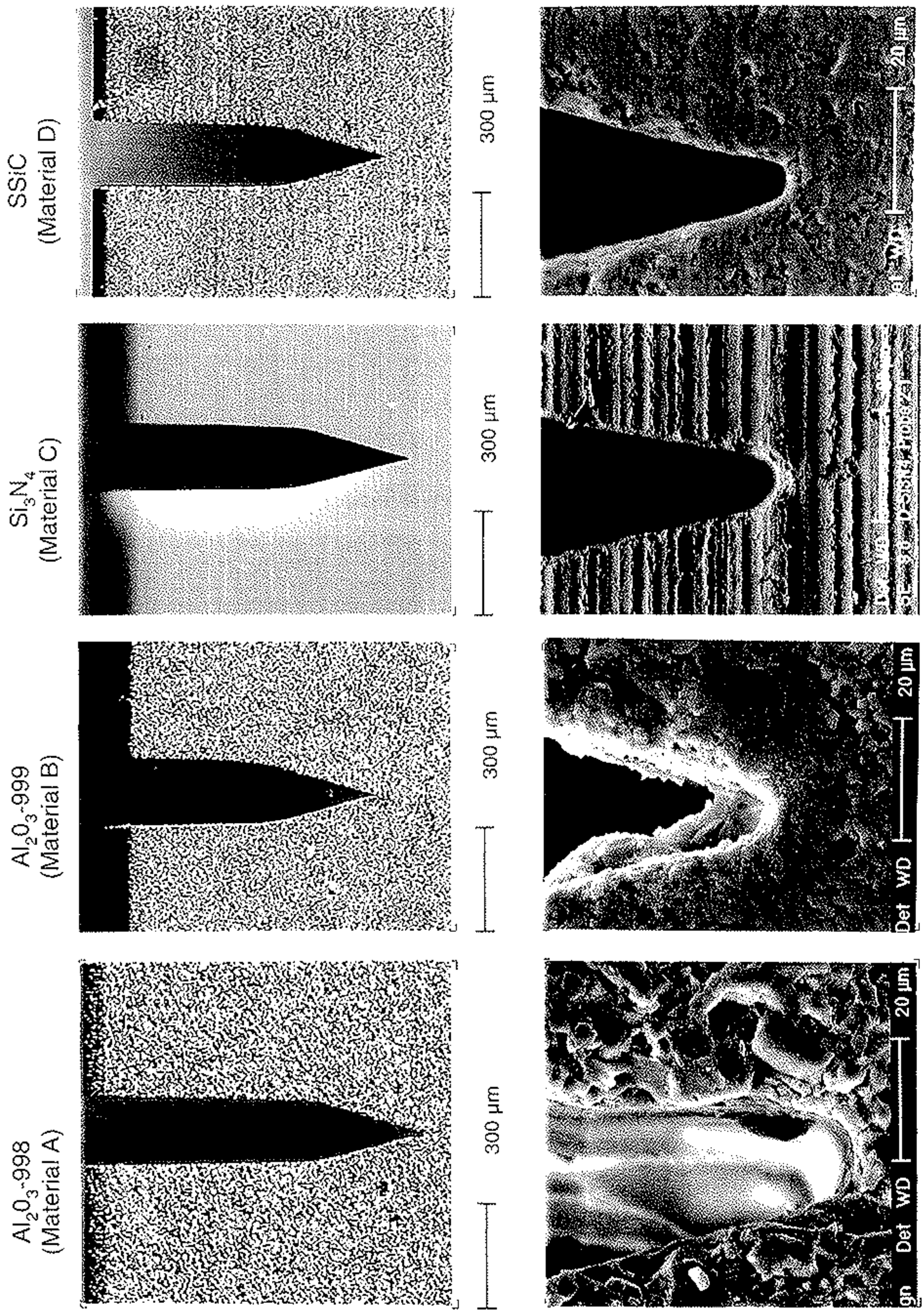


Participant # 4



Remarks:
The photo above was taken from the mini-round robin this participant joined previously to verify the instructions. He did not supply photos from the notches of the materials A, B, C, D and E tested in the round robin itself.

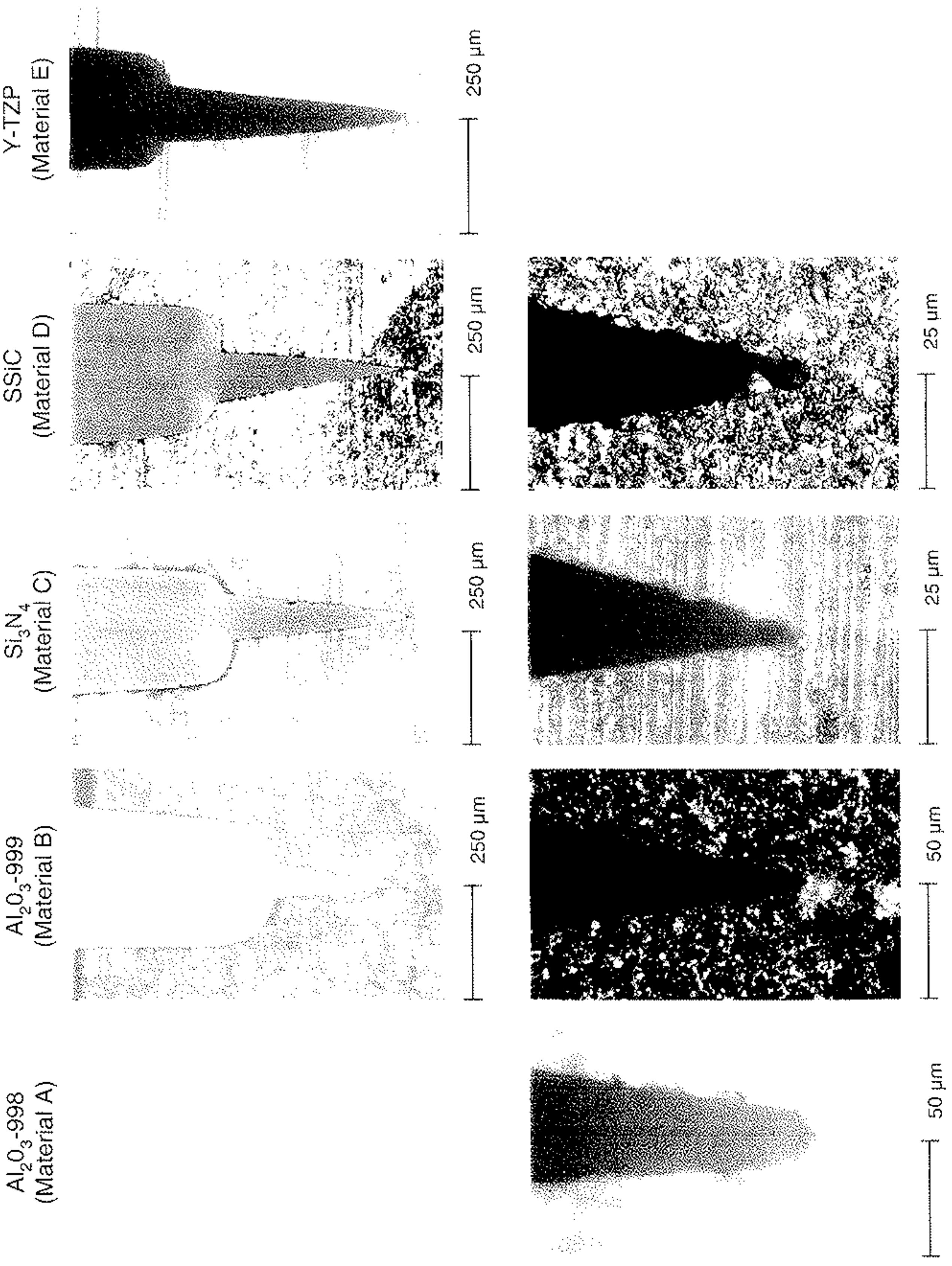
Participant # 5



Remarks:

The participant built his own polishing apparatus and optimised his polishing procedure. Details of the procedure see *appendix tips and tricks.*

Participant # 6



Remarks:

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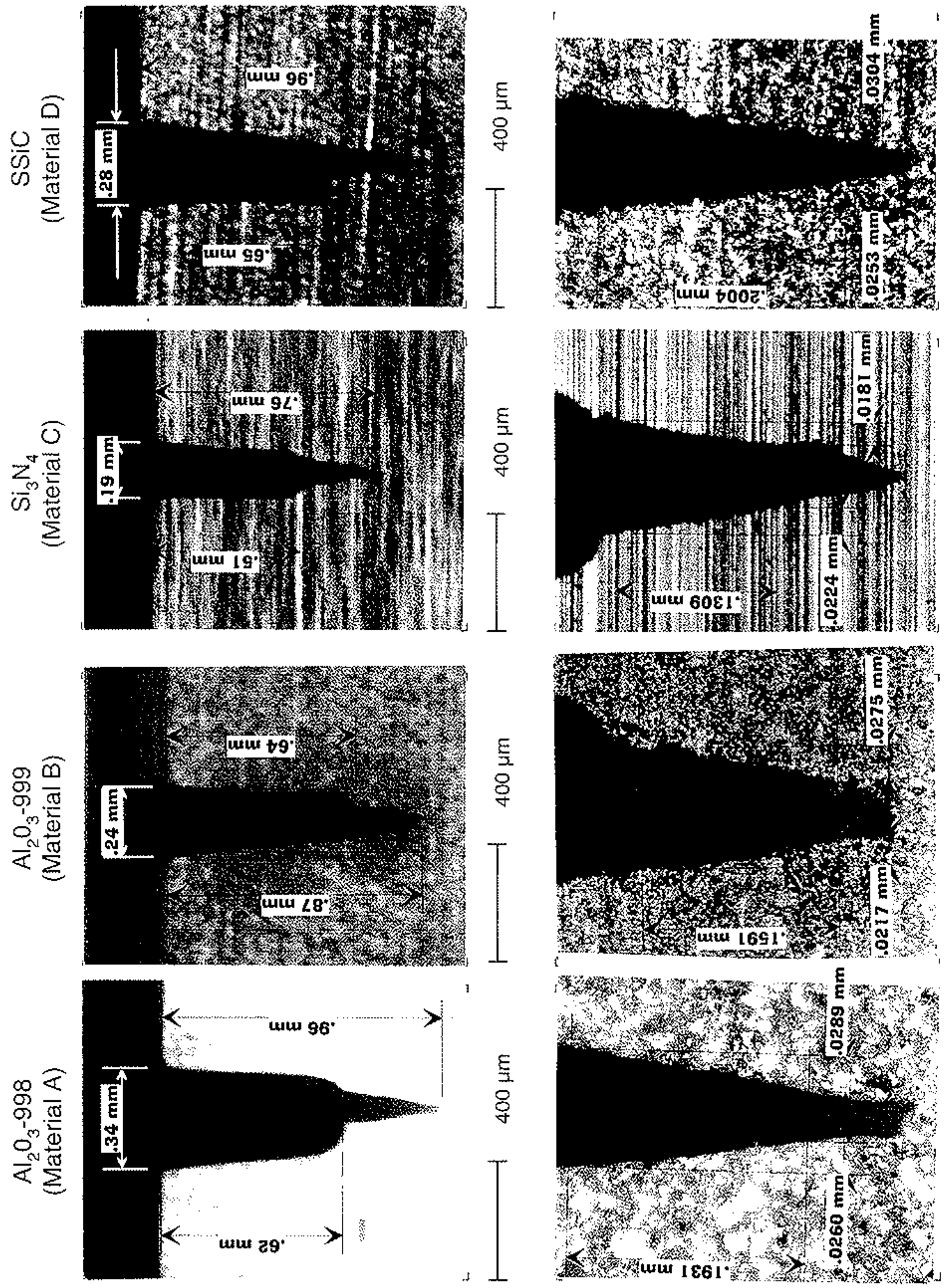
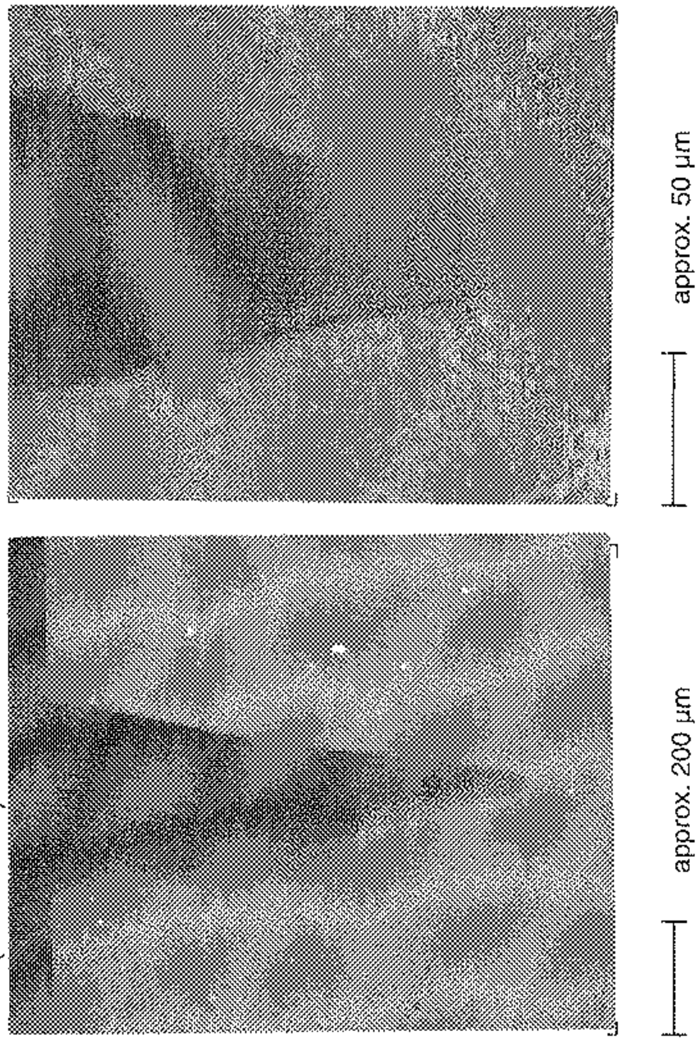
Participant # 7



Remarks:

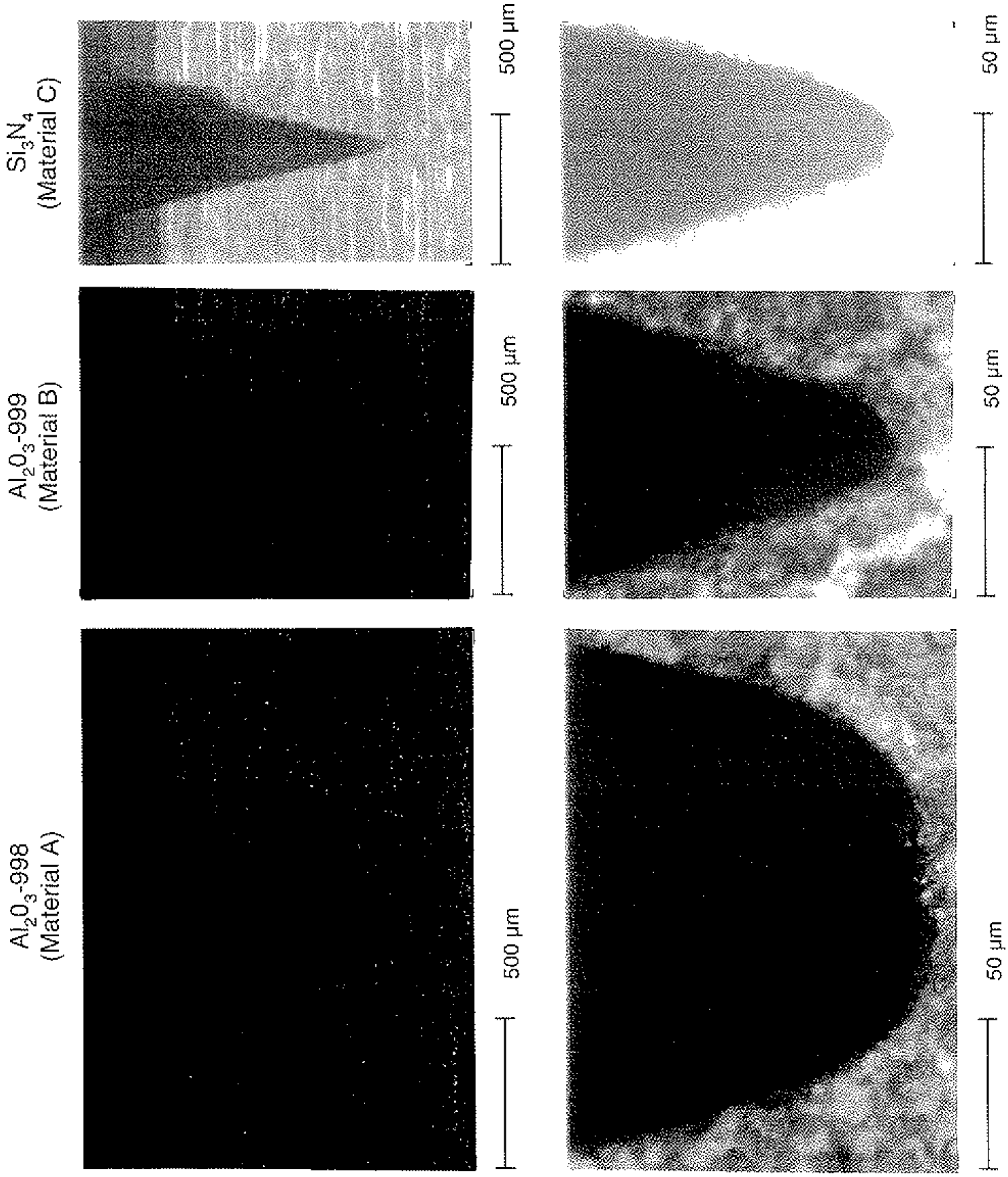
Participant # 8



Al_2O_3-998
(Material A)

Remarks:
No photos from the notches in the also tested materials B and C were supplied by the participant.

Participant # 9



Remarks:

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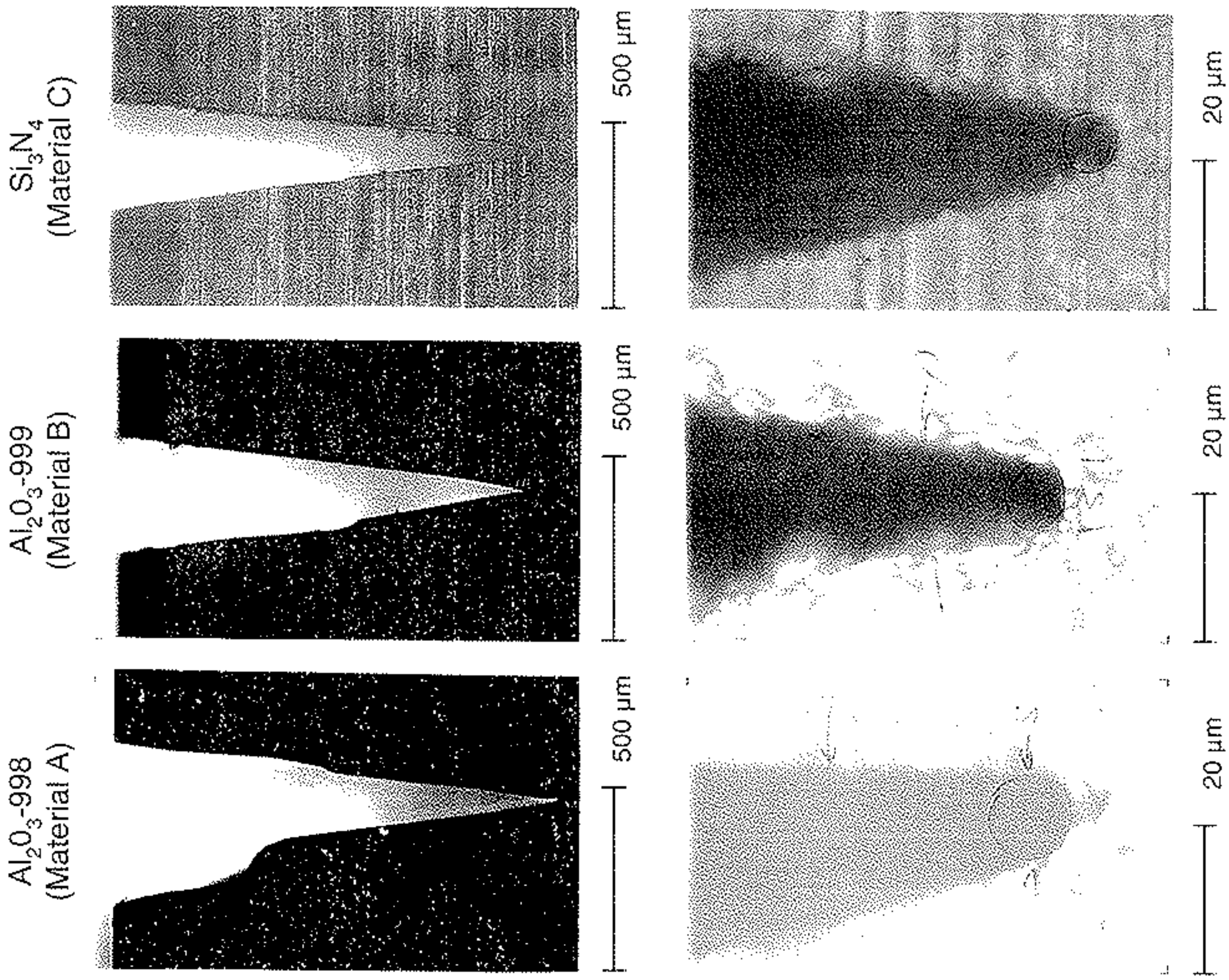
Participant # 10



Remarks:

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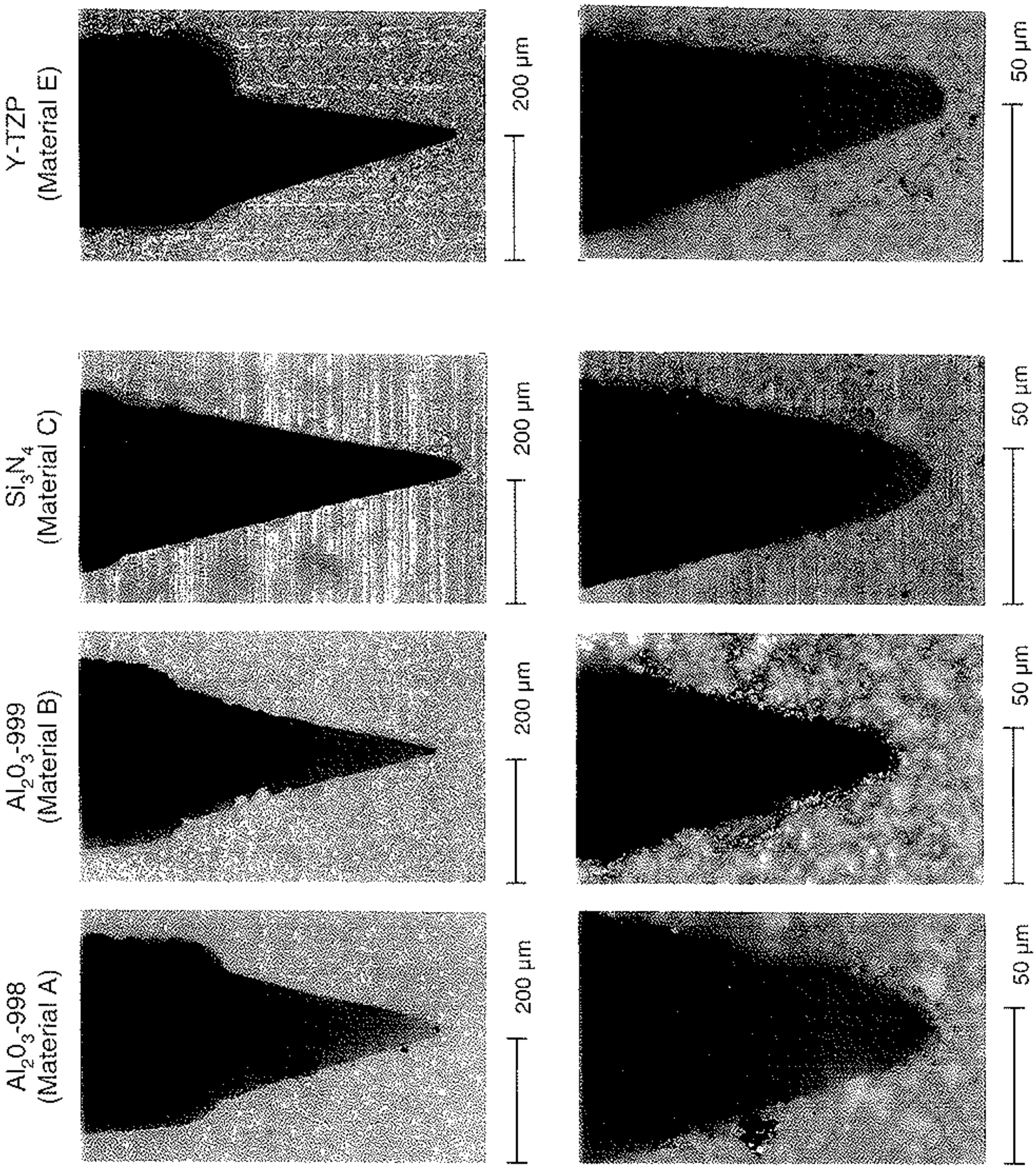
Participant # 11



Remarks:

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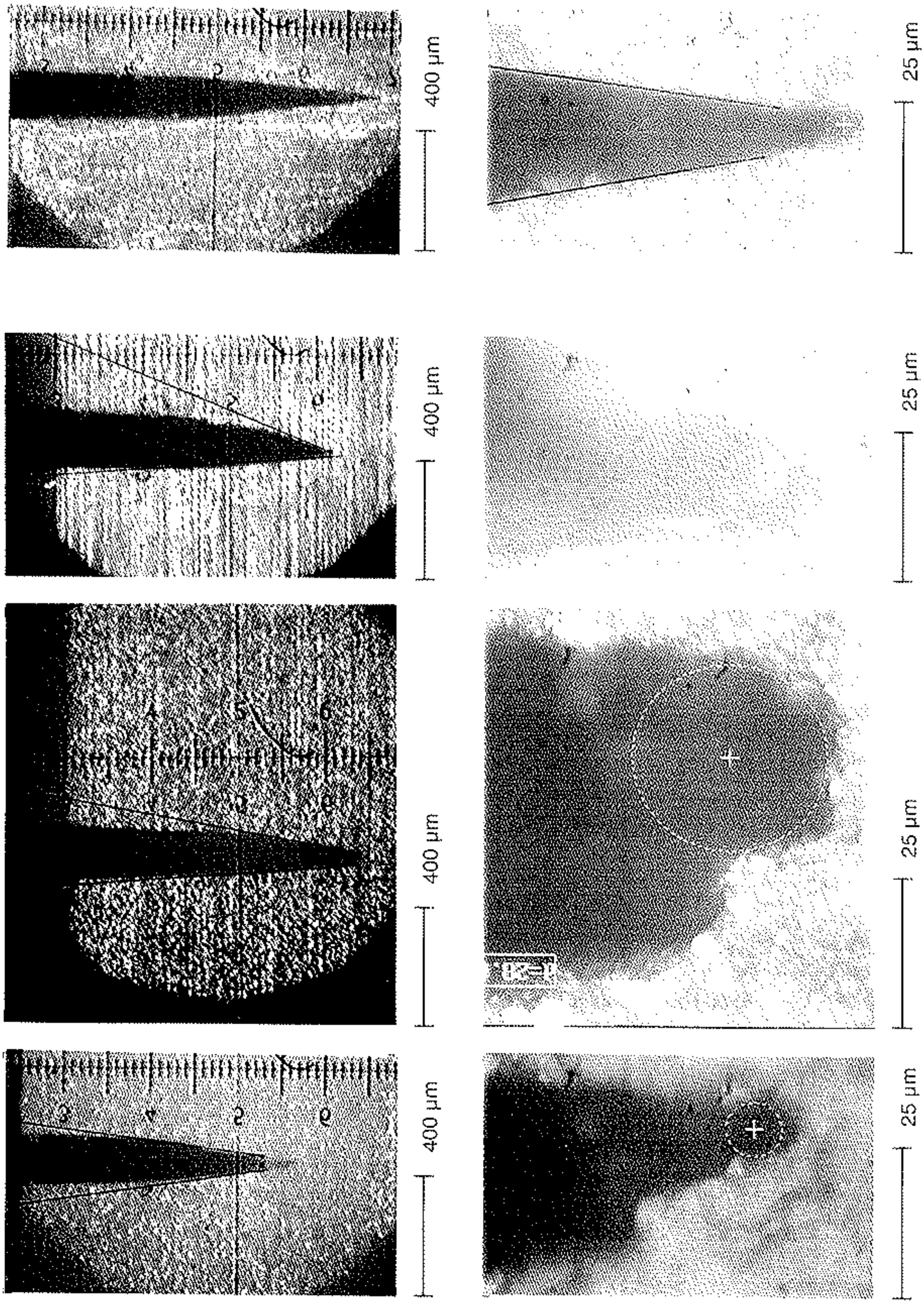
Participant # 12

Y-TZP (Material E)

Si_3N_4 (Material C)

Al_2O_3-999 (Material B)

Al_2O_3-998 (Material A)



Remarks:

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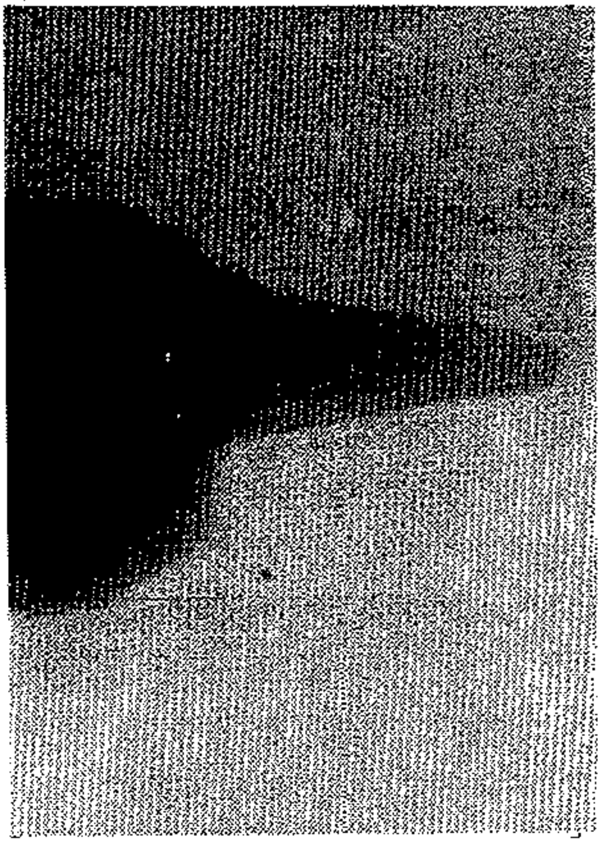
Participant # 14

Al_2O_3-998
(Material A)

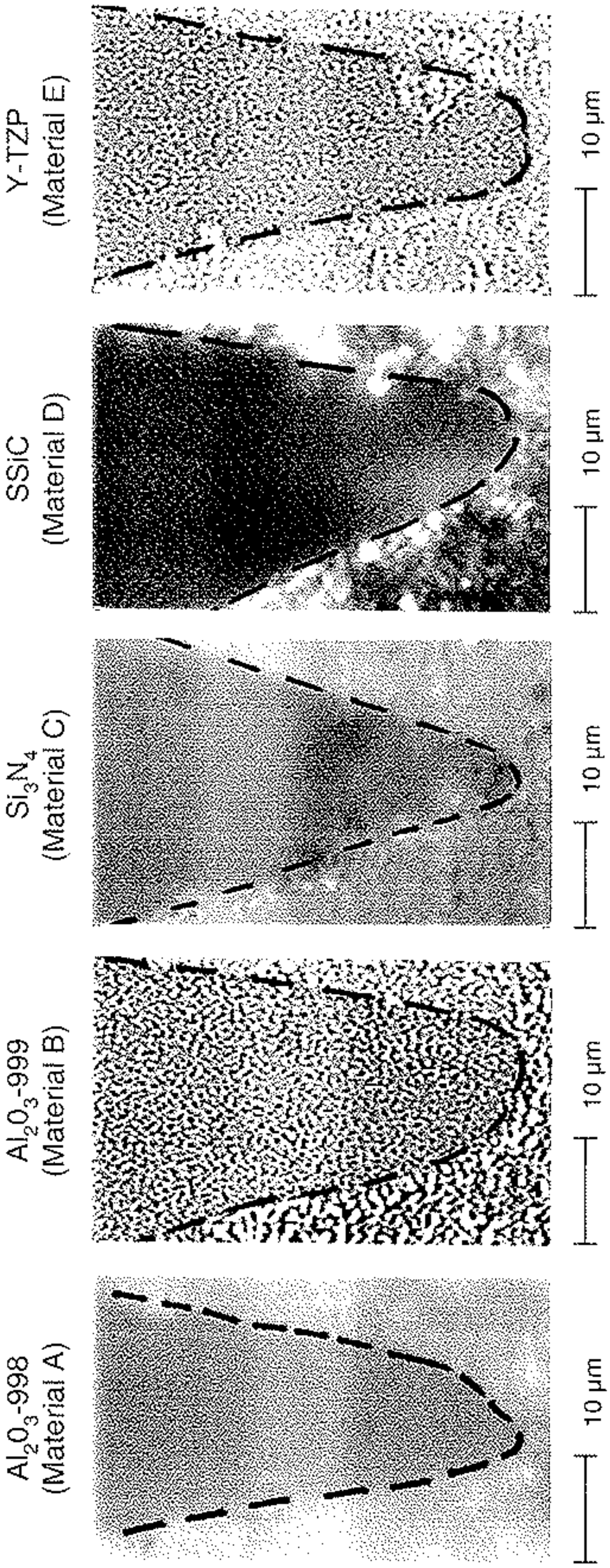


magnification unknown

Remarks:

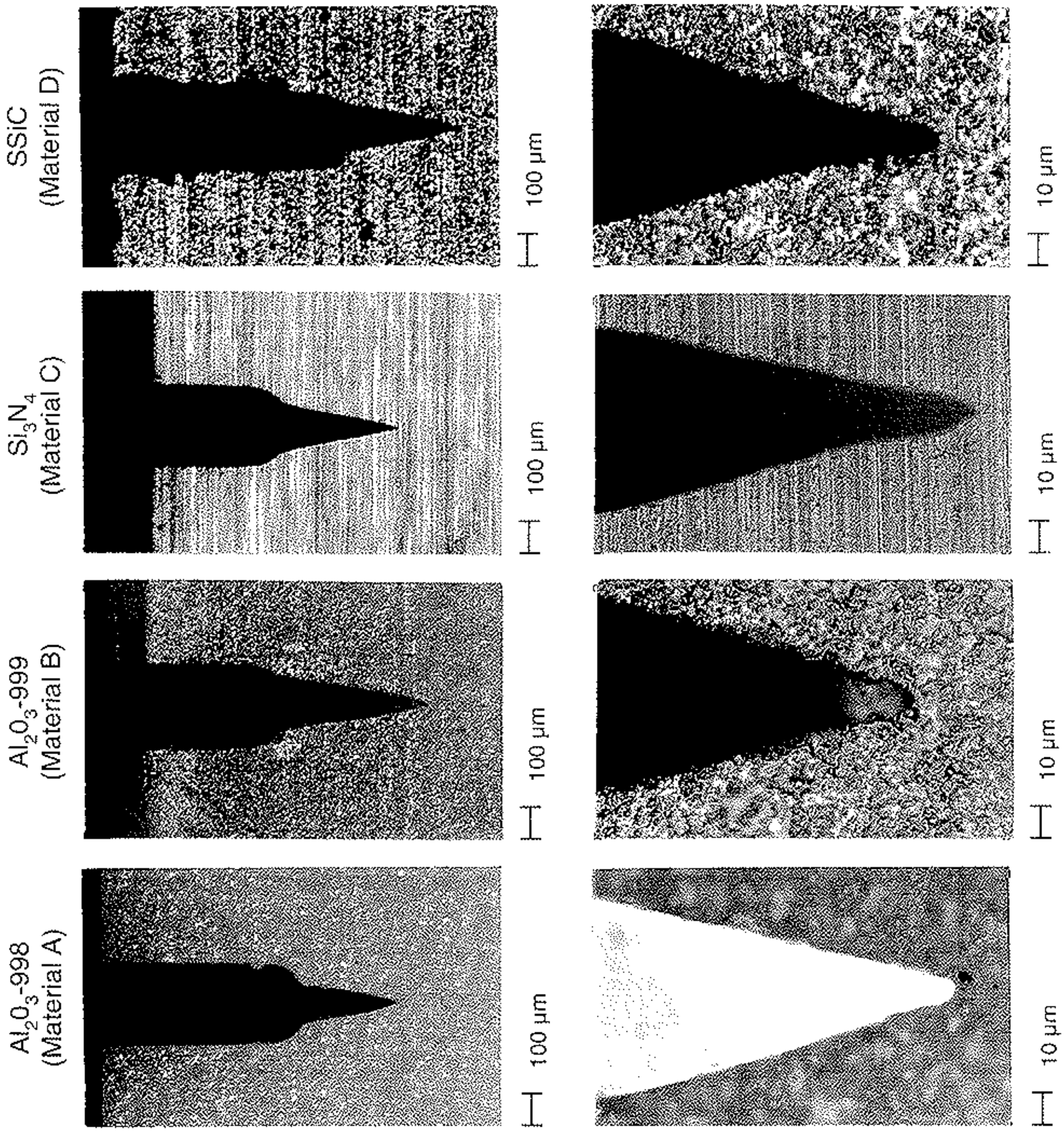
-

Participant # 15



Remarks:
The dashed lines were drawn by the round robin organiser.

Participant # 16



Remarks:

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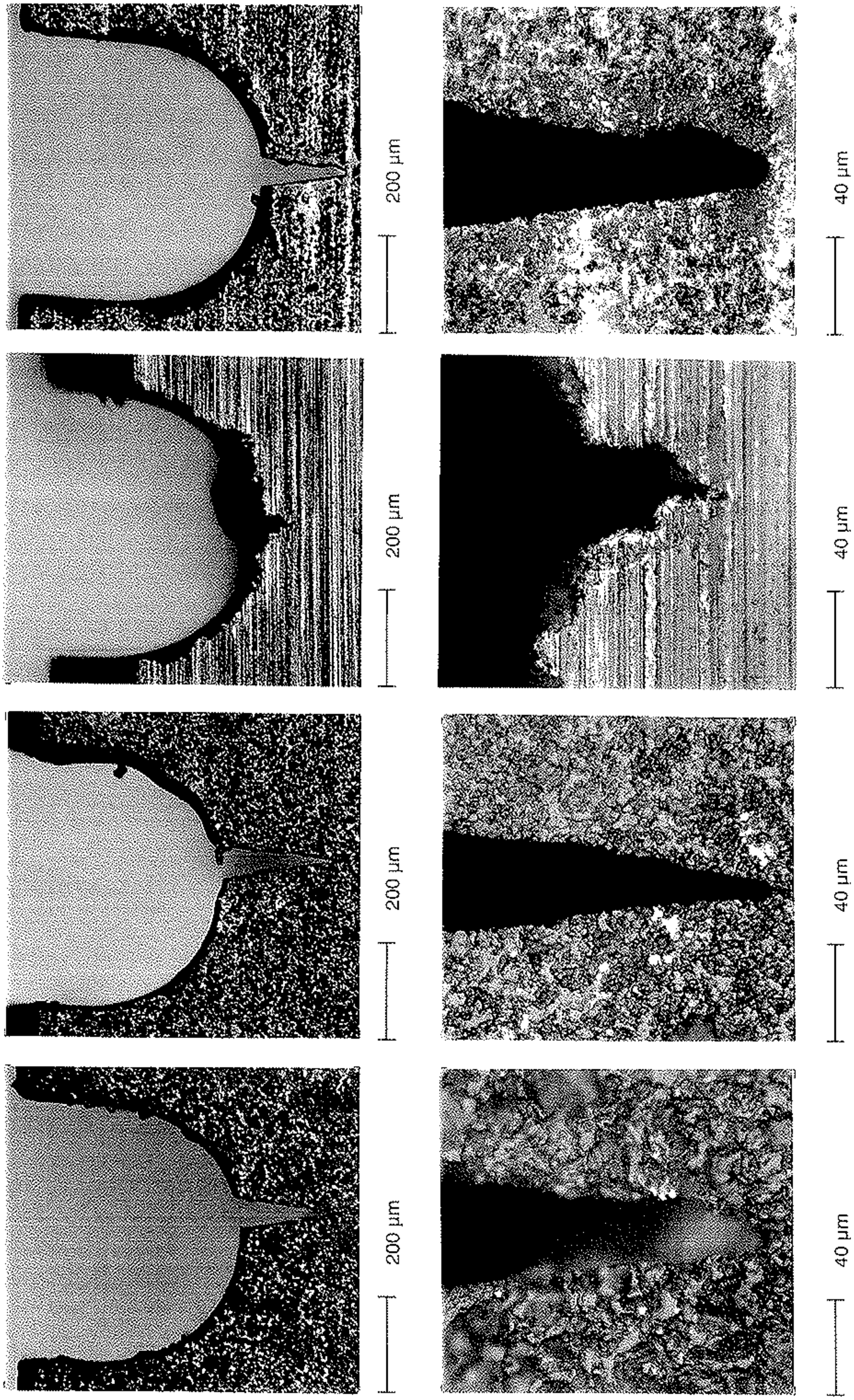
Participant # 17



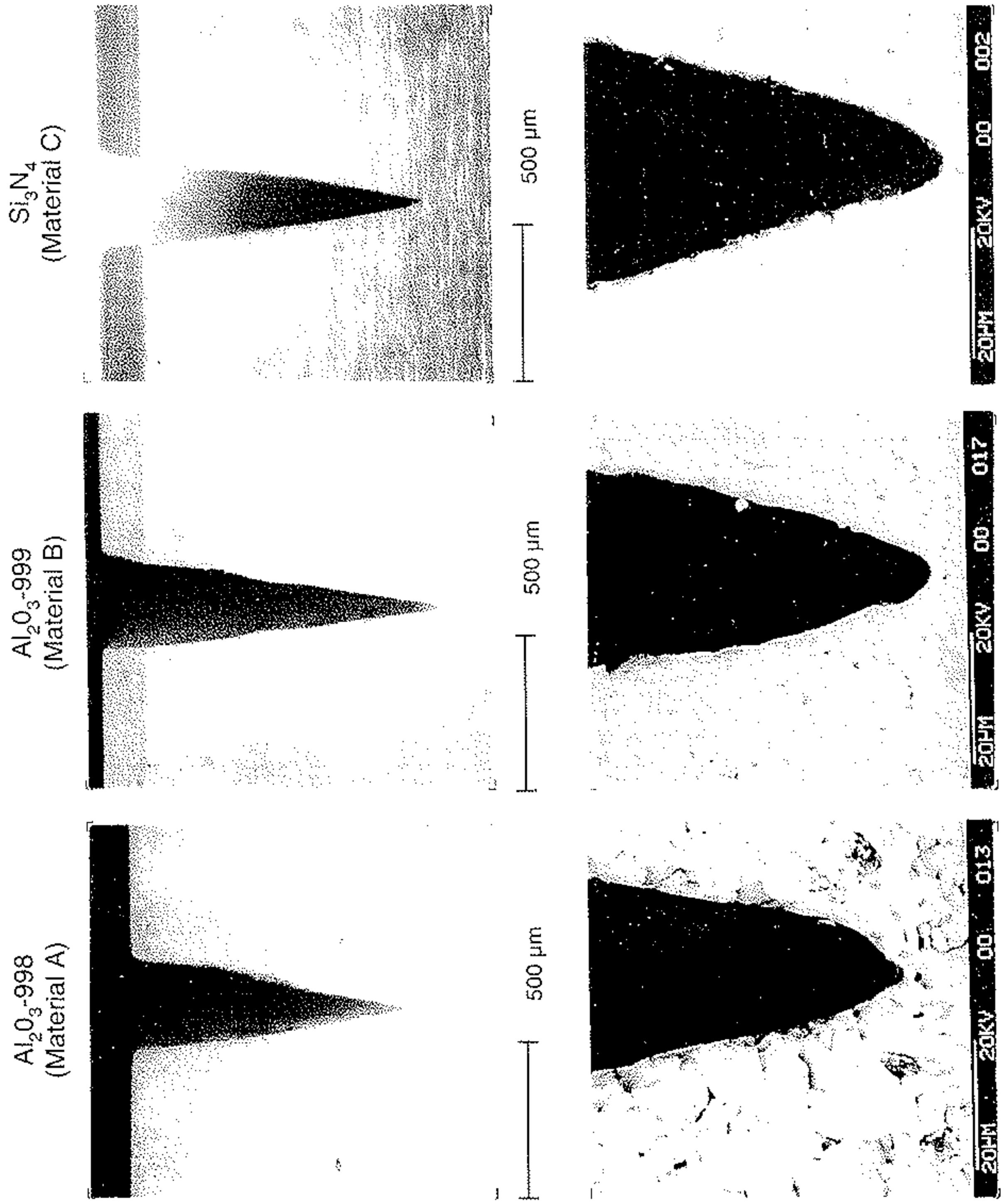
Al_2O_3-998 (Material A)

