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VAMAS Round Robin on Fracture Toughness Measurement of Ceramic matrix Composite

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Date 23 September 1997

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VAMAS Round Robin on Fracture Toughness Measurement of Ceramic Matrix Composite

by

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SUMMARY

Fifteen laboratories in seven countries participated in the VAMAS round robin on fracture toughness of silicon carbide whisker reinforced silicon nitride. The fracture toughness at room temperature was measured by three methods; single edge precracked beam(SEPB), single edge notched beam(SENB), and single edge V-notched beam(SEVNB). The values of fracture toughness, K_{IC}(obtained by the SEPB method) and $K_{c,n}$(by the SENB and SEVNB methods), were measured with four different flexure modes ; 3-p with a 16 mm span, 3-p with a 30 mm span, 3-p with a 40 mm span, and 4-p with 40 & 20 mm spans.

Fracture toughness of the ceramic composite can be measured by the SEPB, SENB and SEVNB methods. However, the SENB values show larger scatter. No obvious effect of flexure mode on the fracture toughness is observed in the SEPB and SEVNB methods except a flexure mode with 16 mm in the SEPB method. Accurate jigs and some experience are required to precrack the SEPB specimens reliably. The $K_{c,n}$ values show saw-cut width or V-notch tip radius dependence, although the profiles of a saw-cut for the SENB method and a V-notch for the SEVNB were different among labs. The SEVNB values from the specimens with a V-notch tip radius less than 30 μm are in the range from 6 to 7 $MPa\cdot m^{0.5}$. These values are similar to the SEPB values. The SEPB and SEVNB were considered to be the proper methods. These results give some recommendations on the fracture toughness measurement which are expected to contribute to future standardization.

Keywords:
Round robin, Composite, SiC(w)/Silicon nitride, Fracture toughness,
SEPB, SENB, SEVNB.

CONTENTS

APPENDICES

1. INTRODUCTION

The Versailles Project on Advanced Materials and Standards (VAMAS) is an international collaboration undertaking prestandardization research[1]. Among the 22 technical working areas (TWAs) in the projects, the TWA #3 "Ceramics" has carried out eleven round robin tests for mechanical properties of advanced ceramics since 1986.

Fracture toughness is one of the important mechanical properties of advanced ceramics for structural use, because it shows the resistance to crack propagation from a defect in ceramics. There are many methods proposed for fracture toughness measurement of advanced ceramics. Among these methods, single edge precracked beam (SEPB) and indentation fracture (IF) methods became Japanese Industrial Standard (JIS) methods at room temperature[2] in 1990, and the SEPB method became the JIS at high temperatures in 1994[3]. As for the ISO (International Standard Organization), ISO/TC206 "Fine Ceramics" has organized the working group (WG)7 in 1996. "Fine Ceramics" is equal to advanced ceramics there. The SEPB method is being discussed as a candidate standard method for the ISO in the WG7. Single edge notched beam (SENB) and SEPB methods became German standard methods in 1991[4].

The Japan Fine Ceramics Center (JFCC) has focused on fracture toughness since 1988 and carried out three round robin tests in the VAMAS project. The first was a room temperature fracture toughness round robin that started in 1989. Three methods were adopted; the SEPB, IF and indentation strength (IS). The materials used were gas-pressure sintered silicon nitride and sintered zirconia-alumina composite. The results from 13 laboratories were summarized and issued in 1990 as a VAMAS Technical Report 9[5] and elsewhere[6]. Subsequently, Quinn[7,8] compiled the results from 18 labs including additional five in the U.S.A.

The second was a high temperature fracture toughness round robin that started in 1990. Three methods were adopted; single edge V-notched beam (SEVNB), SEPB and chevron notched beam (CNB). The material used was a gas-pressure sintered silicon nitride. The results from eight laboratories returned to JFCC by June in 1993 and were issued as a VAMAS Technical Report No.16[9] and elsewhere[10].

Following the above round robins, a fracture toughness round robin by the Surface Crack in Flexure (SCF) method was proposed by Quinn et. al. Two kinds of silicon nitride and a Y-TZP Zirconia were used. The round robin started in 1992 and the results were issued as a VAMAS Technical Report [11].

In 1994, the JFCC started their third round robin on fracture toughness of ceramic matrix composite. The material employed was a silicon carbide whisker reinforced silicon nitride ($SiC(w)/Si_3N_4$), which has being developed as high fracture toughness and high strength material. Three methods were adopted in the round robin; the SEVNB, SENB and SEPB. The SEVNB and SENB methods are considered to have potential to become standard. The objective of this round robin is to evaluate fracture toughness test methods. The fracture toughness is considered to show saw-cut width or notch tip radius dependence due to difference in stress concentration and machining damage at the bottom of a saw-cut or notch tip[9,10]. Investigation of the notch radius and saw-cut width dependence of fracture toughness is another objective of this round robin. This report is based on the results from the 15 labs in seven countries.

2. MATERIALS and SPECIMENS

Silicon carbide whisker reinforced silicon nitride[12] was used for this round robin. The material was manufactured by Japan Metals and Chemicals Co.,Ltd., and contained 20 vol% of the whisker. This composite was formed by slip casting method using a slip containing silicon nitride powder[#1], silicon carbide whisker[#2] and sintering additives[#3], mixed with water and deflocculant. The average diameter and length of a whisker were 1.3 μm and 50 μm, respectively. The green compacts were dried and then sintered at 1825°C for 2 hours at 1 MPa of nitrogen.

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[1] grade;SNP-8S, Japan Metals and Chemicals Co. Ltd., Tokyo Japan.

[2] grade;TWS-400, Tokai Carbon Co., Tokyo, Japan.

[3] mixtures of yttria, alumina and cordierite.

Main properties of the material are shown in Table 1. The density measured by the Archimedes method was 3.20 x 10^3 kg/m^3, showing to be an almost full dense material. Flexural strength, measured with a 3-point fixture and a span of 30 mm[13], was 880 MPa at room temperature. The fracture toughness measured by the Indentation Fracture method[3] with the indentation load of 98 N was 6.2 MPa· $m^{0.5}$.

As shown in Fig.1, SiC whiskers distribute randomly and in parallel to one plane of the plate. This plane is parallel to that of molds of plaster of Paris, which was used for forming the composite by slip casting.

Flexural bar specimens for fracture toughness measurements were machined at the JFCC from billets with dimensions of 70 x 50 x 5 mm. The obtained specimens had dimensions of (4.0±0.1) x (3.0±0.1) x 48 mm. The whiskers mainly distributed in parallel to the narrow 3 x 48 mm face. The parallelism[14] of upper and lower surfaces was better than 0.01 mm. The roughness of top, bottom and side surfaces was not more than 0.2 mm Ra, specified in JIS B 0601[15]. The participating labs precracked a specimen and introduced a saw-cut and/or V-notch into a specimen.

3. EXPERIMENTAL PROCEDURE

3.1 Outlines of Round Robin

Fifteen laboratories took part in this round robin. They are the representative laboratories in the field of advanced ceramics from Belgium, Germany, Italy, Japan, the United Kingdom, Switzerland, Canada and the United States of America (Table 2).

The experimental procedure for this round robin was specified in detail in the instructions[Appendix-1], which were sent to the participants with 30 specimens.

In order to assess methods to measure fracture toughness of ceramics matrix composite at room temperature, following three methods were adopted.

Single Edge Pre-cracked Beam (SEPB) method.
Single Edge Notched Beam (SENB) method.
Single Edge V-Notched Beam (SEVNB) method.

Table 1. Properties of SiC(w)/Si_3N_4, Kryptonite™

Manufacturer	Japan Metals & Chemicals Co.,Ltd.
Main Component	Silicon Nitride
SiC-whisker	20 vol%
Density	3.20×10^3 kg/m^3
Porosity	less than 0.1%
Flexural Strength	880 MPa (RT)
	880 MPa (1000°C)
	490 MPa (1100°C)
Fracture Toughness	6.2 MPa$\cdot$m$^{0.5}$ (IF method; 98 N)
Thermal Expansion Coefficient	4.1×10^{-6} °C^{-1} (RT to 1200°C)
Thermal Conductivity	19.2 W/m·K (RT) 14.0 W/m·K (1200°C)
Thermal Shock	950 °C

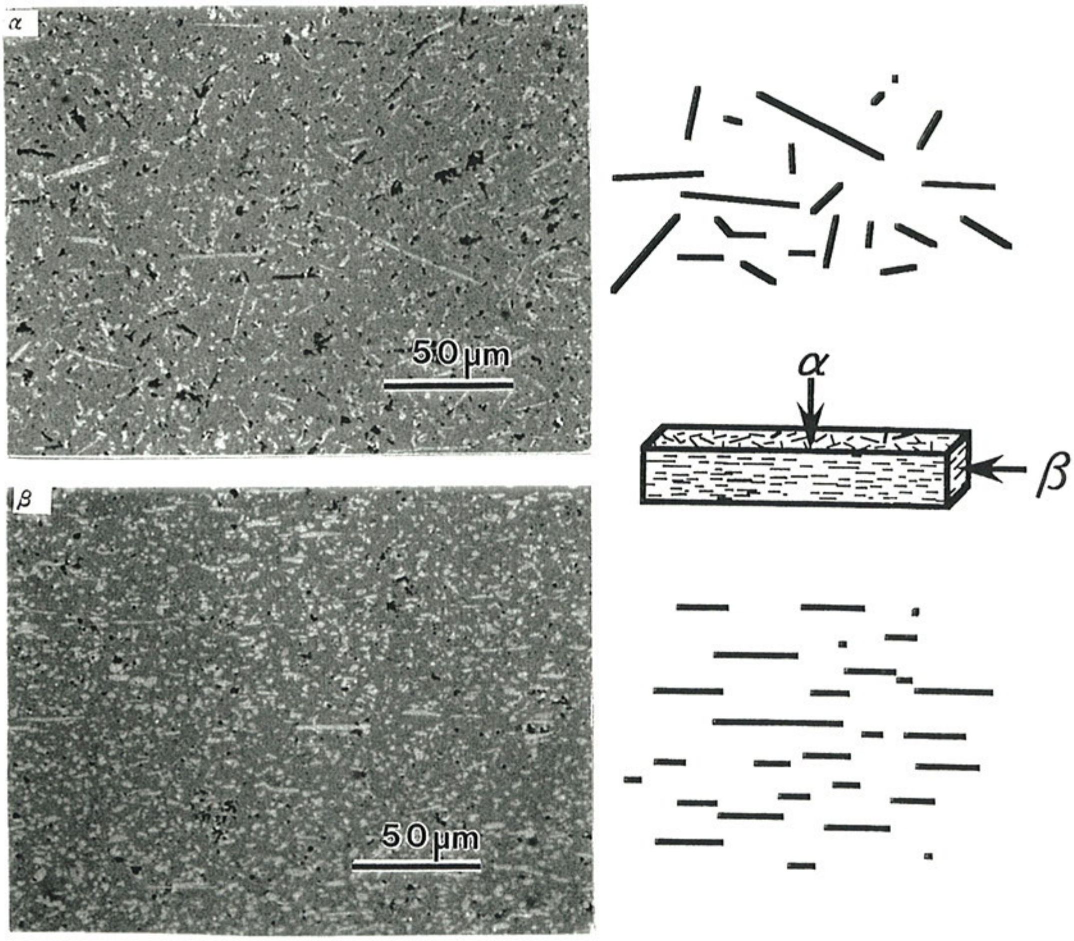


Fig.1. Optical Microphotograph of SiC(w)/Si_3N_4 Showing Whisker Distribution.