Al_2O_3-999 (Material B)

Si_3N_4 (Material C)

SSiC (Material D)

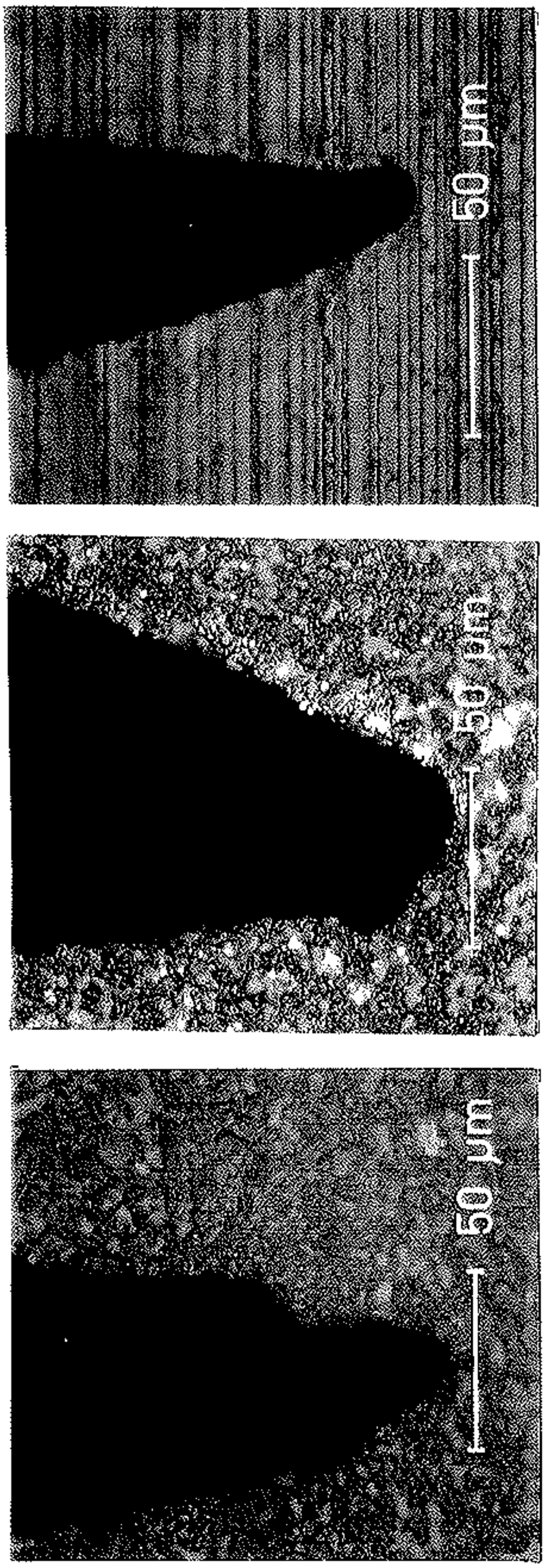
Participant # 19



Remarks:

-

Participant # 21



Al_2O_3-998 (Material A)

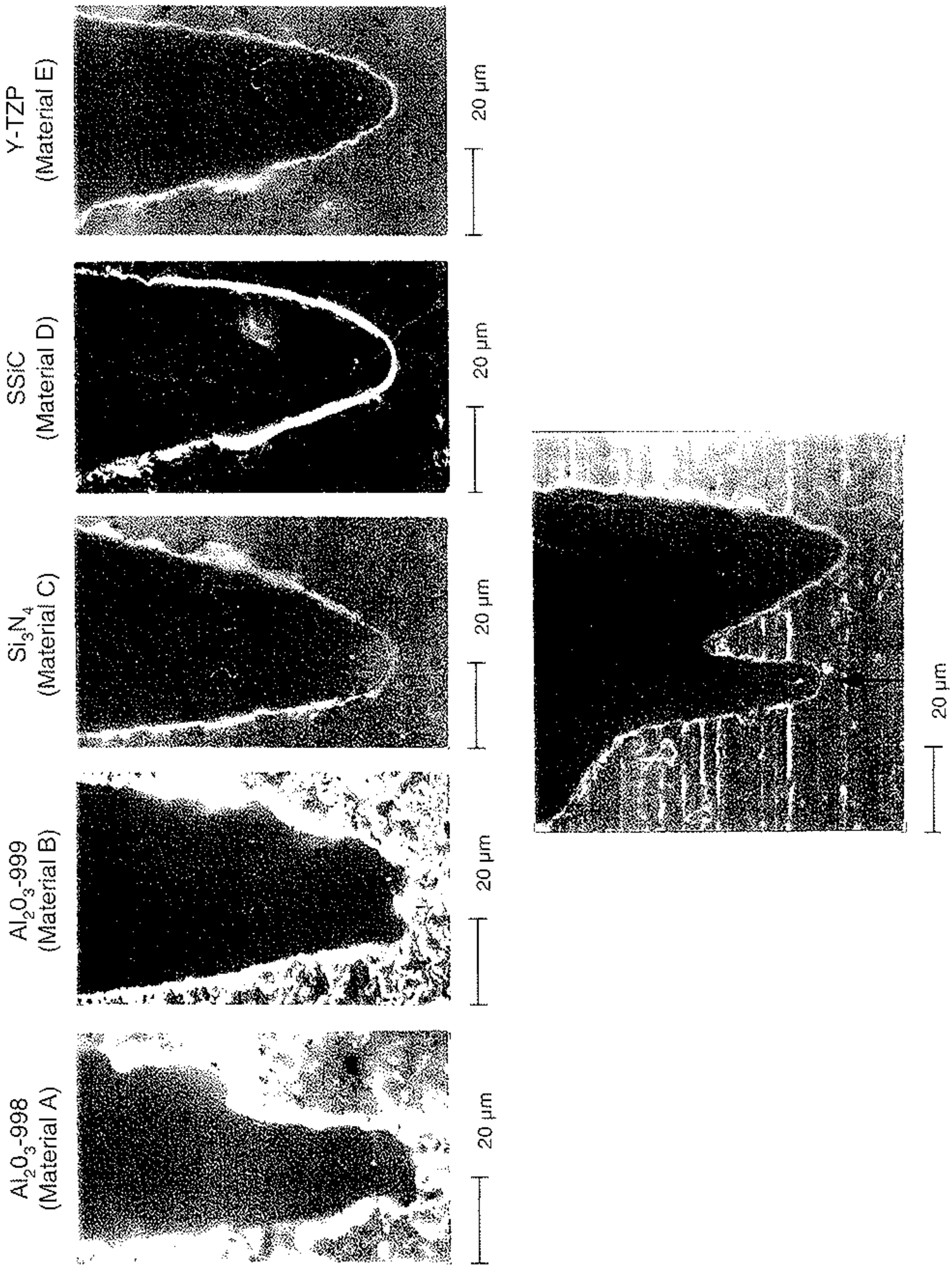
Al_2O_3-999 (Material B)

Si_3N_4 (Material C)

Remarks:

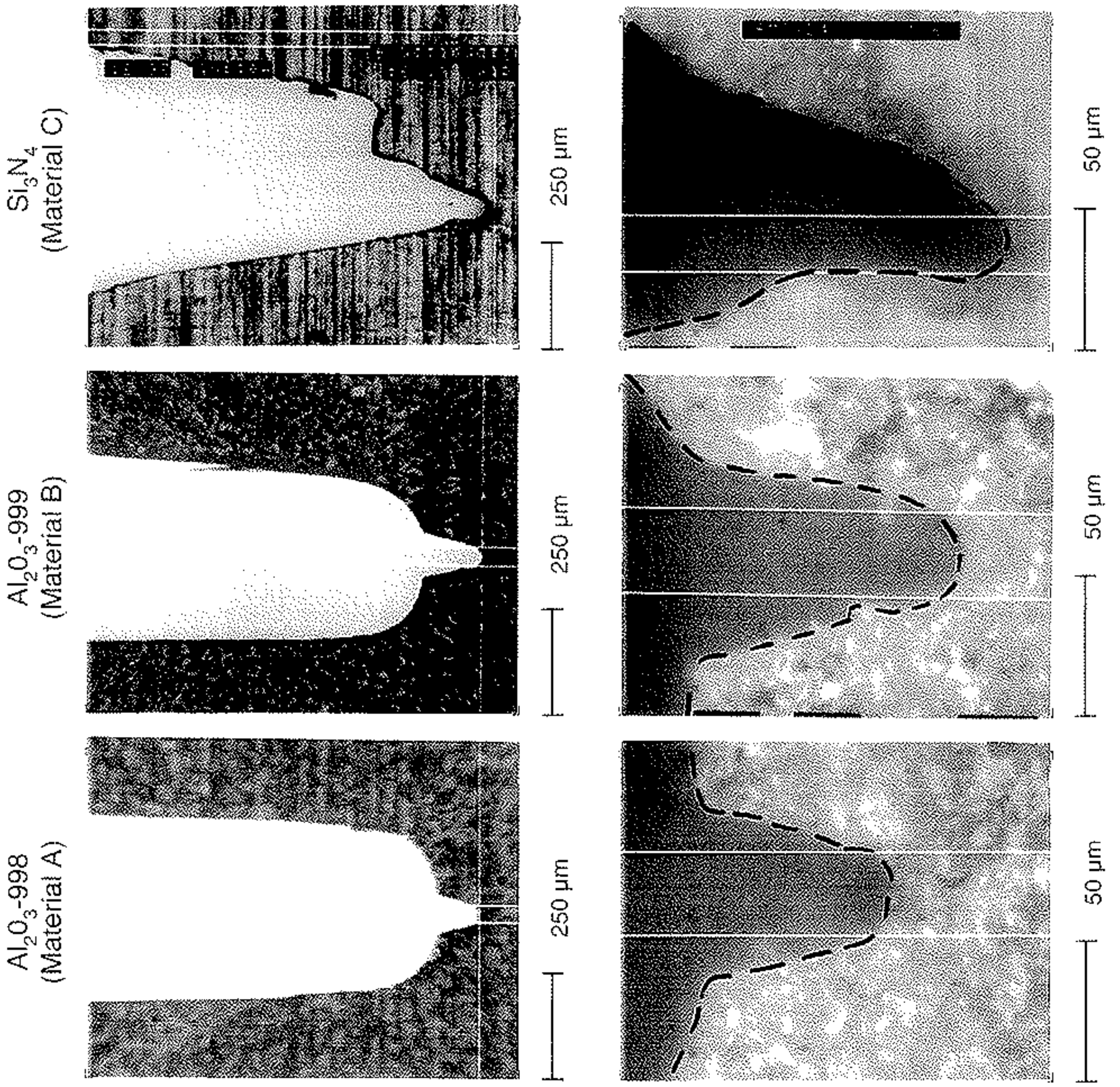
-

Participant # 22



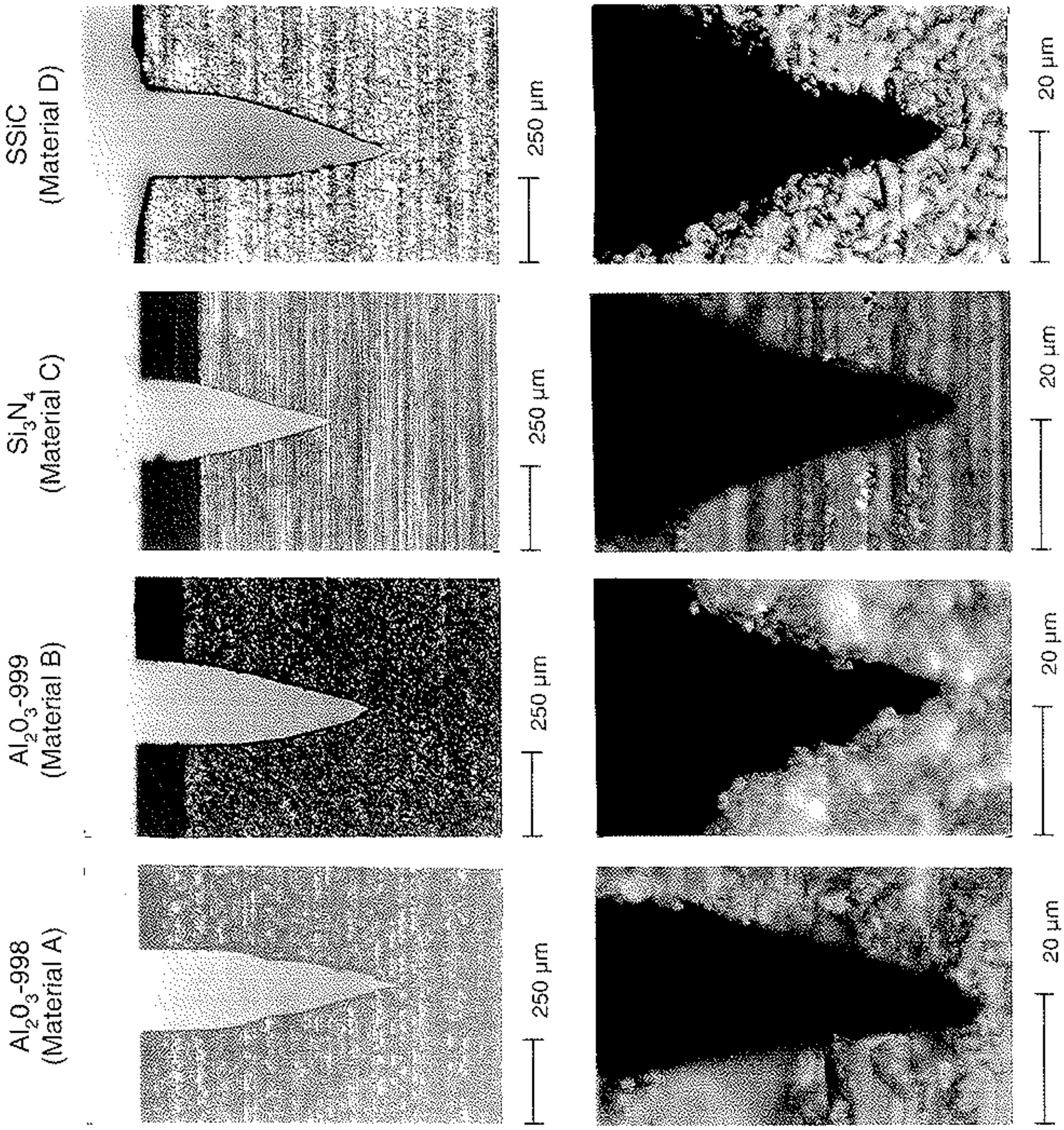
Remarks:
Both ends of the V-notch tip of a Si_3N_4 bend bar are shown.

Participant # 23



Remarks:
The dashed lines were drawn by the round robin organiser.