Table 2. Participant List for Composite Fracture Toughness Round Robin.

Dr. Philippe Descamps	Belgian Ceramic Industrial Research Center, Mons (Belgium)
Dr. Edith Rudolph	Federal Inst. for Materials Res. and Testing, BAM, Berlin (Germany)
Dr. Thomas Hollstein	Fraunhofer-Institute für Werkstoffmechanik, IWM (Germany)
Dr. Goffredo de Portu	IRTEC, Faenza (Italy)
Dr. Mineo Mizuno	Japan Fine Ceramics Center, Nagoya (Japan)
Dr. Sung Choi	NASA-Lewis Research Center, Ohio (U.S.A.)
Mr. Shuji Sakaguchi	National Industrial Res. Institute of Nagoya, Nagoya (Japan)
Dr. Hidehiko Tanaka	National Inst. for Res. in Inorganic Materials, Tsukuba (Japan)
Dr. Roger Morrell	National Physical Laboratory, Teddington (United Kingdom)
Mr. Tetsuro Nose	Nippon Steel Corporation, Kawasaki (Japan)
Mr. Jakob Kübler	Swiss Federal Research Labs EMPA, Dübendorf (Germany)
Dr. S. Lauf	University of Stuttgart, Materials Testing Office, Stuttgart (Germany)
Dr. Roberto Dal Maschio	University of Trento (Italy)
Dr. Thomas W. Coyle	University of Toronto, Ontario (Canada)
Prof. Isa Bar-on Dr. Kyo Chu Dr. V.Champagne	Worcester Polytechnic / U.S.Army Res. Lab, Mass. (U.S.A.)

All the participants were required to measure fracture toughness by the SEPB method. Moreover they were required to do the measurement by the SENB and/or SEVNB method. They chose at least one of the following fixture modes.

3-point flexure with a span of 30 mm.
3-point flexure with a span of 40 mm.
4-point flexure with spans of 40 and 20 mm.

Ten specimens were used for measuring fracture toughness in each condition. The crosshead speed was 0.5 mm/min. Each lab introduced a precrack and a saw-cut and/or V-notch into the specimens and then measured the fracture load. The results were sent back to the JFCC to be analyzed.

3.2 Methods for Fracture Toughness Measurement

Measurement procedures of the SEPB, SENB and SEVNB methods are schematically shown in Fig. 2. The difference in the three methods derives from the different kind of defects introduced into the specimen. They are a pop-in crack, saw-cut and V-notch, respectively[16].

3.2.1 SEPB Method

The SEPB method uses a pop-in crack. Specimens were precracked from a Vickers indent or saw cut on the 3 mm wide face. A bridge-anvil was used for precracking the specimen. The precrack was usually dye-penetrated and then dried for measurement of the precrack length. Subsequently the specimen was fractured. The crosshead speed was 0.5 mm/min.

This procedure is based on JIS R 1607[2] and the similar specifications are shown in DIN 51 109[4].

3.2.2 SENB and SEVNB Methods

The single edge notched beam(SENB) method generally uses a saw-cut with the width of usually more than 150 μm. The saw-cut width is due to blade thickness of a diamond wheel which is commercially available.

The SEVNB uses a specially prepared diamond wheel having a V-shaped tip with very small root radius from 10 to 40 mm. The fracture occurs from the V-notch tip so that the fracture toughness shows quite small scatter[16].

Instead of a V-shaped diamond wheel, some participants used metal razor blade with a diamond abrasive slurry for introducing a V-notch into a specimen. In this case, the tip usually does not show so round as that machined by a V-notch wheel.

3.2.3 Calculation of Fracture Toughness

In this report, the fracture toughness (K) for a notched specimen is termed $K_{c,n}$, which is distinguished from K_{IC} as measured with a precracked specimen.

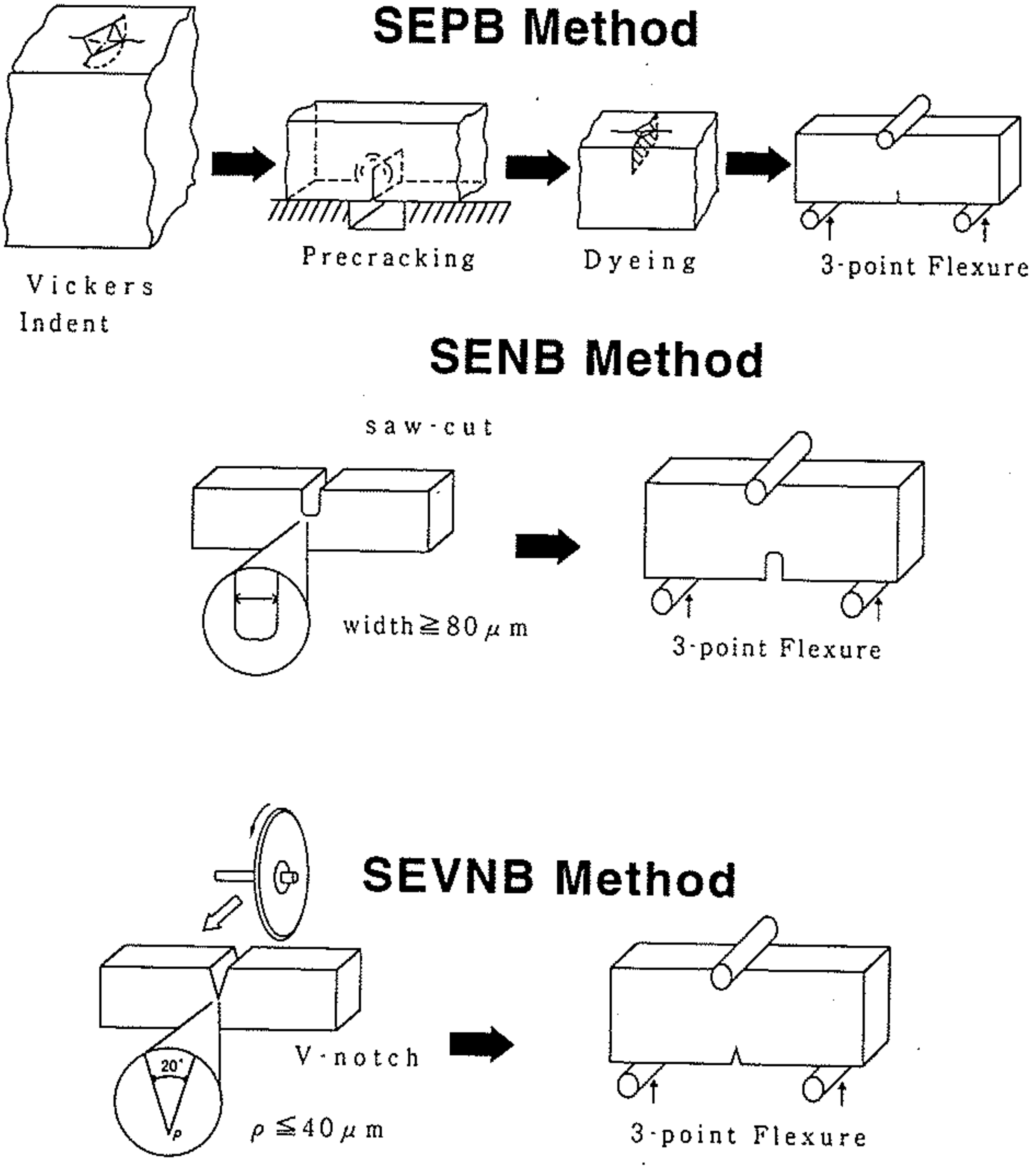


Fig. 2. Measurement Processes of the SEPB, SENB and SEVNB Methods.

In case of a 3-point flexure with a span of 30 mm, 40 mm or 16 mm, K was calculated according to eqns.1, 2 and 5; eqns.1,3 and 5; and eqns.1, 4 and 5, respectively[2, 9,10]. The equation for 3-point flexure with 30 mm span was used for calculation of $K_{c,n}$ for 3-point flexure with 40 mm as shown in the instructions[Appendix-1] , because the F(α) versus α curve for a span to width ratio of 7.5 is almost the same as that for a ratio of 10[17].

$$K = \frac{PS}{BW^{1.5}} \cdot \frac{3F(\alpha)\,\alpha^{0.5}}{2} \quad \text{------------------------}(1)$$

$$F(\alpha) = 1.964 - 2.837\alpha + 13.71\,\alpha^2 - 23.25\,\alpha^3 + 24.13\,\alpha^4 \quad \text{--------}(2)$$

$$F(\alpha) = 1.972 - 2.746\,\alpha + 13.44\,\alpha^2 - 22.84\,\alpha^3 + 23.86\,\alpha^4 \quad \text{-------}(3)$$

$$F(\alpha) = \frac{1.99 - \alpha(1-\alpha)(2.15 - 3.93\alpha + 2.7\alpha^2)}{(1+2\alpha)(1-\alpha)^{1.5}} \quad \text{----------------}(4)$$

$$\alpha = a/W \quad \text{------------------------}(5)$$

In case of a 4-point flexure with spans of 40 and 20 mm, K is calculated according to eqns.6, 7 and 5[9,10].

$$K = \frac{P(S_1 - S_2)}{BW^{1.5}} \cdot \frac{3F(\alpha)\,\alpha^{0.5}}{2(1-\alpha)^{1.5}} \quad \text{--------------------}(6)$$

$$F(\alpha) = 1.9887 - 1.326\alpha - \frac{(3.49 - 0.68\alpha + 1.35\alpha^2)\alpha(1-\alpha)}{(1+\alpha)^2} \quad \text{---------}(7)$$

K : Fracture toughness ($MPa \cdot m^{0.5}$), K_{IC} or $K_{c,n}$.

P : Fracture load (MN),

B : Thickness of specimen (m),

a : Precrack length or Notch depth (m),

S, S_1,S_2 : Support span (m)

α : Initial precrack relative length (m)

W : Width of specimen (m)

4. RESULTS AND DISCUSSION

4.1 SEPB Method

4.1.1 Precracking

The SEPB measurement requires the introduction of a valid precrack into a specimen before the flexure test. The precracking technique is important, because the precrack length and its propagating direction shall meet the specification in Appendix-1. It is said that the precracking requires some skill and experience[9, 10].

In this round robin, all of the fifteen labs tried the precracking. Table 3 compiles precracking conditions and the rate success rate(N_p/N_{all}), which is defined as N_p(number of precracked specimens met the specification) divided by N_{all}(number of all the precracked specimens).

The instructions recommended one Vickers indent with 98 N as crack starter. However some labs used 3 or 5 indents with higher load of 196 N. A groove width of the bridge anvil was about 5.0 mm in all labs. The pop-in load for precracking showed wide scatter among labs. The success rates from 7 labs were 70 % and higher, but the rates from 6 labs were less than 30 %.

The success rate is considered to relate to a precracking technique including jigs used in each lab. The jig configuration is considered to relate to the pop-in load, number of indents and indentation load. Figure 3 shows the jigs that were used in the labs. Labs 6, 7, 8, 9, and 10 used the jigs manufactured by Maruto Co., Tokyo Japan, or the Maruto-type jigs modified or improved by the each lab (Fig. 3a). The other labs made their jigs according to the figure 4 of the instructions[Appendix-1].

Labs 6, 8 and 10 succeeded to pre-crack specimens without failure. Their success rates were 100%(40/40, 10/10 and 10/10, respectively). They used one Vickers indent with an indentation load of 98 N as crack starter. The groove width was 5 mm and the pop-in load was less than 25 kN. Figure 4a shows an example of a crack front on the fracture surface from Lab 6.

Labs 6 and 10 used a microphone attached to the precracking jig and monitored pop-in sound by oscilloscope or head-phone, although the sound was audible without any sound equipment.

Table 3. Precracking Conditions for SEPB Specimen

Participant	Load (numer) of Indent	Groove Width, mm	Pop-in Load, kN	Success Rate	
Lab 1	98N (3 or 1)	5.0	49-60	22% (2/9)	AE, No dye
Lab 2	98N (3)			10%(1/10)	Dye(green)
Lab 3	98N (1)	5.0	23-28	30%(3/10)	AE, Op, No dye
Lab 4	98N (3)			75% (6/8)	Dye(MR68 penetrant)
Lab 5	98N (1)	6.0	38-43	30%(3/10)	Ear, Dye(felt pen ink+acetone)
Lab 6	98N (1)	5.0	18-20	100%(40/40)	AE, Dye(red ink+acetone)
Lab 7	98N (1)	5.0		80%(8/10)	AE, Dye(oil-based dye)
Lab 8	98N (1)	5.0	25	100%(10/10)	
Lab 9	98N (1)	5.0	15-20	70%(7/10)	Ear, Opt, No dye
Lab 10	98N (1)	5.0	----	100%(10/10)#2	Ear, Dye
Lab 11	196N (3)		<40	50%(5/10)	AE, Dye(magic ink pen+acetone)
Lab 12	98N (3 or 1)	5.0	78-109	90%(9/10)	AE, Dye(Inkmarker + acetone)
Lab 13	98N (1,3,5)	4.93	3-50	0%(0/10)	AE
Lab 14	98N (3)	5.5#1	70-75	0%(0/10)	Ear, Dye(ink+acetone)
Lab 15	98N (3)			40% (2/5)	Dye(felt pen)

AE : acoustic senser used for precracking. Ear: ear phone monitor.
Dye: dye penetration. Opt: optical microscope.
#1 The sample fails during the precracking test in case of using grooves larger than 6mm or narrower than 5mm.
#2 for 16mm span.

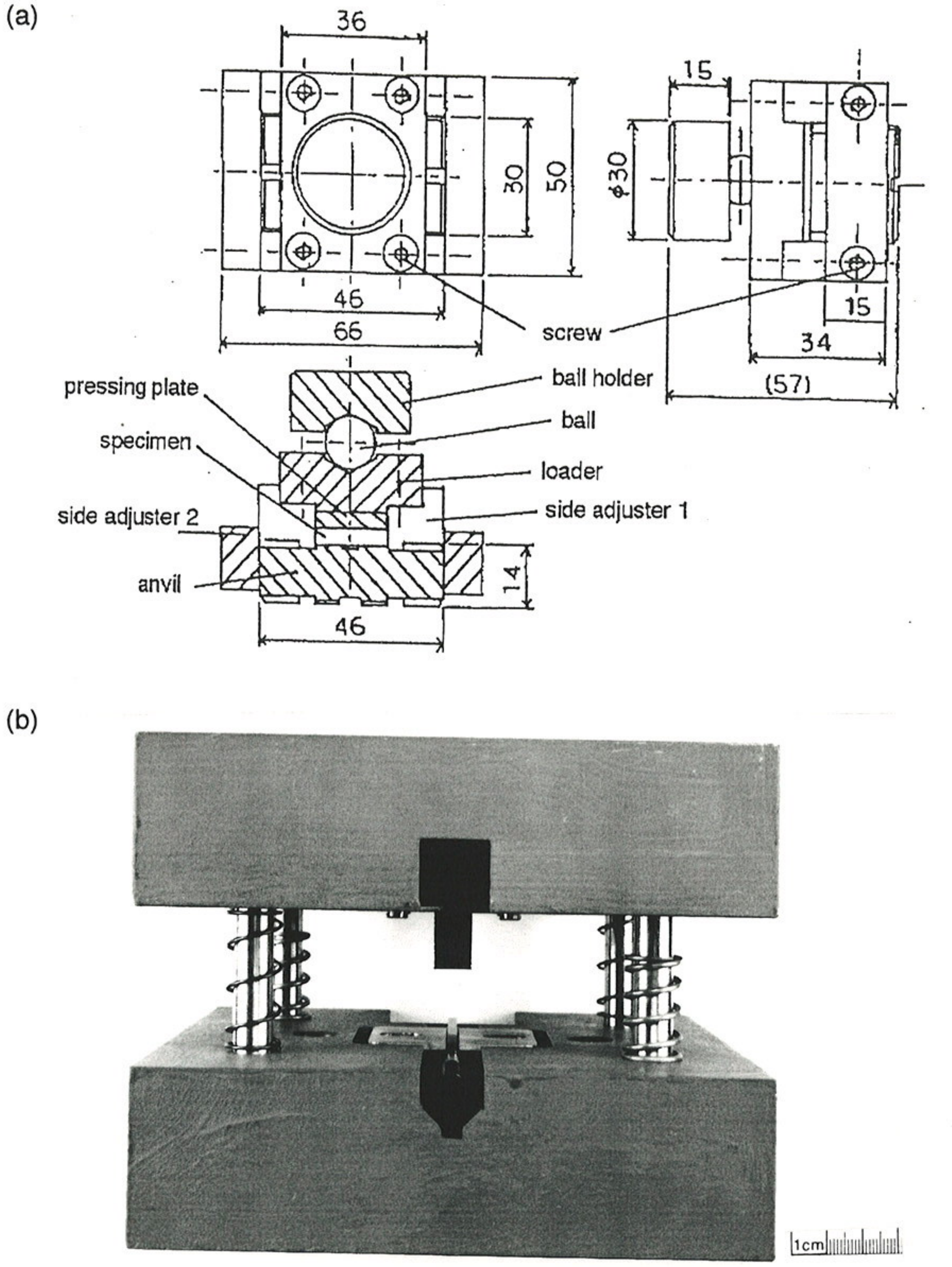
The success rate of Lab 7 was 80%(8/10). Precracking was achieved by using an indentation load of 98 N with one indent for each specimen. The jigs with a groove width of 5 mm were used. Precracking event was monitored by an acoustic emission probe system. To facilitate the demarcation of precrack, an oil-based dye penetrant was placed and air-dried. The obtained precrack length ranged from 1.54 to 2.07 mm. The requirement that each precrack must be normal to the specimen surfaces within 10° was met for all 10 specimens. However, two out of the 10 specimens exhibited more than 10 % in the crack length variation requirement of $(a_{max}-a_{min})/a \leq 0.10$. Therefore the success rate was 80%.

Lab 9 succeeded to precrack specimens except three specimens, whose precracks were out of the requirement. The success rate was 70%(7/10).

The success rate of Lab 12 was 90%(9/10). One crack length was out of requirement ($(a_{max}-a_{min})/a=0.26 \geq 0.1$). Their jigs are shown in Fig. 3b. The pop-in was detected by using an acoustic emission sensor (PAC; Pico 300 - 1000 kHz; resonance at 500 kHz), which was connected to a standard scope and a memory recorder with printer. In the moment of the scope's trigger peak, the machine was stopped and immediately unloaded. The pop-in loads were 78 to 109 kN, which were much higher than that from the other labs.

Lab 4 succeeded in precracking. The success rate was 75%(6/8).

Lab 11 made two types of the jig. One was steel jigs with various groove widths. Another was steel jigs with a sialon plate (Fig. 3c) and the bridge was formed by a gap between two adjustable tungsten carbide(WC) cylinders loaded onto the surface. The top part was guided relative to the bottom part by a device. This lab precracked specimens with various conditions and then their success rate was 50%(5/10). The crack front lines, which were valid or invalid for the following fracture toughness measurement, were shown in Fig. 4b. The pop-in load was from 28 to 35 kN in the steel jig, and approximately 46 kN in the jig with sialon plate and WC cylinders. Red marker pen with organic ink was used to mark all precracks before testing. The precracked face with oblique illumination was photographed at calibrated magnification. The precrack length was measured from the photos.