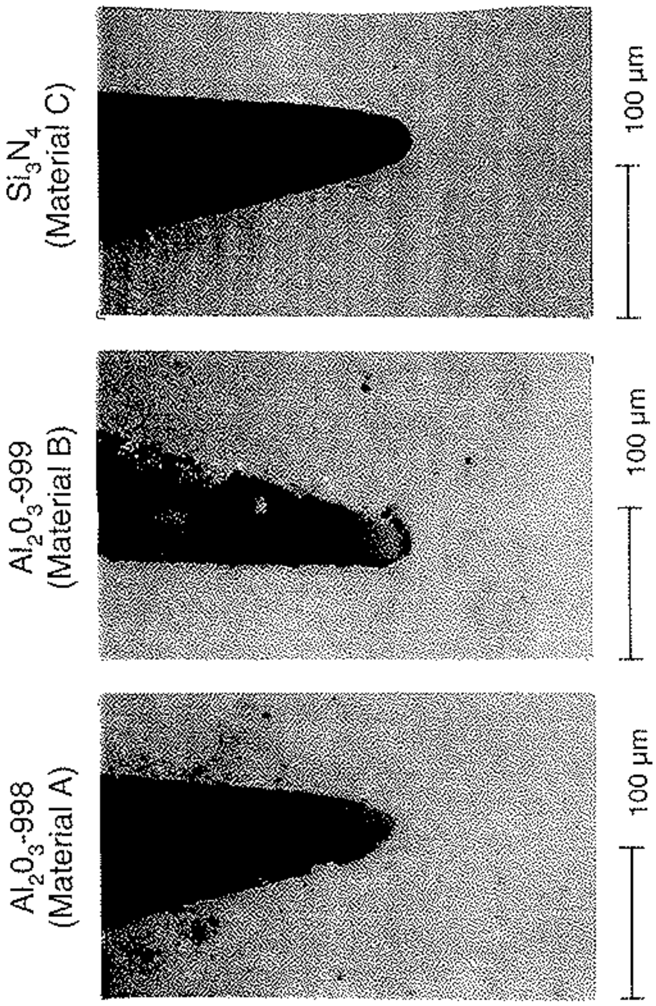
Participant # 24



Remarks:

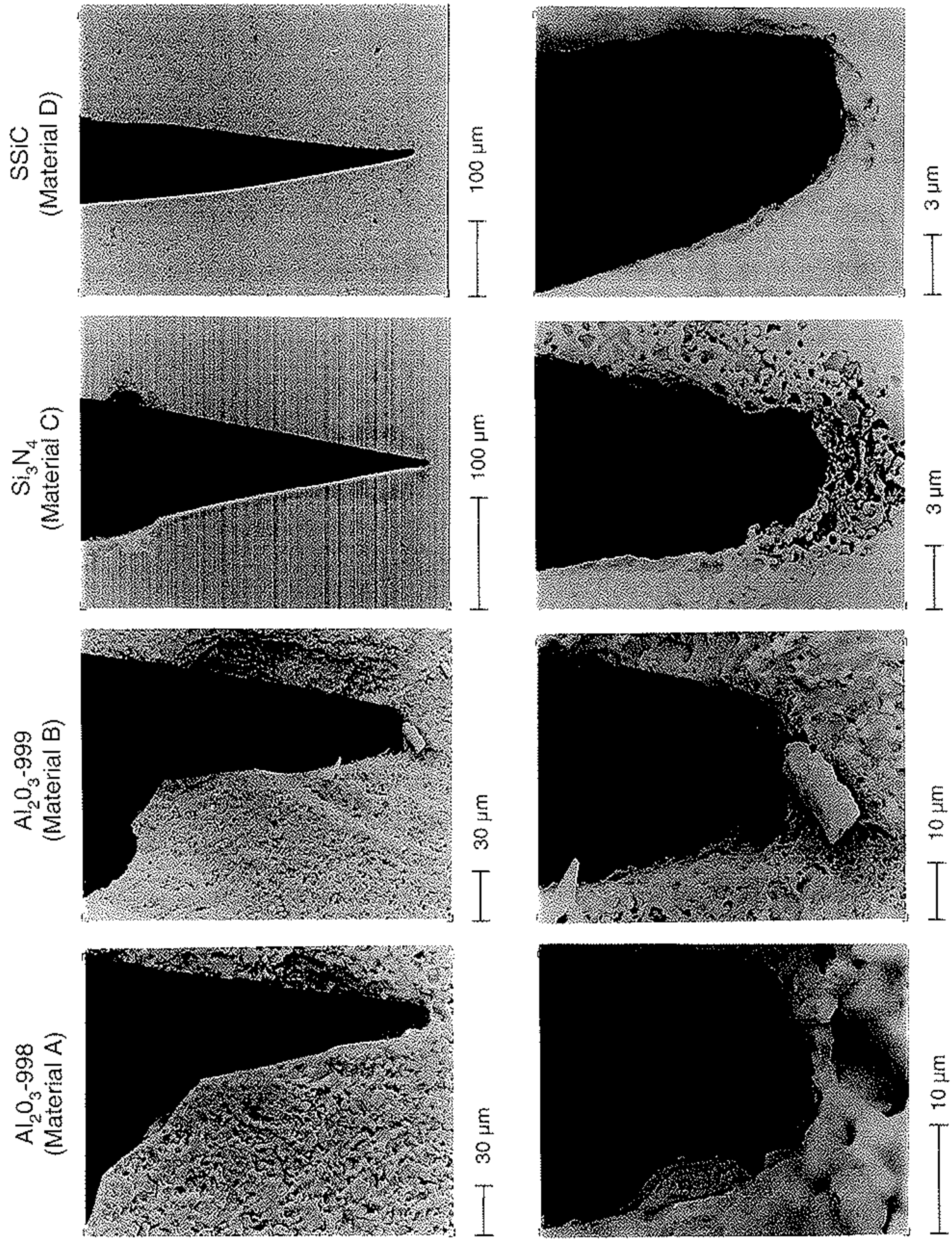
-

Participant # 25



Remarks:

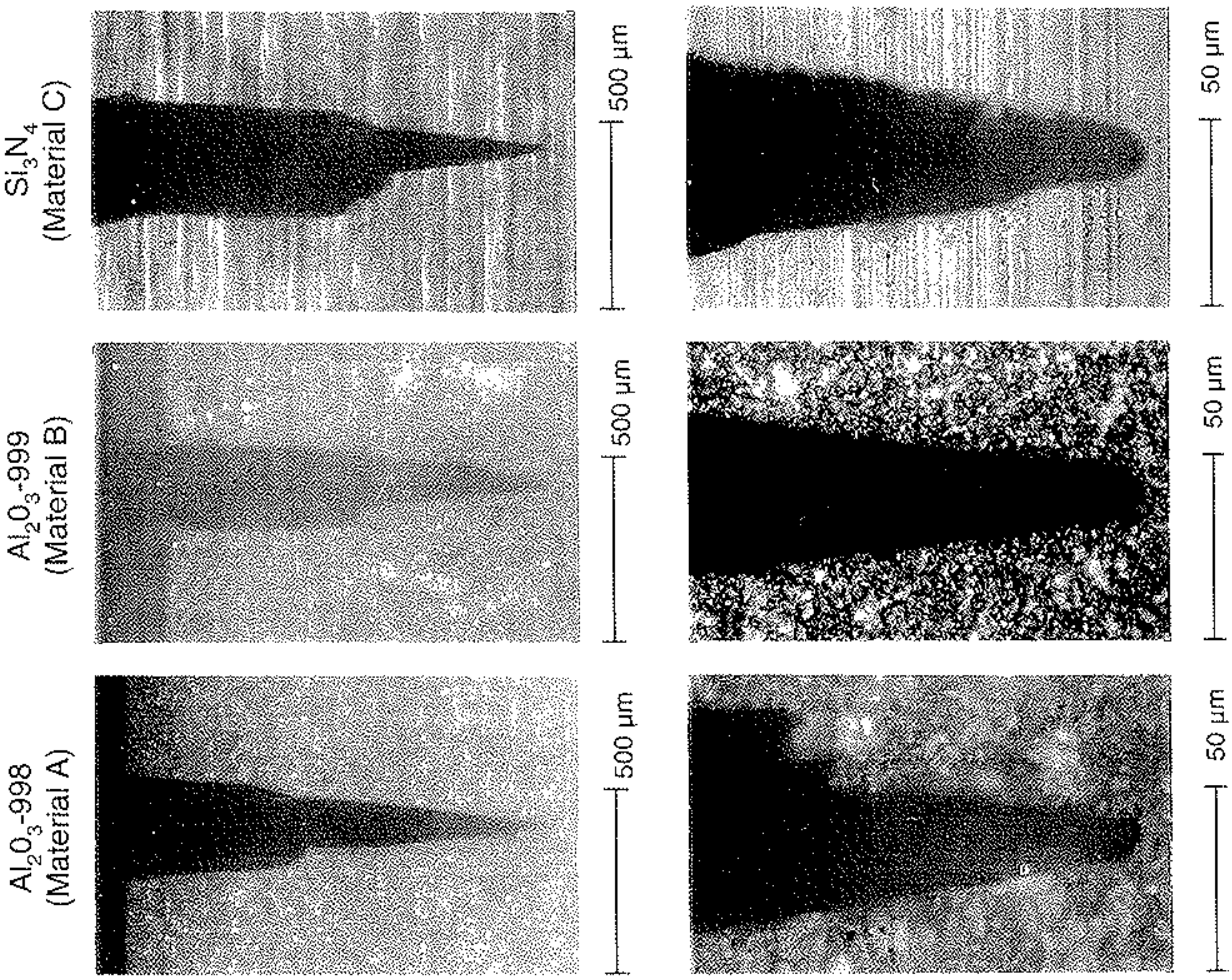
Participant # 26



Remarks:

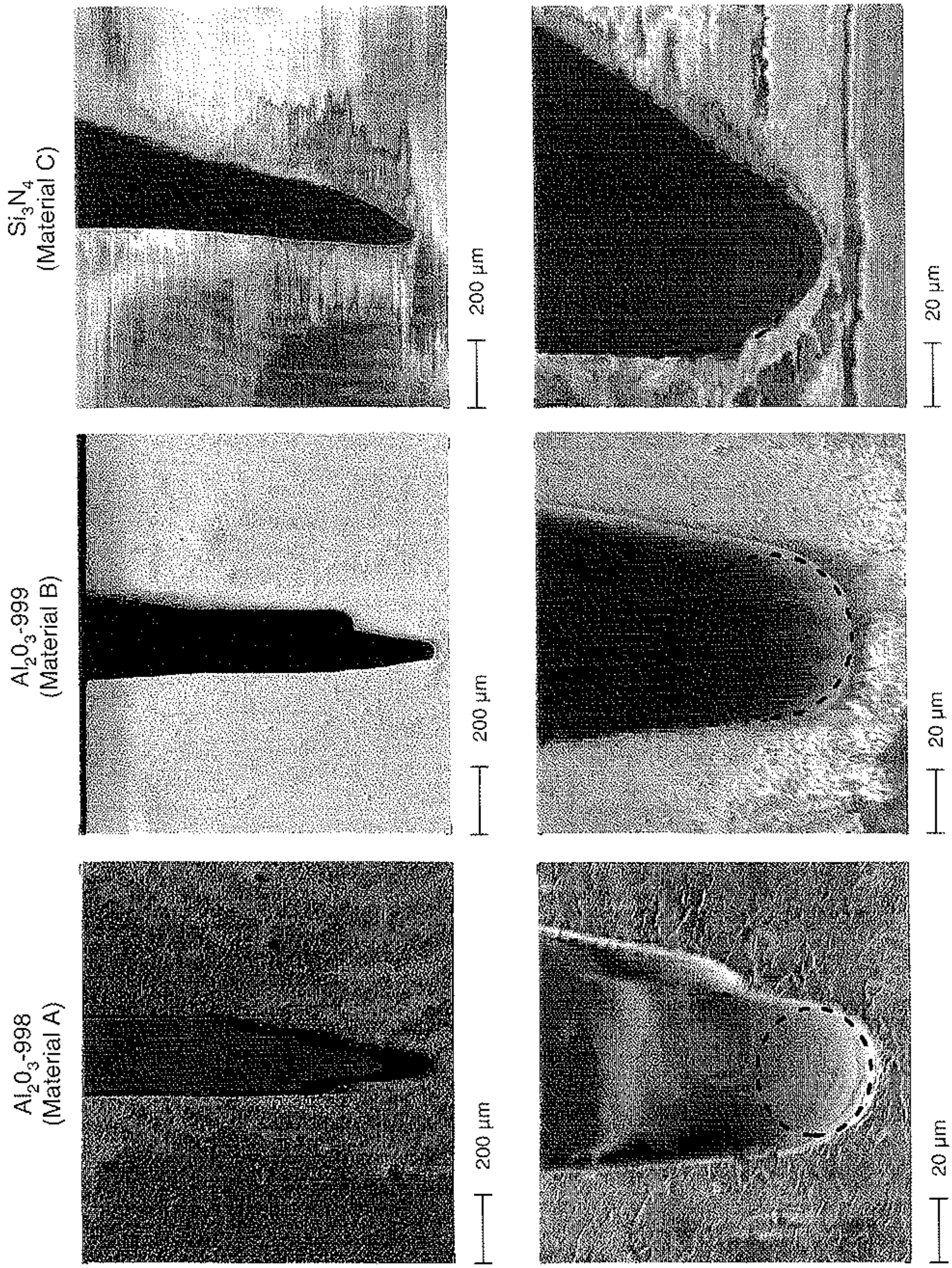
-

Participant # 28



Remarks:

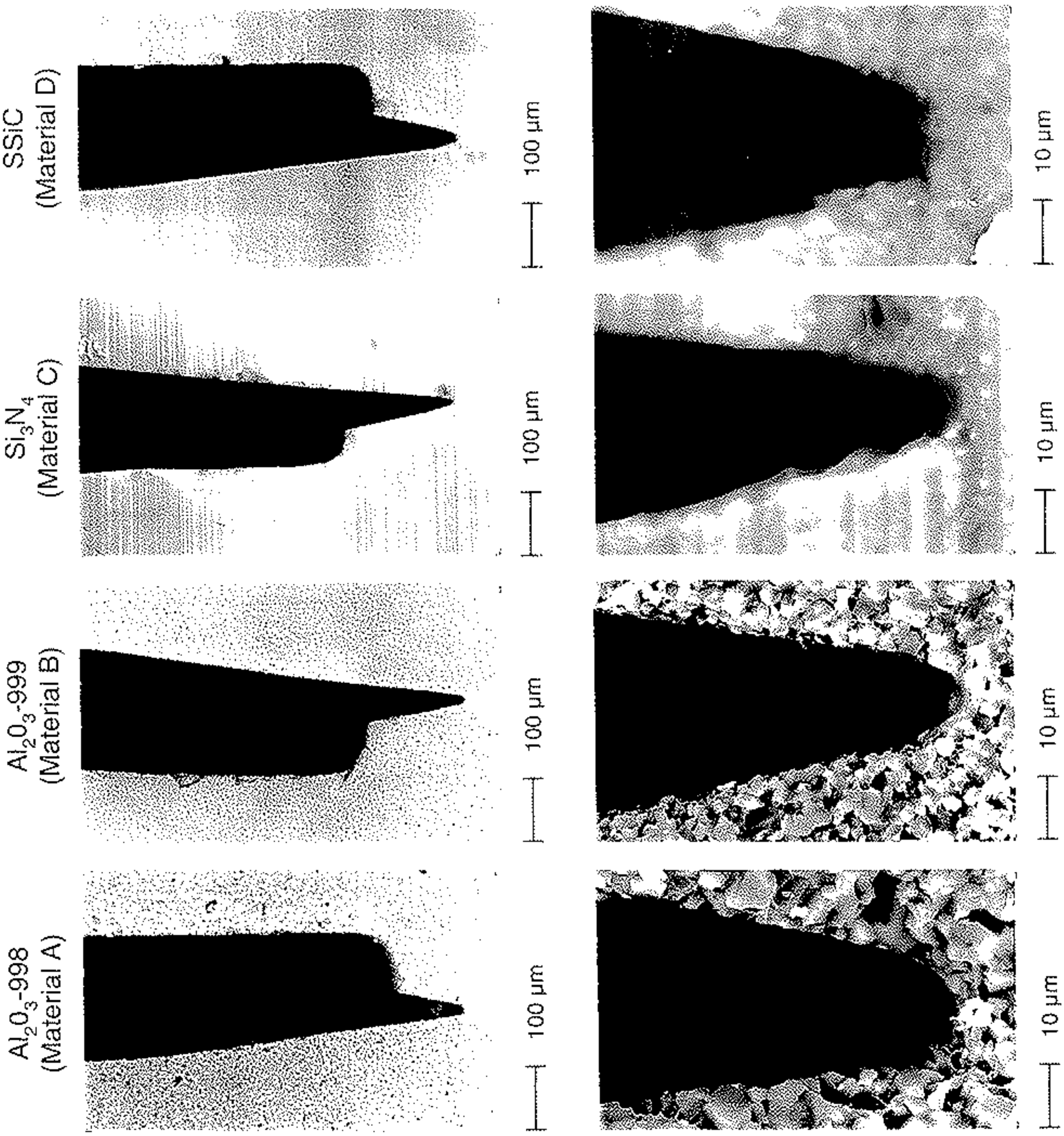
Participant # 29



Remarks:

-

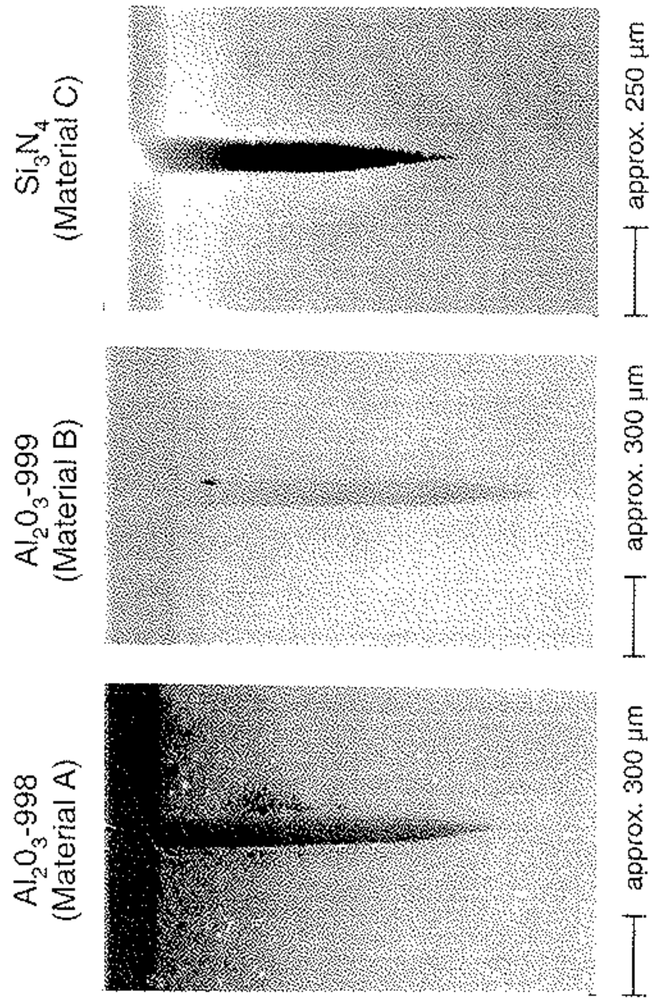
Participant # 30



Remarks:

-

Participant # 31



Remarks:

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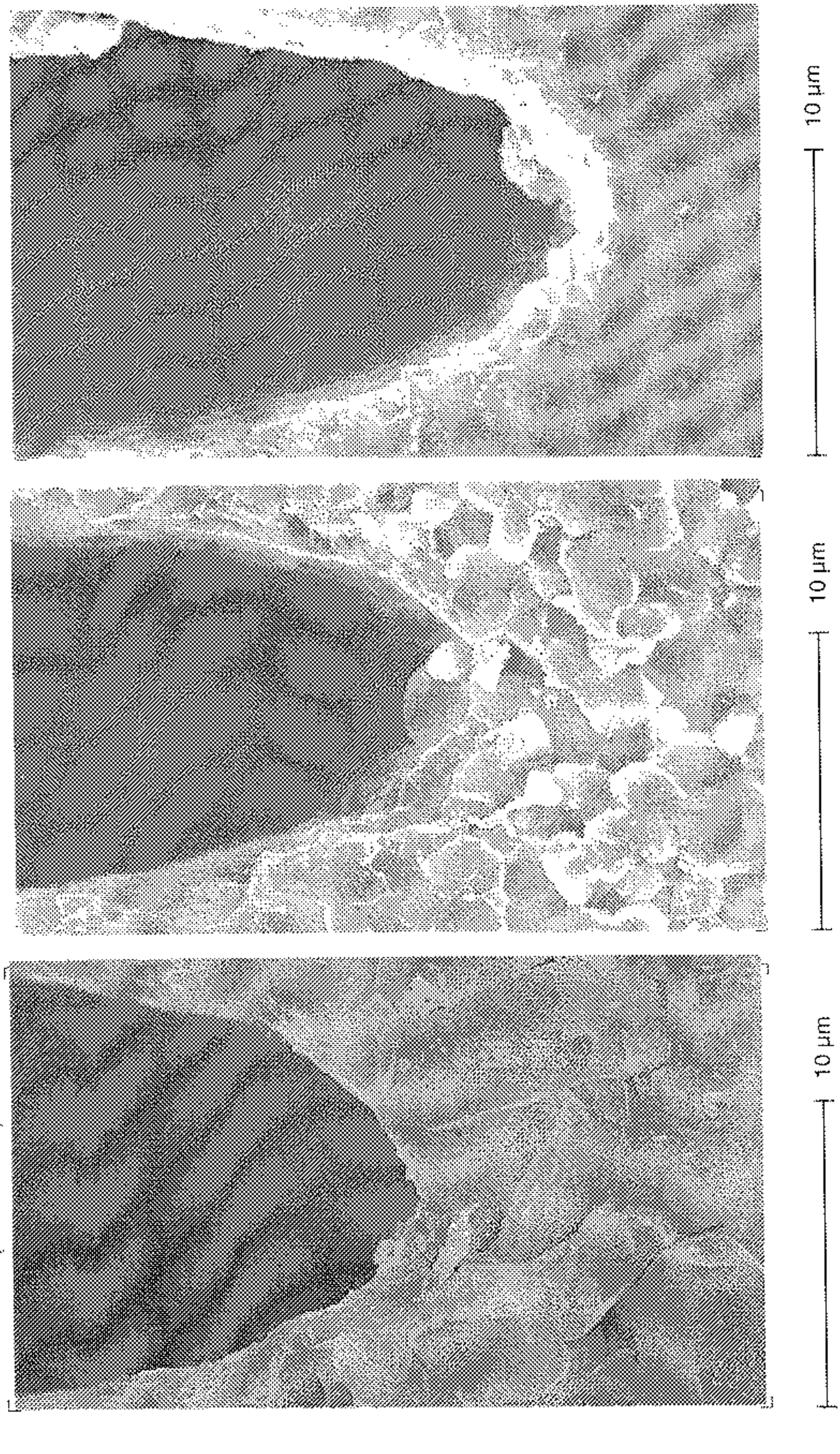
Participant # 32



Si_3N_4 (Material C)

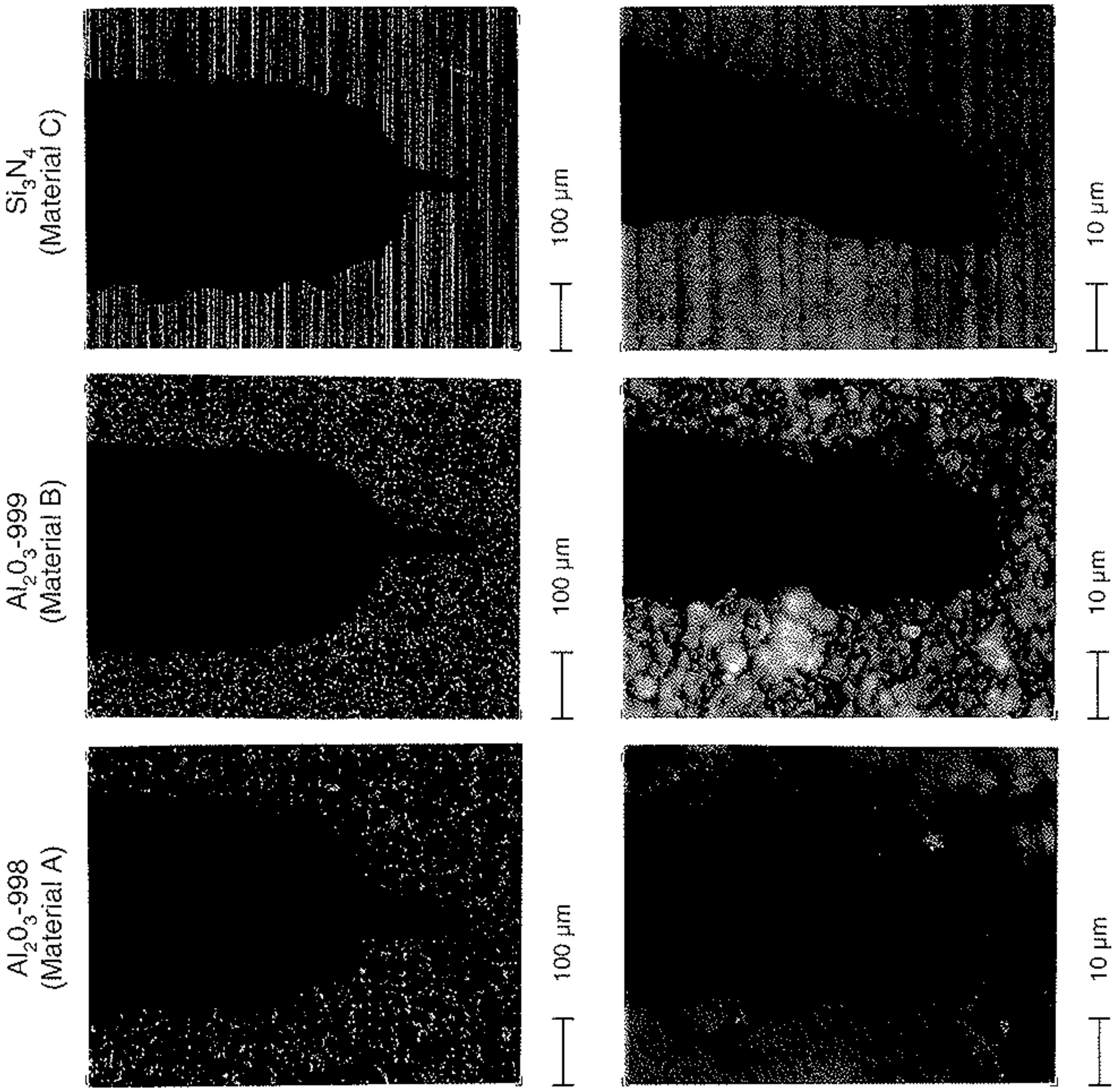
Al_2O_3-999 (Material B)

Al_2O_3-998 (Material A)

Remarks:

-

Participant # 34



Remarks:

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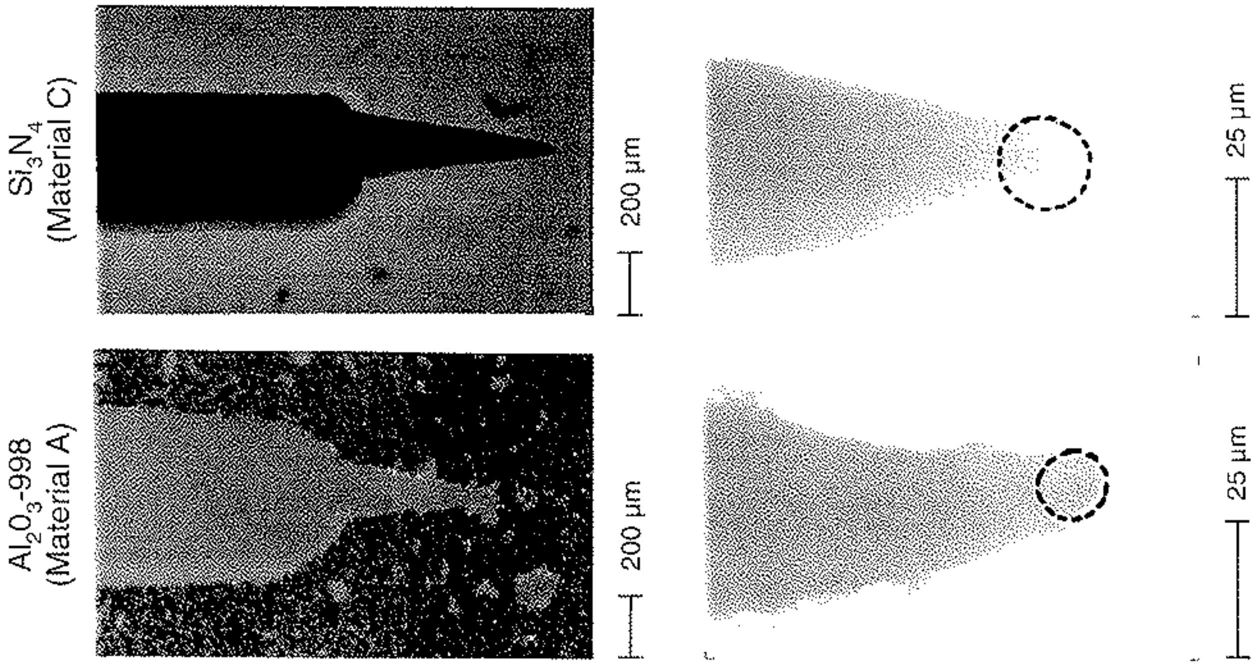
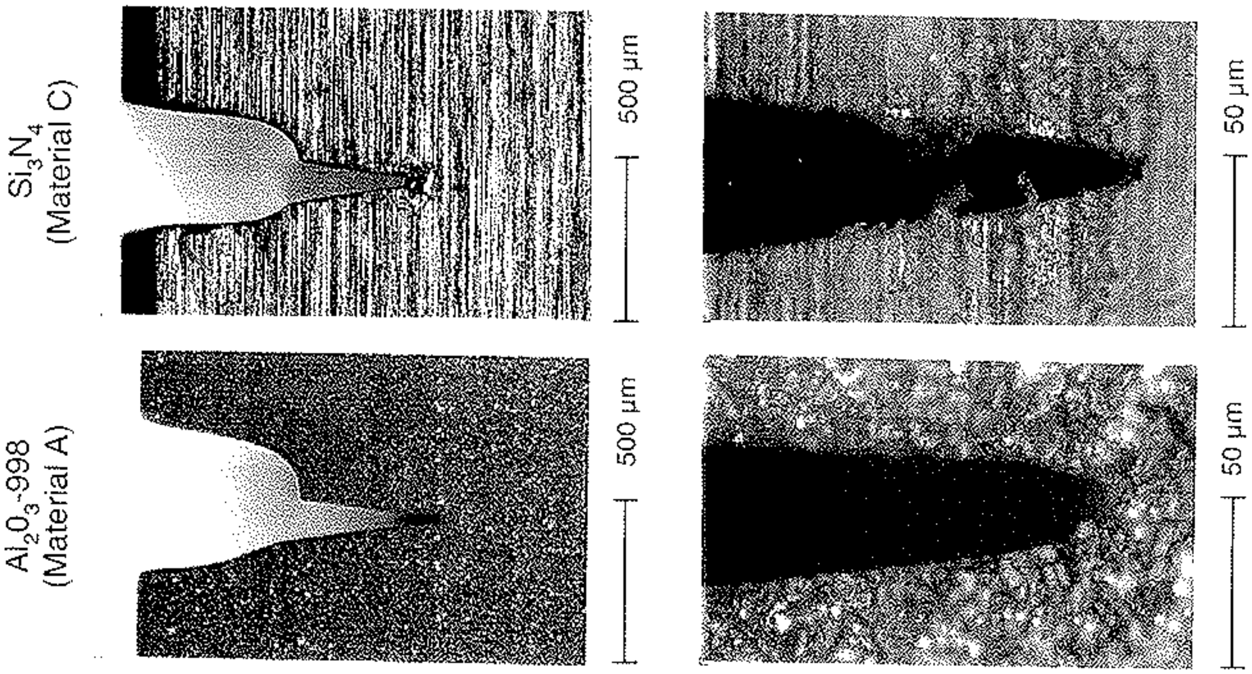
Participant # 35



Remarks:
The dashed lines were drawn by the participant.