Comment: The upper plate with the pusher is guided via four linear bearings. Four springs hold the pusher normally close over the specimen.

Fig. 3. Precracking Jigs Used in the Round Robin.

(a) Labs 6, 7, 8, 9, and 10.

(b) Lab 12.

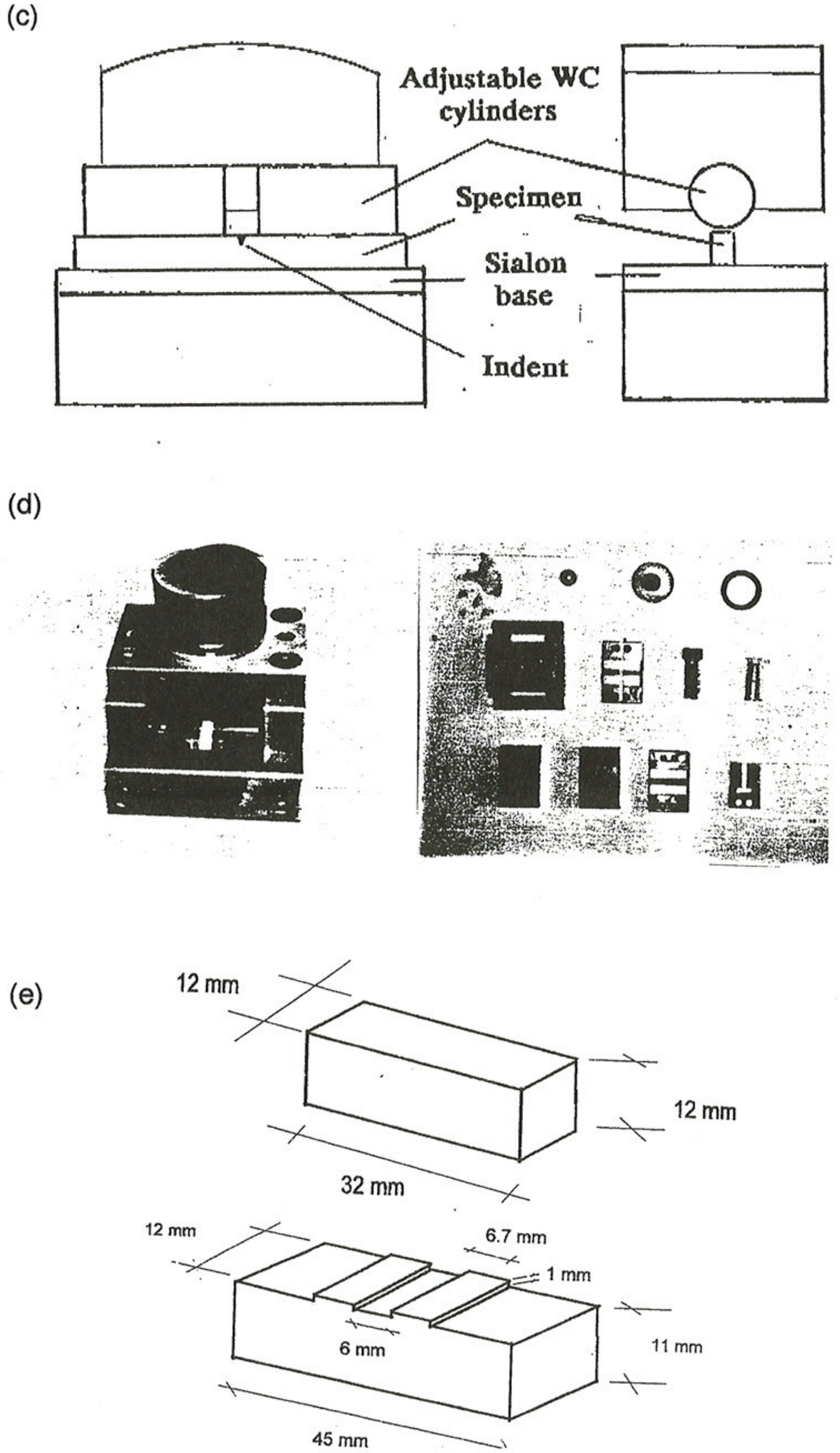


Fig. 3. Precracking Jigs Used in the Round Robin.
(c) Lab 11, (d) Lab 3, (e) Lab 5.

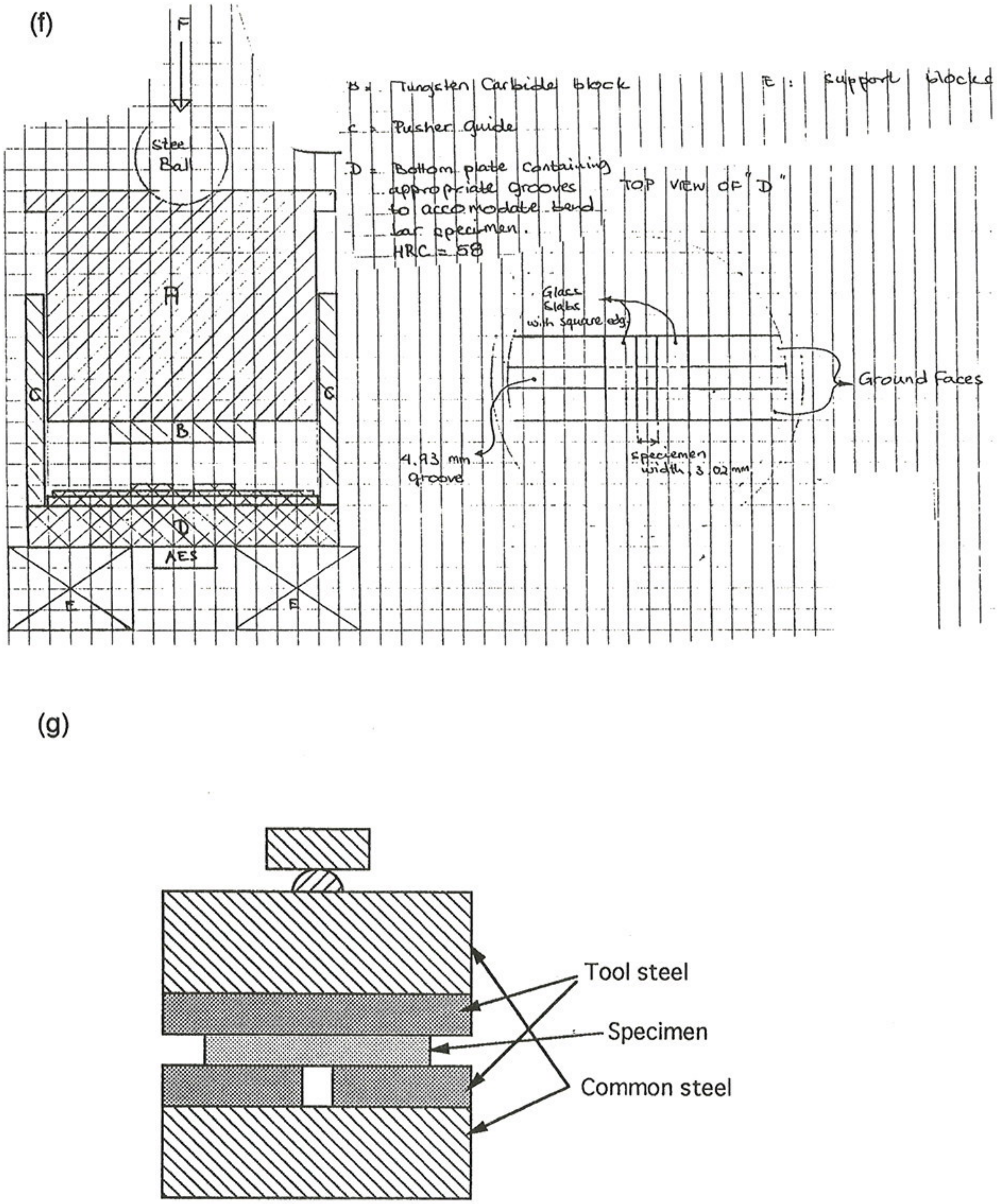


Fig. 3. Precracking Jigs Used in the Round Robin.
(f) Lab 13, (g) Lab 14.

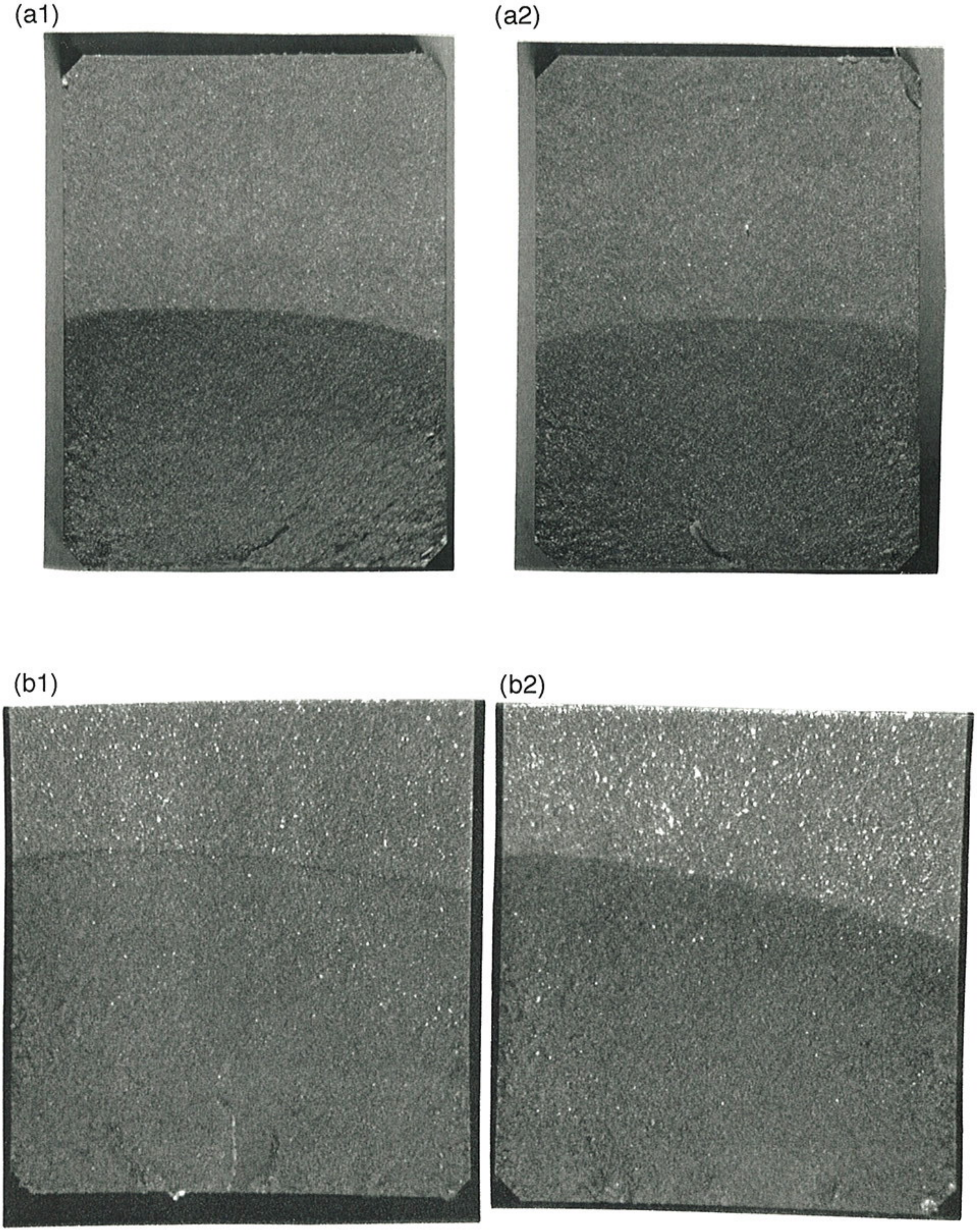


Fig. 4a,b. Fracture Surface Showing Precrack Front Line in the SEPB Method.

(a1) Lab 6 ; 7.0 MPa·m$^{0.5}$ (Specimen No.24-5, 3p-16 mm span)
(a2) Lab 6 ; 6.5 MPa·m$^{0.5}$ (Specimen No.8-4, 3p-30 mm span)
(b1) Lab 11 ; 5.9 MPa·m$^{0.5}$ (valid precrack)
(b2) Lab 11 ; 5.9 MPa·m$^{0.5}$ (invalid precrack)

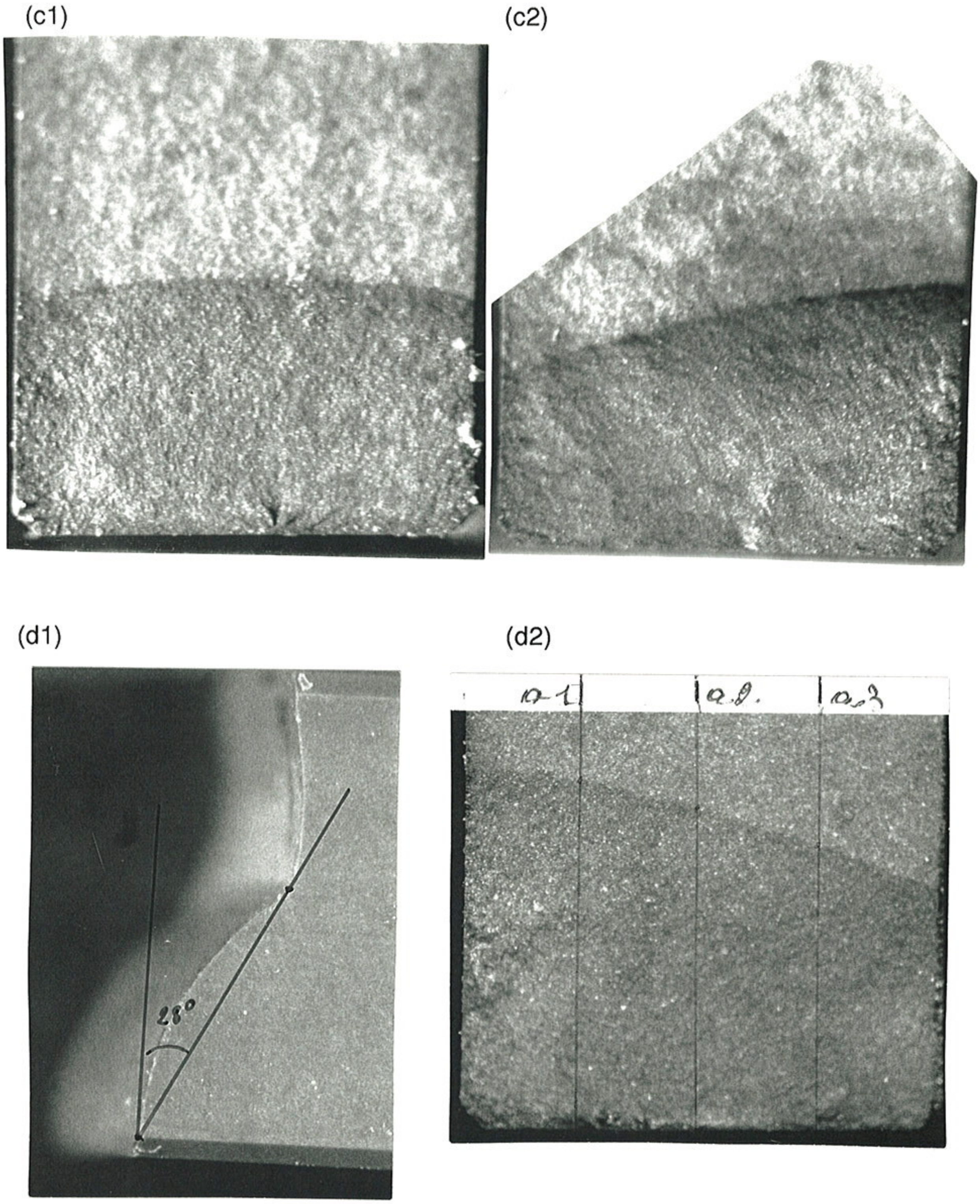


Fig. 4c,d. Fracture Surface Showing Precrack Front Line in the SEPB Method.
(c1) Lab 5 ; 6.9 MPa·m$^{0.5}$ (Specimen No.1-10, valid precrack)
(c2) Lab 5 ; 7.3 MPa·m$^{0.5}$ (Specimen No.3-3, invalid precrack)
(d1) Lab 2 ; (Specimen No.28-12, invalid precrack)
(d2) Lab 2 ; (Specimen No.21-8, invalid precrack)

Lab 3 used house-made jigs in accordance to the instructions with a slightly different anvil (Fig. 3d). Instead of grooves to align the specimens, two bolts were used on each anvil end. They reported that they had no real problems for precracking. However, the success rate was 30%(3/10).

Lab 5 used house-made jigs (Fig.3e) and the success rate was 3/10. The pop-in crack was audible by the operator without the use of any particular device. Figure 4c shows the examples of valid and invalid crack front lines on the fracture surface.

Lab 1 used house-made jigs and the success rate was 22%(2/9). They monitored the introduction of the precrack with a sonic sensor connected to an oscilloscope. When a great peak was indicated on the oscilloscope, the loading machine was stopped and unloaded. The maximum value was recorded from the peak value. Simultaneously they made an analog-recording of the load. They tried to penetrate the precrack with white oil paint mixed with acetone, but the dye penetration was not successful.

The success rate of Lab 2 was only 10%(1/10). Their cracks as shown in Fig.4d apparently are out of the specification of precracking, suggesting that their precracking technique was improper. Lab 2 thinks that the precracking technique can not be successfully used at a laboratory scale and is not appropriate as a routine test in production lines. They think that the SEPB method is not suitable for further standardization procedure.

Lab 13 did not succeed in precracking and the success rate was 0/10. The jig they used is shown in Fig. 3f. They had had success in the past with the SEPB measurement for Al_2O_3 and $SiC_{(whisker)}$-Al_2O_3 materials. However they could not work out the proper condition for $SiC_{(whisker)}$-Si_3N_4. Furthermore their acoustic sensor was too sensitive to make it difficult to separate fracture from other sources.

Lab 14 also did not succeed in precracking and the success rate was 0%(0/10). The precrack length was more than 2.4 mm, resulting that their data were invalid. A precrack length is usually controlled by a groove width. They tried to precrack specimens with different number of indents and groove width, but they could not find the proper conditions. The jig they used is shown in Fig. 3g. Their pop-in load ranged from 70 to 75 kN, which was much more than that from the other labs except Lab 12. It therefore appears that the Lab 12 precracking jig was not suitable.

As for the elasticity of the precracking jig material, Lab 11 gave a comment that precracking becomes more difficult as the materials of the bridge jig get stiffer[18]. Lab 5 also gave a similar comment that a low Young's modulus jig would be better when precracking a specimen with a very high Young's modulus, and vice versa. There is yet room for improvement in the jig material as well as the jig configuration.

It is true that the precracking was not easy to perform for some labs in this round robin, however six labs including four Japanese labs has succeeded in precracking and measurement of fracture toughness. It probably depends on the skillfulness and experience of this technique among operators, because the writers feel that the precracking technique is not difficult for operators with some experience. The SEPB method was selected and registered in 1990 as Japanese standard JIS R1607 after 3-year R&D on fracture toughness measurement of advanced ceramics. The SEPB has been popular now in Japan. The accurate jigs for precracking are commercially available in Japan. The technique for precracking would require some experience and accurate jigs.

4.1.2 Measurement of K_{IC}

The precracked specimens were fractured using several kinds of testing machines as shown in Table 4. The crosshead speeds were mostly 0.5 mm/min and the times to failure were in the range from 3 to 13 seconds. The large scatter in time to fracture is probably due to the difference in the fracture load of specimens and the elasticity of the measurement machine and jigs.

4.1.3 K_{IC} Values by SEPB Method

The results from 15 labs are compiled in Table 5. Most of the labs in European countries and the USA adopted 4-p flexure with spans of 40 and 20 mm. The labs in Japan and one lab in Europe adopted 3-p flexure.