Participant # 36



Remarks:

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Appendix A4: Comments by participants to the SEVNB method

Most participants sent some comments and opinions to the SEVNB method as asked for in the instructions. This appendix contains all comments and opinions. For better readability some were shortened.

Remark: Some comments and opinions were translated into English as accurately as possible by the organizer of the round robin.

Participant # 1

The repeatability coefficient of variation is small compared with other methods for example SEPB. The fracture toughness measured with the SEVNB method compares very well with values from the SEPB and CN methods as long as they are not influenced by an R-curve effect. Agreement is excellent with small crack data R-curve measurements.

"Valid" SEVNB fracture toughness values can be measured if the V-notch width is less than about 4 to 5 times the size of a major microstructural feature for example the grain size. For all tested materials it was possible to polish V-notches with a tip width between 5 µm and 20 µm.

The SEVNB method proved to be user-friendly, easy to conduct and therefore is a potential standard test method.

Participant # 2

The method can easily be applied with a machine; own pretests show a good reproducibility. We would apply this method also in the future; this however requires a revision of our old data sheets, since with the old method generally higher values were achieved.

Problem: Due to the wear of the razor blade it is not easy to follow up respectively to reach the exact notch depth.

Scattering of batch material B is very high; why is not clear, noticeable are only the very high values for the basic notch diameter.

Participant # 3

The major problems occurred using the SEVNB, are simple to solve. The difficulties found here to achieve the notch depth required couldn't be attributed to the test method procedure, but to small improvements required in the equipment/jigs available in the present institution.

Compared to other test methods used to measure fracture toughness of ceramic materials, the SEVNB allows a very good control on the notch execution. In my opinion, the SEVNB is quite friendly to be used. Looking at the data, the reliability is quite good. Comparing the data of Si_3N_4 tested by SEVNB and SCF, it is possible to observe that SEVNB showed a more consistent result.

Participant # 4

......My only comment is I think the grit size of the polishing paste should be limited to below the ceramic grain size – to avoid introducing extrinsic (polishing) defects......

Participant # 5

All together it can be said that we've made very good experiences with an equipment similar to that suggested by VAMAS, and constructed solely for the purpose of inserting notches without prior sawing of the notch........

Participant # 6

The instructions were clear and easy to follow. Producing notches by hand was simple (after a little practice) and surprisingly effective. However, only Alumina was tested, the prospects of having to spend four hours at a time to produce notches in Silicon Nitride by hand is not appealing. I have now built and tested a machine similar to yours and the initial results are promising.......I completed the SEVNB testing quite some time ago without problems.........

Participant # 7

--

Participant # 8

The instructions ...... were generally clear and very easy to follow. The time needed for the exercise was reasonable, and with more experience this would be a relatively fast test method. A few observations were made during the work. It was necessary to remove the razor blade in order to check the progress of the notch, and that led to problems with reseating the razor blade properly into the notch. A couple of times this resulted in a small additional notch at the root of the saw cut. This could more easily be avoided when some experience was gained and a deep enough notch was made before removing the razor blade the first time........

Participant # 9

...... We feel that this method is quite easy and shorter in time than other standard methods, as SENB or SCF. The V-notches were produced in about 1 ½ hour and in the Si_3N_4 we spent ½ hour more. Our main problem was cutting the samples with a diamond saw, because the required dimensions (ranging from 0.5 to 0.6 mm) were so small, and we had to cut them very carefully.

Participant # 10

First letter (after SEVNB round robin)

a. The method seems to be fairly easy and straightforward to do. With more experience, we probably can get the saw notches even more precise and sharp.

b. We did see evidence of an 50-150 μm "initiation" at the root of the notch. Twist and wake hackle marks were present which converged as the crack developed and extended away from the notch root. In some cases, particularly for the coarse grained alumina, material "A", the notch root had steps or little jogs suggesting that the small cracks had popped in on slightly different planes and orientations before they converged to one main crack.

I confess that I still have concerns about the SEVNB method. My worries are rooted in the belief that one important criterion for a proper ceramics fracture mechanics test is that it has a sharp crack. (This is analogous to a paradigm of metal fracture testers: "the only good precrack is a fatigue precrack") Our results from this project seem to be fairly credible, but I wonder if the method is beneficiary of two offsetting errors:

1. The blunt crack tip requires a slight overload to cause crack extension, the classic behaviour and the reason why all old SENB data is too high. In the case of the sharper

SEVNB specimens in the new work, the overload is less, say 5-10 %, but it still causes an overestimate of toughness.

2. The true SEVNB crack lengths once the cracks have initiated and popped in away from the notch root are slightly longer than the depth of the notch (which is used in the SEVNB calculated Kc). Therefore, the calculated apparent toughness is underestimated by 5-10 %.

The method may not strictly satisfy the strict (idealistic?) requirements of a true fracture toughness test. On the other hand, the results are credible, easily obtained, and the method may be the beneficiary of offsetting errors at the notch root. Beware of slow crack growth effects, however!

Second letter (after additional analysis by the participant)

We tried to detect stable crack extension at the notch roots in materials A and B. Is it possible to correct the crack length for it under lab ambient conditions? We were not successful and we are disappointed. We can see SCG-intergranular fracture markings in "B", but they were mixed with some other features such as geometric tilting of the crack, or crack start up on several places, or machining damage. We had no success with "A".

Most of this letter is not intended to be critical of SEVNB. I just want to warn you about the SCG effect on toughness in the aluminas. SCG will affect both SEVNB and SCF, as well as SEPB or other methods I am sure.

Participant # 11

.....The method is reasonably simple; assuming the V-notch can be introduced using an appropriate machine, and could be taken into account as possible standard. However a definite answer to this question depends on the results of the R.R. ......

Participant # 12

The notch polishing procedure worked fine with PZT, Y-TZP and Ce-TZP, with a notch tip width of 3 to 8 µm for both TZP. Problems arose for Al_2O_3 and Si_3N_4: the finer razor blade was strongly eroded and was damaged during notch cutting in a way that blade fragments got stuck in the notch tip. These fragments inhibited further notch polishing and resulted in the premature breaking or crack growth in several specimens. In fact, in these materials, we could produce either no or only a short, sharp notch segment with our second notch cutting step. This is probably due to the fact that we collected our notch cutting experience (and of course, our processing parameters) only on PZT and TZP materials where it is quite easy to make sharp notches.

Conclusions about the SEVNB method

The method is very reliable and easy to use. It works in an excellent way for a few materials. It is much more difficult to produce fine notches in harder materials. More information concerning several parameters of the machine should be made available, i.e. the degree of alignment of the blade and the moving fixature and the maximally allowable vertical oscillation of the blade due to misalignment and blade erosion. A maximum notch angle should also be prescribed.

Participant # 14

...... Looking forward I hope that we will be able to get good V-notches soon (an experience is necessary) and the method will be the most useful one in the practice of fracture toughness testing.

Participant # 15

...... In my opinion the SEVNB method is really nice and reliable (and also very cheap). Actually we are using it as a standard in our laboratory for fracture characterization of monolithic ceramics and ceramic matrix composites. I hope the results of this round robin will be good enough for wide standardization of this method.

Participant # 16

First letter (after SEVNB round robin)

Notwithstanding the limited practices, this SEVNB method using razor blade with diamond paste gave rise to much improvement in fracture toughness testing over the previous SEVNB technique that used a v-shaped diamond wheel [1]. .... As a result of very fine notch widths of about $10x10^{-3}$ mm obtained by the new SEVNB method, the fracture toughness resulted in good agreement between the SEVNB and SEPB methods, as verified for the silicon nitride material. It is believed that this range of notch widths of > $10x10^{-3}$mm is almost equivalent to a sharp precrack. It is also noteworthy that the scatter in K_{Ic} in the SEVNB method was small with coefficient of variation ranging from 1.7 % (AD998) to 6.6 % (Si_3N_4). The SEPB method usually results in a coefficient of variation of about 10 %.

As compared with the SEPB method, the SEVNB method, however, has some drawbacks. The major drawback is that it takes too much time in specimen polishing .... (4 h for Si_3N_4). These drawbacks should be improved through a parametric study on polishing time as functions of several variables........ It is also recommended that instead of the narrow range of final v-notch depth of 0.2 W to 0.3 W specified in this round robin, a wider range of notch depth ranging from 0.2 W to 0.6 W (W = specimen height = 4 mm) be used for more flexibility....... The effect of v-notch depth on fracture toughness should be carried out for several different advanced ceramics to give a better guidance in choosing appropriate v-notch depth to give a minimum preparation time. If the drawbacks can be remedied, the new SEVNB method will be a promising, user-friendly technique in evaluating fracture toughness of advanced ceramics. (Finally, the answer to the questions in NOTE 1 should be made).

NOTE 1: Based on our long experience on fracture toughness testing, good agreement in fracture toughness of advanced ceramics exhibiting a flat R-curve has been observed between the indentation strength (IS) technique (although not rigorous in fracture mechanics concept) and other estimation methods (SEPB, CN, or SCF, etc.). Because of the discrepancy between the IS and the SEVNB methods observed for the AD998, we additionally determined fracture toughness by using the SEPB method for three AD998 specimens. The value of fracture toughness was 4.40 ± 0.06 MPa/m with the precracks ranging from 1.6 to 2.3 mm. This value appears to be very high compared to that determined by the SEVNB (3.60 MPa/m) but to be very consistent with the value (4.37 MPa/m) by the IS technique. An ambiguity thus occurs as to why the discrepancy exists between the SEVNB and the SEPB or the IS method, particularly considering the fact that the material reveals no rising R-curve. Is the evaluation of R-curve done by the indentation techniques not appropriate for this material? Or, does the SEVNB technique tend to give some underestimated fracture toughness for the material? The answer should be sought.

Second letter (after additional analysis by the participant)

......Therefore, the SEVNB method can give a most accurate fracture toughness of a material, with little influence of R-curve behaviour. Any fracture toughness methods except for the SEVNB method would include a certain degree of R-curve effect, if a material exhibits a R-curve (you also thought that "with the SEVNB method the beginning or almost the beginning of a R-curve can be measured" (from your Email dated 7/20/98). I would think this is very important both in our understanding of fracture toughness of advanced ceramics and in developing appropriate fracture-toughness test methodologies. In such a context, the SEVNB method can play a crucial role!

Participant # 17

Although we designed a new polishing machine with high quality components we are not fully satisfied with the width of the notches, compared to the 2 µm width for ZrO_2 reported by the organizer. The most trouble we had with Si_3N_4 and reached a notch width of only 20 µm, best result for Al_2O_3 with 4 µm. We think that it will be necessary to bring together the experience of all participants in order to create a very detailed guide for the polishing machine design and polishing procedure......

We are convinced that the SEVNB is an excellent technique and will become a standard, when the creation of the notches is optimized. In our laboratory we will use the SEVNB for K_{IC} measurement exclusively. The main advantage is the applicability to nearly every ceramic material, which is not possible with the other methods. In this sense, the SEVNB method can be called a very user-friendly technique. One drawback might be the applicability to super-fine grained ceramics (< 1 µm grain size). This aspect should be experimentally and theoretically analyzed.

Participant # 19

.... It seems that the manual notches are quite good and that the results are consistent. We found the method interesting but quite long time consuming, mainly for polishing by razor blades for the harder material. I believe that applying a polishing by machine the effort will be reduced......

Participant # 21

There seems to be a great difference in quality of razor blades available commercially.

Participant # 22

The SEVNB fracture test with notch radii < 10 µm is suitable to get correct values of K_{IC} with 5 or more specimens on condition that the testing time does not exceed 1 second with respect to materials showing undercritical crack growth. Using a crosshead speed of 0.5 mm/min the loading rates in this test were about 1 MPa√m/s. We prefer a testing rate of 1600 N/s to get loading rates of >30 MPa√m/s.

Participant # 23

Participant # 24

We deem the SEVNB method suitable for the determination of K_{IC}-values. However, the cutting of the notch requires some experience even when a machine is used. The weak point in our opinion is the different and partly poor quality of the razor blades (asymmetric grinding, oblique angled grinding). Inspite of these obstacles this method is now standard in our company.

Participant # 25

I used two razor blades, one for rough polishing and one for finishing. However, the notch width was not small (23-32 µm), maybe caused by my hand polishing technique. That gives the average values a little (6-8 %) larger on SEVNB than SEPB on materials B and C. On material A, SEPB showed very high value compared to SEVNB because it was very difficult to catch the pop-in signal on the pre-cracking. Then, the crack extended with excess pre-crack loading as the stable crack growth. The value on SEPB became larger with R-curve effect, and in this case SEVNB is quite useful to obtain the initial fracture toughness value of the material.

Participant # 26

The results from SEVNB in comparison to the results from SEPB have shown less standard deviation and a lower K_{IC} as it was expected for small V-notch tips.......

The SEVNB method is simple, easy, fast, cheap and results in believable values for the K_{IC} with small standard deviations. We would prefer using this method in the future, if the method is well known by the industry and is recommended by standardisation organisations like ISO or CEN.

Participant # 28

The SENB method seems to be a relatively easy to perform and friendly in using it. In the case of Al_2O_3-998 the measured mean value seems to be reliable, the standard deviation is very low. In the case of materials Al_2O_3-999 and Si_3N_4 the standard deviation seems to be a little higher.