The Lab 6 conducted the SEPB measurement with four different flexure modes, which were 3-p with a span of 16, 30 or 40 mm and 4-p with 20 and 40 mm spans. The success rates for precracking were 100%. The results give good data

Table 4. Machines and Test Conditions for SEPB Method.

SEPB Participant	Crosshead Speed, mm/min	Time to Failure, sec	Machine for Flexure Test
Lab 1	0.48	6 to 7	INSTRON 8562
Lab 2	0.5	4.0	ZWICK 1474
Lab 3	0.5	4.1	ZWICK
Lab 4	0.5	6.4	INSTRON 1380
Lab 5	0.5	13.0	INSTRON 1195
Lab 6	0.5	-	SHIMADZU Autograph AG-10TB
Lab 7	0.5	10.0	INSTRON M-0
Lab 8	0.5	10.0	INSTRON 4204
Lab 9	0.5	3 to 5	INSTRON 1123
Lab 10	0.5	-	INSTRON 1122
Lab 11	0.5	5.0	INSTRON 4505
Lab 12	0.5	-	SCHENK S56
Lab 14	0.5	8.0	INSTRON 4502
Lab 15	0.5	-	INSTRON 4201

Table 5. Fracture Toughness (K_{IC}) Values by SEPB Method.

SEPB Participant	Flexure Type -Span(s). mm	K_{IC} ,MPa$\sqrt{m}$		Number of Valid Data
		Average	Standard Deviation	
Lab 1	4P-40/20	6.3	-	2
Lab 2	3P-30	5.6	-	1
Lab 3	4P-40/20	7.6	0.1	3
Lab 4	4P-40/20	6.7	0.4	6
Lab 5	4P-40/20	7.4	0.4	3
Lab 6	3P-16	7.2	0.2	10
	3P-30	6.7	0.1	10
	3P-40	6.9	0.2	10
	4P-40/20	6.7	0.1	10
Lab 7	4P-40/20	6.6	0.2	8
Lab 8	3P-30	6.7	0.2	10
Lab9	3P-30	7.2	0.4	7
Lab 10	3P-16	7.0	0.3	10
Lab 11	4P-40/20	6.1	0.2	5
Lab 12	4P-40/20	7.1	0.4	9
Lab 13	4P-40/20	-	-	0
Lab 14	4P-40/20	-	-	0
Lab 15	4P-40/20	6.3	-	2

to discuss the flexure mode dependence of K_{IC} in the SEPB method. The average values are in the range from 6.7 to 7.2 MPa·$m^{0.5}$ with a standard deviation not more than 0.2 (Table 5 and Fig.5). Considering measurement error belonging to the measured K_{IC} value, this suggests that there is no meaningful flexure mode dependence on the K_{IC} values.

However the K_{IC} values from the mode of 3-p with 16 mm may be a little higher than the others. This is interpreted by the shortness of the 16 mm span. A precrack direction from an indent as precrack starter is not always exactly perpendicular to the tensile surface of the specimen. The specification allows up to 10° difference from the perpendicular direction. Therefore a front line of the precrack generally does not adjust to the central loading axis for the specimen. On the other hand, in case of 3-p flexure mode, tensile stress on the tensile side of a flexure specimen is maximum at a central loading axis. With increasing the distance between the precrack front and loading axis, the calculated fracture load becomes larger than the actual load on the crack plane, leading to a higher K_{IC} value. The shorter span would give the larger value.

Figure 6 shows the flexure mode dependence of average values from all the participants. The mode of 4-p flexure with 20 and 40 mm spans shows wide scatter in the range from 6.1 to 7.6 MPa·$m^{0.5}$. This scatter is due to the difference of K_{IC} values from large number of 11 labs. However, the average values from five labs, which measured 5 and more of valid data, were from 6.1 to 7.1 MPa·$m^{0.5}$. The SEPB measurement is considered to require some technique and precise jigs for precracking to obtain the reliable values. Although the scatter may be also due to slow crack growth or R curve effects in the material, or problems in measuring crack length, it is not clear how large they effected to the K_{IC} values.

In the result, there is no mode dependence in the modes with spans of 30 or 40 mm. The long span such as 30 or 40 mm is recommended to make measurement error small in the SEPB method.

4.2 SENB and SEVNB Methods

4.2.1. Saw-cut and V-notch

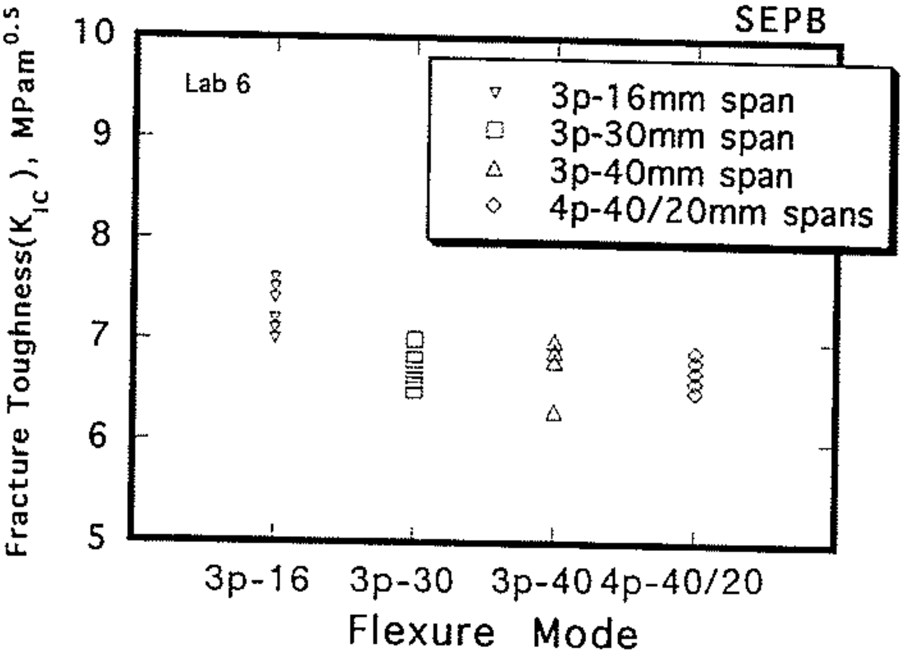


Fig.5. Flexure Mode Dependence of Fracture Toughness (Lab 6 ; SEPB Method).

(Each point crresponds to one specime.)

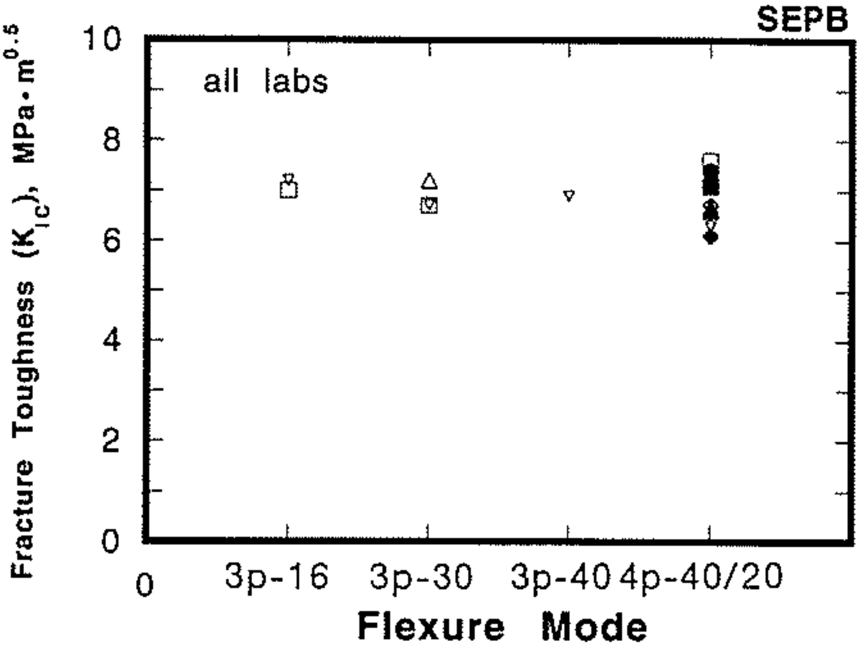


Fig.6. Flexure Mode Dependence of Fracture Toughness (all labs ; SEPB Method).

(Each point represents the average result from a laboratory.)

As shown in Table 6, twelve labs used different machines with their conditions to introduce a saw-cut or V-notch into a specimen. When a specimen with a saw-cut or V-notch is flexured, the critical crack propagation initiates from a point around the tip of the saw-cut or V-notch. Therefore the profile and machining damage around the tip are important factors which effect the measured K_{IC} values.

The micrographs of a saw-cut (Fig.7a-g) show the profiles with a constant or inconstant width and semi-round or square-like tips. The saw-cut introduced even by the same lab shows different profiles in shape (Fig.7a, c, e and g ; Labs 2, 5, 9 and 14).

As for the SEVNB, five labs introduced V-notch into a specimen by themselves. Labs 6 and 7 used specially-prepared diamond wheel for V-notch machining. The V-notch profiles from Lab 6 show symmetrically round shape (Fig.8b), but those from Labs 3 and 7 are not symmetrical (Fig.8a, c). Lab 3 introduced a V-notch by hand machining using razor blade and diamond paste. The tip profile are not symmetrically round.

These observation suggests that machining conditions for a saw-cut and V-notch are not constant among labs except Lab 6 and may cause inconsistent machining damage to the specimens.

Ceramics are generally sensitive to the defects which would work as a crack starter when fractured. Therefore the precise machining and the specification may be required in order to make the SENB and SEVNB methods popular.

4.2.2 Measurement of $K_{c,n}$

The saw-cut or V-notched specimens were flexured using several kinds of testing machines as shown in Table 6. As well as the SEPB method, the crosshead speeds were mostly 0.5 mm/min and times to failure were in the range from 3 to 13 seconds. The large scatter in time to fracture is probably due to the difference in the fracture load of specimens and the elasticity of the measurement machine and jigs.

4.2.3 $K_{c,n}$ Values by SENB and SEVNB Methods

The $K_{c,n}$ values from 13 labs are compiled in Table 7. Each lab adopted the same flexure mode as for their SEPB measurement. The half width of a saw-cut

Table 6. Machines and Conditions for SENB and SEVNB Methods.

SENB SEVNB Participant	Test Method (NW, μm)	Machine for Introducing Saw-cut or Notch	Machine for Flexure Test	Flexure Test	
				Crosshead Speed mm/min	Time to Failure, sec
Lab 1	SENB	SAFAG 3031S; WS(22m/sec), DF(2.8mm/min)	INSTRON 8562	0.048	4.75
Lab 2	SENB(56)	CG400(FR); WS(5m/sec), Wh(metal bond)	ZWICK 1474	0.5	4.5
	SENB(73)			0.5	6.0
Lab 3	SENB(99-100)	Horizontal Boring Machine; BD(120mm), Rpm(6000)	ZWICK	0.5	6.2
	SENB(16-44)	Horizontal Boring Machine; BD(75mm), Rpm(6000)	ZWICK	0.5	6.3
	SEVNB	Hand; Razor blade & Diamond paste	ZWICK	0.5	6.3
Lab 5	SENB	BERNEY T34; WS(32, or 17m/sec), DF(50 μm/pass)	INSTRON 1195	0.5	12 to 13
Lab 6	SEVNB	TOSHIBA MACHINE USM-200; Wh(SD1000N100M), WS(41m/sec), DF(2μm/pass), TS(150mm/min)	SHIMADZU, Autograph AG-10TB	0.5	-
Lab 7	SENB	BOMAS; Wh(ϕ76.2mm), Rpm(6000),DF(0.00635 mm/pass)	INSTRON M-0	0.5	11.0
	SEVNB	BOMAS; V-shaped diamond wheel	INSTRON M-0	0.5	10.0
Lab 8	SENB, SEVNB	COMMEC with high speed spindle; Wh(#400,ϕ60mm), Rpm(12000),	INSTRON 4204	0.5	10.0
				0.5	10.0
Kab 9	SENB		INSTRON 1123	0.5	3 to 5
Lab 10	SEVNB(15-18)	TOKYO SEIKI TSK2510S; Wh(SD1000N100M6)	INSTRON 1122	0.5	-
	SEVNB(44-47)	TOKYO SEIKI TSK2510S; Wh(SD400R13B01)		0.5	-
Lab 12	SENB	STRUERS/ACCUTOM; 3000 U/min	SCHENCK S56	0.5	-
Lab 13	SENB	ACCUTOM-2/STREURS; TS(0.5-0.7mm/min), Rpm(2400-3100)	INSTRON 1102	0.5	8.1
Lab 14	SENB(30. 43)	BERNEY T34; WS(17m/sec), Wh(ϕ100mm), DF(50μm/pass)	INSTRON 4502	0.5	9.0
	SENB(50, 65)	BERNEY T34; WS(32m/sec), Wh(ϕ100mm), DF(50μm/pass)		0.5	9.0
Lab 15	SENB		INSTRON 4201	0.5	-

NW; Half of notch width(SENB) or Notch tip radius(SEVNB), WS; Wheel speed, DF; Down feed, Wh; Wheel, BD; Blade diameter, Rpm; Revolutions per min., TS; Table speed.

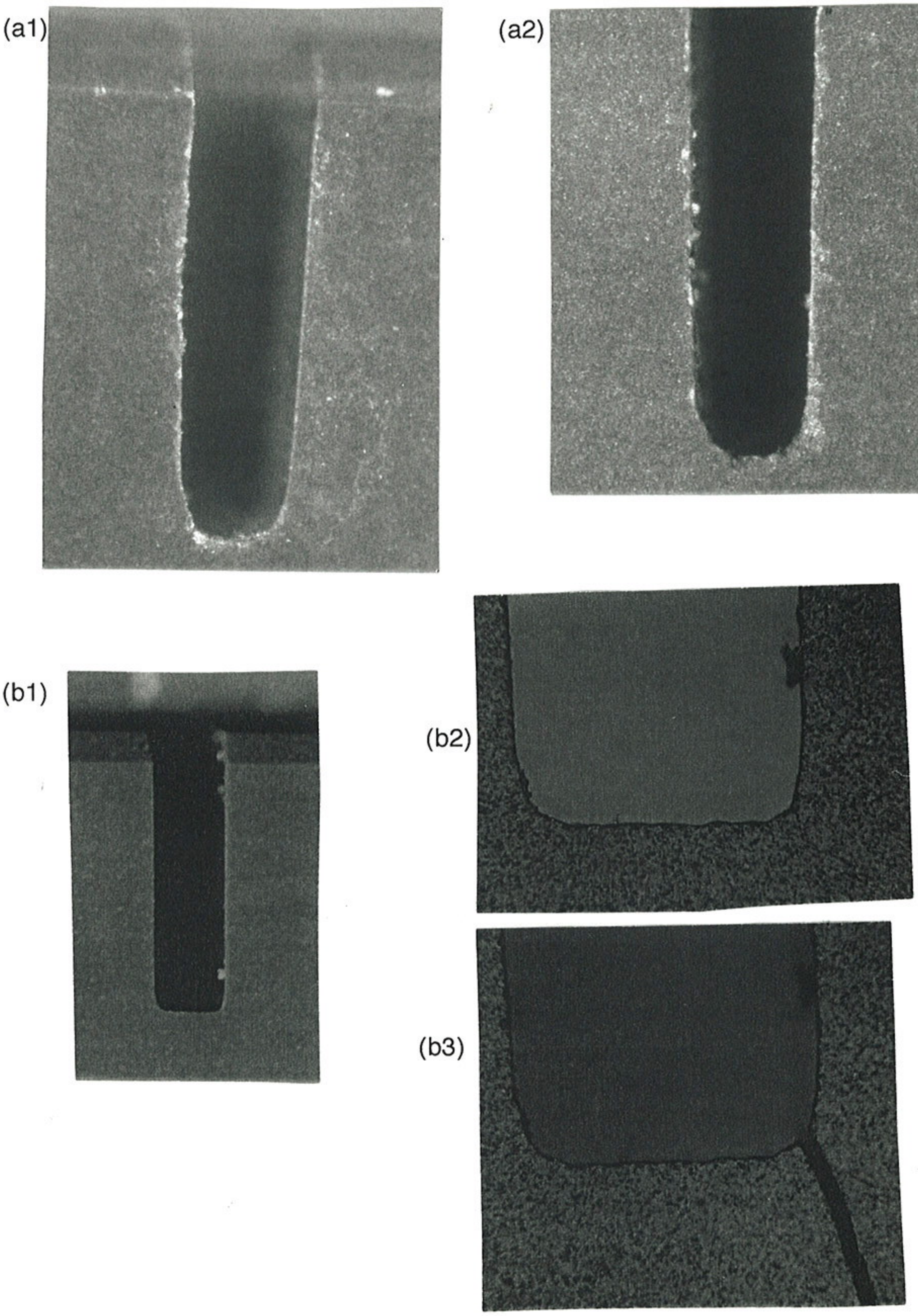


Fig. 7a,b. Profiles of Saw-Cut from Participants.

(a1) Lab 2; 290 μm in width, Specimen No.17-30, 8.5 MPa·$m^{0.5}$,

(a2) Lab 2; 290 μm in width, Specimen No.24-70, 7.8 MPa·$m^{0.5}$,

(b1-3) Lab 3; 394 μm in width, Specimen No.32-2, 9.6 MPa·$m^{0.5}$.

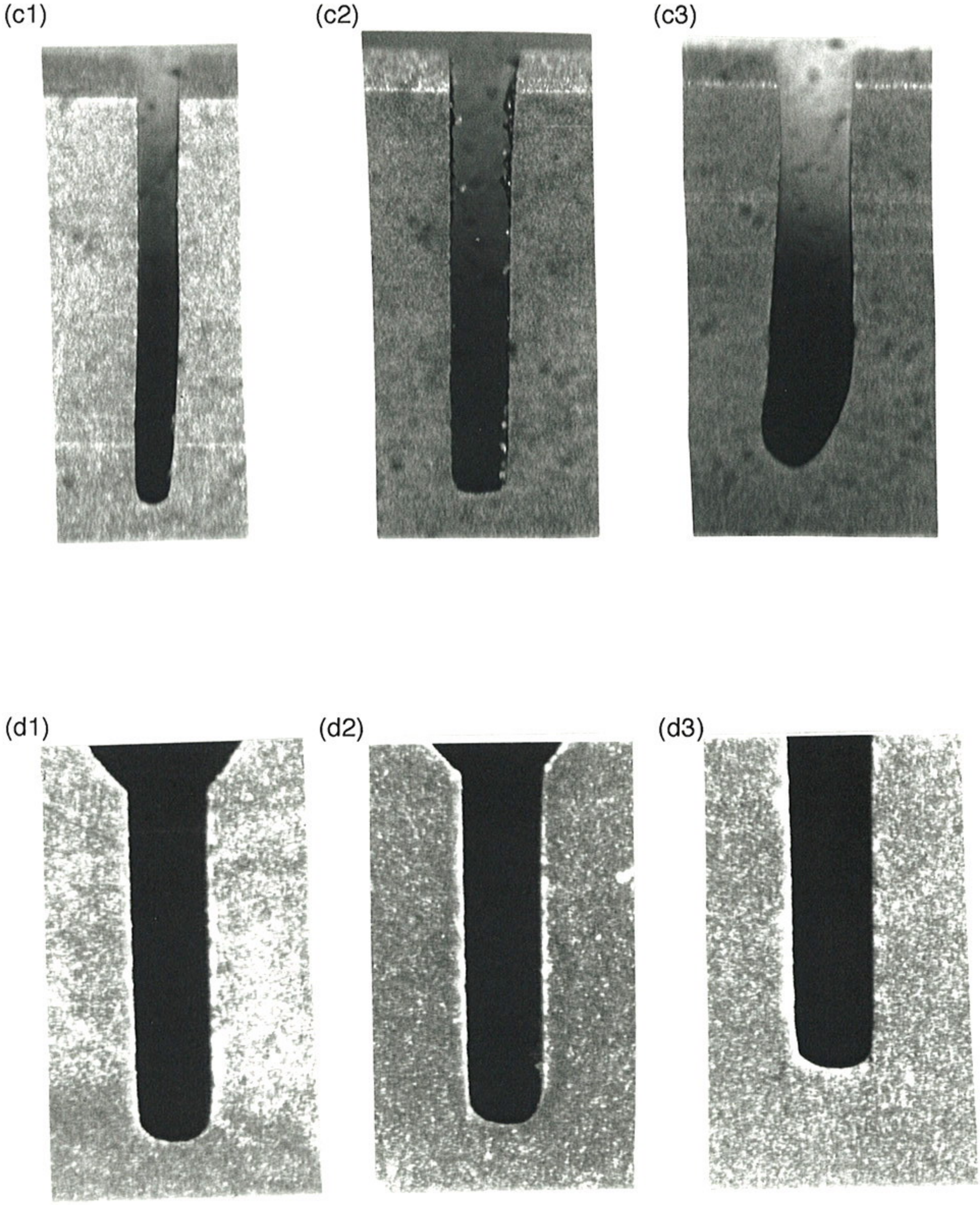


Fig. 7c,d. Profiles of Saw-Cut from Participants.