Participant # 29

It has been verified that the SEVNB method is a very time and cost effective method giving apparently reliable data with a small scatter. The preparation of the V-notches is such simple that it can be carried out at any laboratory........ The fracture surfaces show that the notch grounds were well polished. Either grain pullouts or inherent defects below the surface were the fracture origins. Hence, a single defect caused apparently the fracture. In order to determine accurately the toughness from the crack length, the size and the geometry of these defects have to be considered in the calculations. Adding simply the size of the defect to the notch length is a rough, conservative estimation of the crack length at failure. Although both factors, the reliability and the ease of testing are great advantages, it is recommended that the role of the size and shape of the defects on the toughness values is elucidated before proceeding to claim the method as a standard test method.

Participant # 30

SEVNB method can become a standard test method and can be recommended for inclusion into the drafts of national and international standards. It is advisable to consider the cases of 4-point flexure with spans of 40 and 20 mm and 3-point flexure with a span of 16 mm with the depth of V-notch equal to about 50 % of the height (width W in instruction) of a specimen with a cross-section of 3 x 4 mm^2. To process the results it is feasible to use formulas from ASME Standard "Standard Test Method for the Determination of Fracture Toughness Advanced Ceramics".

Considering the fact that it is sufficient for the V-notch radius to be equal $\leq$ 10 µm, the methods of polishing the V-notch by hand and machine may turn out to be alternative and practicable for general use. Probably it is reasonable to use machine polishing of V-notch in cases when a more exact determination of K_{IC} values is required. Additional experiments are necessary for the determination of the minimum allowable V-notch tip radius and cleared method for measuring it.

When advertising this method, it is necessary to draw attention to the fact that its usage is reliable for any class of materials, since with the V-notch polishing the probability of phase transformations in the stress concentrator zone is much lower than with other methods of fracture toughness testing. For instance, it is common knowledge that rough polishing (which is similar to cutting specimens by the diamond saw blade) results in phase transformations in PSZ and TZP ceramics. The same is observed for zirconia crystals ......

Participant # 31

Participant # 32

1. The method has its advantages in its simplicity.
2. Not needing to polish samples is the major advantage.
3. All results are clearly above crack tip toughness for the respective materials.
4. A much lower scatter in results is obtained than generally found using the SEPB method.
5. I am still unsure whether I would use this method if it became a standard as it would be difficult to compare results between various workers due to notch tip variations.

Participant # 34

Participant # 35

By hand, we have some difficulties to center the notch exactly in the middle of the saw cut. Otherwise, this technique allows to obtain notches having a very small tip radius and therefore to avoid the problems associated with the conventional SENB method. Even if the method needs some training, the notch geometry obtained for the last material which has been polished by hand (Si_3N_4) already appears very good. In fact, we believe that the only limitation of the method is the long time and therefore the high cost to polish by hand the notch which is not really compatible with in plant laboratories. However, this limitation can be hopefully overcome using the machine described in section 3.3.2, which we recommend for the standard.

Participant # 36

The SEVNB method seems to be a promising new method for the reliable determination of the fracture toughness of ceramics. To be emphasized are:

- simple and relatively fast process
- low influence when the notch geometry is measured by different persons
- low expenditure of samples (it may be possible to use leftovers of fracture toughness tests with 40/20-support to carry out the SEVNB method with 20/7-support)
- low standard deviation (material A: 2%, material B: 5%)
- further improvement possible by machine polishing; thereby reducing time consumption and influence of the worker and producing smaller notches.

Email approx. one year after round robin

Some time ago you have machined for me razor blade notches on a filled polymer-derived ceramic material. At the same time we determined the fracture toughness on manually notched specimens (by means of a razor blade), using the SENB and the ICL method. The measurements of the specimen were 25x3x4 mm; the bending test was carried out with a 20/7 support. Following are the results obtained:

SEVNB (manually notched, notch width ≈ 25 µm)	K_{Ic} = 2.05 MPa √m, Std.Dev. 3.5 %
SEVNB (machined notch, notch width ≈ 10-20 µm)	K_{Ic} = 2.05 MPa √m, Std.Dev. 6.4 %
SENB (notch width ≈ 350 µm)	K_{Ic} = 3.0 MPa √m, Std.Dev. 24 %
ICL	K_{Ic} = 2.7 MPa √m

In our opinion this method is very rugged and delivers good results for this material with a low scattering. Considering the structure sizes given within the material it is understandable that no difference can be measured between manually or machine made notches.

Appendix A5: Notch width – Theory and Model

Jakob Kübler [1], Robert Danzer [2], Theo Fett [3], and Rajiv Damani [2]

[1] EMPA, High Performance Ceramics, Dübendorf, Switzerland
[2] University of Leoben, Department of Structural- and Functional Ceramics, Leoben, Austria
[3] Karlsruhe Research Center, Institute for Materials Research II, Karlsruhe, Germany

In the previously applied analysis of SENB-S (S = saw cut) fracture toughness testing it had been assumed that the stress field of a machined notch sufficiently approximates that of a sharp crack. This is evidently not the case because notches have more moderate stress fields than cracks and, therefore, it was found that wide notches yielded high values of "fracture toughness". The original idea for explaining the influence of the notch width on the fracture toughness test results of ceramics was presented by Damani, Gstrein and Danzer [L-A5.1] who suggested that failure is initiated from a small crack in front of the machined notch. To describe the influence of the notch on the stress field, they used a solution presented previously by Fett and Munz [L-A5.2, L-A5.3] to estimate the stress intensity factors of cracks at the root of notches, see Figure A5.1:

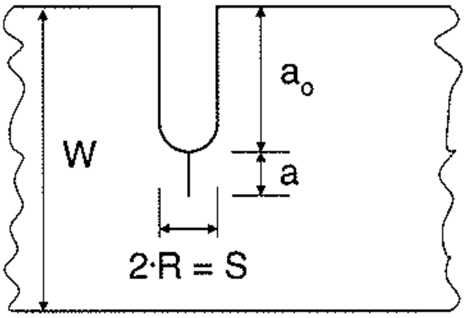


Figure A5.1
Bend bar with a semi-circular edge notch and a small crack in front of the notch tip.

$$\frac{K}{K^*} = \tanh\left(2 \cdot Y_{SC}^* \cdot \sqrt{\frac{a}{R}}\right) \qquad \text{(E-A5.1)}$$

where K is the stress intensity factor of the cracked system, Y_{sc} is the geometric factor of a short crack at the surface of an uncracked specimen (for a straight through crack, $Y_{sc} = 1.12$; in his original work, [L-A5.2], Fett and Munz only described this special case instead of the more general case of equ. E-A5.1) and

$$K^* = \sigma \cdot Y^* \cdot \sqrt{\pi \cdot (a_0 + a)} \qquad \text{(E-A5.2)}$$

is the stress intensity factor of a long and sharp straight through (= edge) crack of length a_0+a loaded in bending and Y^* is the standard solution for corresponding geometric factors also used in the text, equations E-A5.1 and E-A5.2. For

$$\frac{R}{a} \rightarrow 0 \qquad \text{(E-A5.3)}$$

the crack at the notch tip is much longer than the radius of the notch tip, and K* approaches K. As a consequence the standard solution, E-A5.2 of an edge crack can be used. For

$$\frac{a}{R} \rightarrow 0 \qquad \text{(E-A5.4)}$$

K/K* approaches zero, which reflects the shielding influence of the notch. The stress intensity factor is much smaller than expected and the external load has to be increased to get the same value as calculated for a long and sharp edge crack.

In general, the exact geometry and size of the small crack ahead of the notch is unknown but is assumed to be related to microstructural features or machining damage, e.g. grain size, pores or scratches. Damani, Gstrein and Danzer [L-A5.1] suggested a lower and an upper limit for the geometric factor Y_{sc} : $2/\pi < Y_{sc} < 1.12$; the lower limit is the geometric correction factor of a small penny shaped crack (which is a little bit smaller than the geometric factor of a small surface halfpenny shaped crack) and the upper limit is that for a small edge crack.

Damani et al, [L-A5.1] used the property $a(Y_{sc})^2$ as a fit parameter to describe the notch root influence on fracture toughness testing. The crack length a can be estimated from this fit, if we remind that Y_{sc} is restricted between narrow bounds. The resulting variation is in the range of the accuracy when determining the microstructural feature size. Figure A5.2 shows some original results. Plotted is the value of K^* over the square root of R. In the figure, the properties K, a and Y_{sc} are called K_T, δa and Y, respectively. In the figure, Y_{sc} is set equal to the lower bound $Y_{sc} = 2/\pi$ giving a crack length of $\delta a = a \approx 30$ µm. This corresponds to the length of a typical bad sintered grain size. If the upper bound $Y_{sc} = 1.12$ is inserted, the fit line would not change but the size of the crack ahead of the notch tip would be $a \approx 10$ µm, which could correlate with a possible machining damage. This observation confirms the assumption that microstructural features act as cracks at the notch root. K (= K_T) would not change in either of the cases.

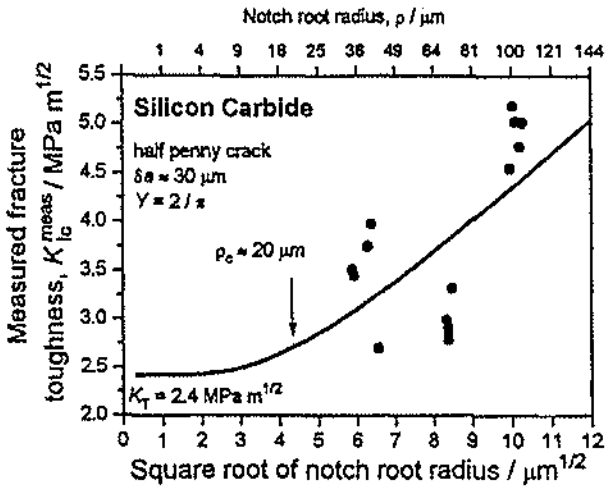


Figure A5.2
Best fit through data points measured with the SEVNB method on a silicon carbide ($K_T = K$).
Source: Damani et al [L-A5.1]

Remark: The silicon carbide used by Damani et al is identical with the one used in the present VAMAS / ESIS round robin.

In a yet unpublished paper, [L-A5.4], Fett proposed a more appropriate solution for the geometric factor: He modelled the behaviour of a very small half-elliptic crack at the root of a notch in a bend beam. Fett found, that the geometric factor depends on the position of the crack front and on the ratio a/R. A halfpenny shaped crack for example has a higher geometric factor at the edge of the crack near the surface than at the deepest point of the crack. Therefore, during loading and if the stress intensity factor reaches the fracture toughness, the crack starts to propagate along the surface of the notch. At the same time the crack changes its shape from a half circle to an elongated half ellipse. With increasing aspect ratio of the ellipse, the geometric factor at the surface decreases up to the point where it reaches the value of the geometric factor normal to the surface of the notch root. Then the crack propagates in the direction of the notch and final fracture occurs. The "equilibrium" shape of the crack (before final fracture occurs) depends on the ratio a/R but does not depend on the initial shape of the crack.

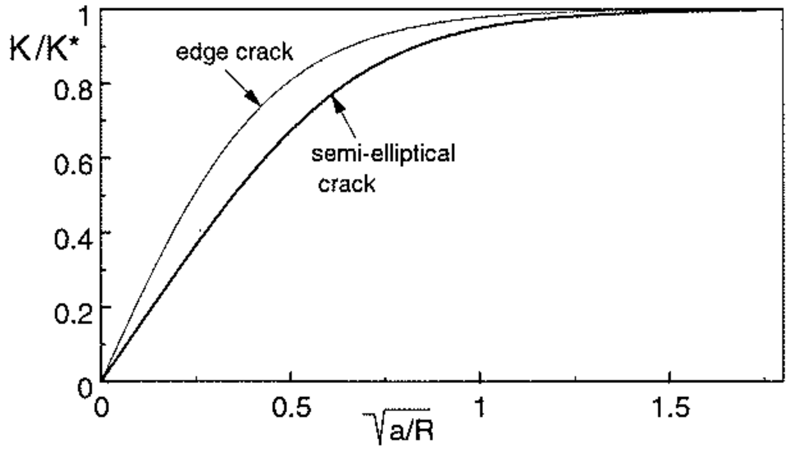
Fett, [L-A5.4], calculated an approximate solution for the geometric factor of a short "equilibrium" surface crack

$$Y_e = 1.12 \cdot g\left(\frac{a}{R}\right) \cong 1.12 \cdot \left(\frac{2}{3} + 0.178\left[1 - \exp\left(-\frac{1.64 \cdot a}{R}\right)\right]\right) \qquad \text{(E-A5.5)}$$

If cracks start from natural defects the lower bound for Y_e is $Y_e = 0.75$ (for $a/R = 0$) and the upper one is $Y_e = 0.95$ (for $a/R = \infty$). Inserting equ. E-A5.5 into equ. E-A5.1 gives:

$$\frac{K}{K^*} \cong \tanh\left(2.24 \cdot g\left(\frac{a}{R}\right)\sqrt{\frac{a}{R}}\right) \qquad \text{(E-A5.6)}$$

This relation is plotted in Figure A5.3 together with the solution for the short edge crack (Y_{sc} = 1.12). Since halfpenny-shaped cracks grow stable into elongated (scratch like) elliptical cracks before they become instable, the critical semi-elliptical crack solution is quite similar to the simple edge crack solution.



(Remark: R = 0.5·S)

Figure A5.3
Representations of equations E-A5.1 and E-A5.6 *Source: Fett [L-A5.4]*

Equation E-A5.6 can be used to estimate a tolerable notch root width. For a measuring uncertainty of 5 % or K/K^*=0.95 respectively would require a notch width of $S < \approx 2a$, [L-A5.1]. This corresponds well with microstructural feature sizes as found by Damani et al, [L-A5.1] and Kübler [L-A5.5].

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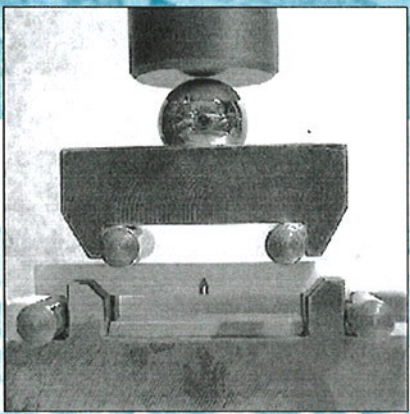

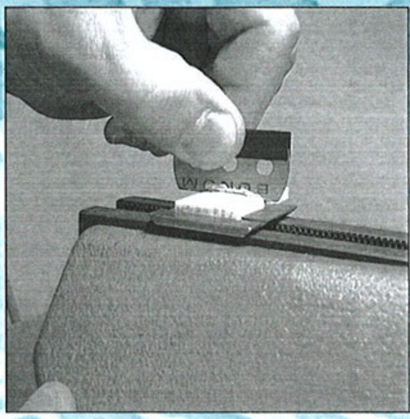

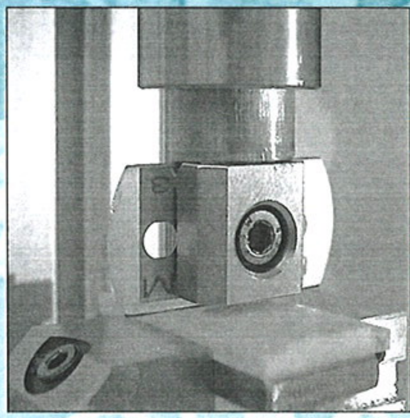

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