(c) Lab 2; (c1)113 μm(w), No.32-7, 8.2 $\mathrm{MPa \cdot m^{0.5}}$, (c2) 180 μm(w), No.30-13, 9.2 $\mathrm{MPa \cdot m^{0.5}}$, (c3) 337 μm(w), No.16-5, 9.4 $\mathrm{MPa \cdot m^{0.5}}$

(d) Lab 7; (d1)175 μm(w), No.9-8, 8.0 $\mathrm{MPa \cdot m^{0.5}}$, (d2) 175 μm(w), No.25-9, 8.8 $\mathrm{MPa \cdot m^{0.5}}$, (d3) 180 μm(w), No.28-8, 8.6 $\mathrm{MPa \cdot m^{0.5}}$

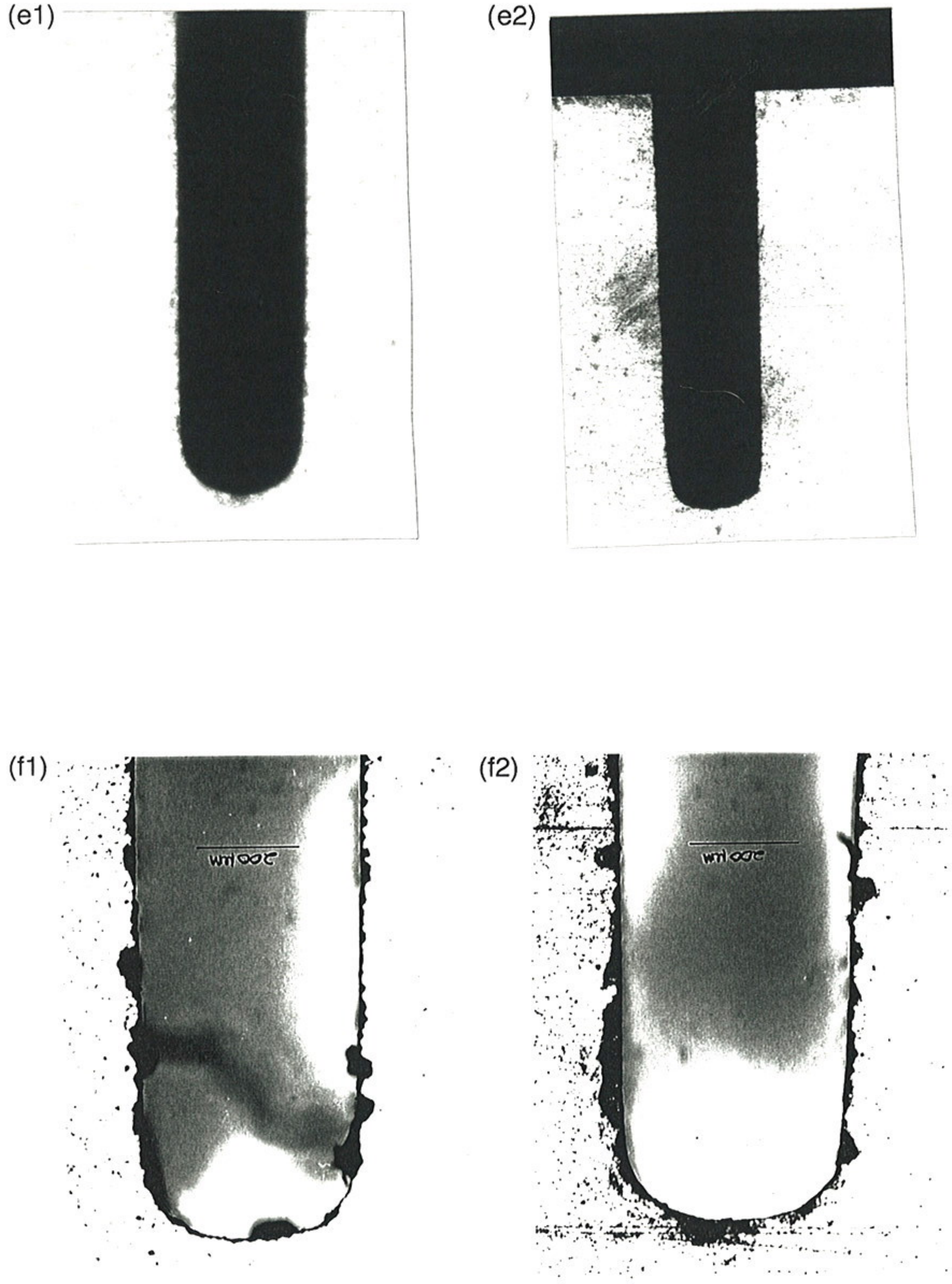
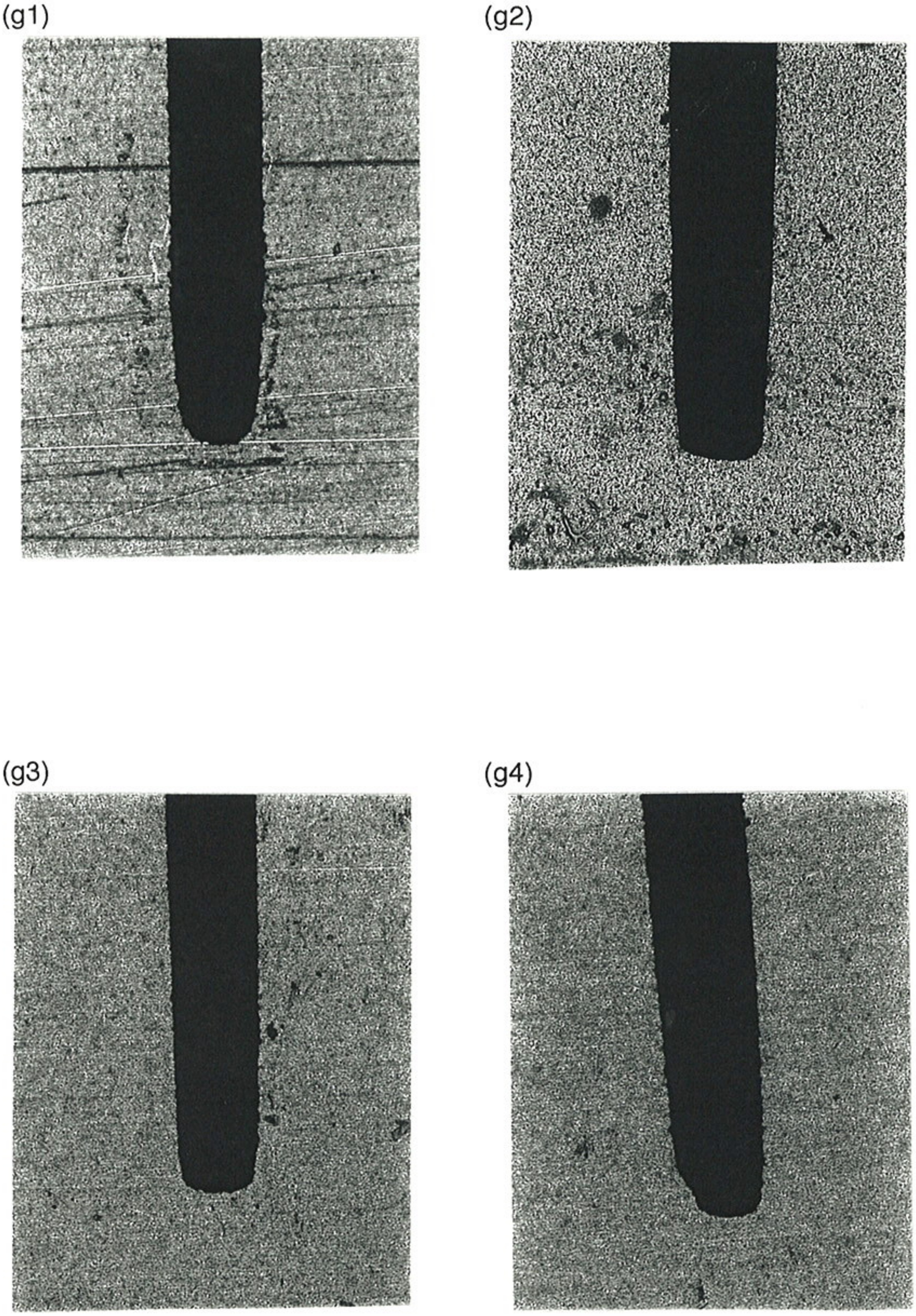


Fig. 7e,f. Profiles of Saw-Cut from Participants.

(e) Lab 9; (e1)99 μm(w), No.4-8, 8.1 MPa·$m^{0.5}$, (e2) 313 μm(w), No.25-6, 10.9 MPa·$m^{0.5}$

(f) Lab 13; (f1)285 μm(w), STN02, 8.2 MPa·$m^{0.5}$, (f2) 250 μm(w), STN04, 8.3 MPa·$m^{0.5}$



(g1) (g2) (g3) (g4)

Fig. 7g. Profiles of Saw-Cut from Participants.

(g) Lab 14; (g1)244 μm(w), No.5-1, 9.6 MPa·m$^{0.5}$, (g2) 226 μm(w), No.6-1, 9.3 MPa·m$^{0.5}$
(g3)204 μm(w), No.7-1, 9.6 MPa·m$^{0.5}$, (g4) 250 μm(w), No.8-1, 9.3 MPa·m$^{0.5}$

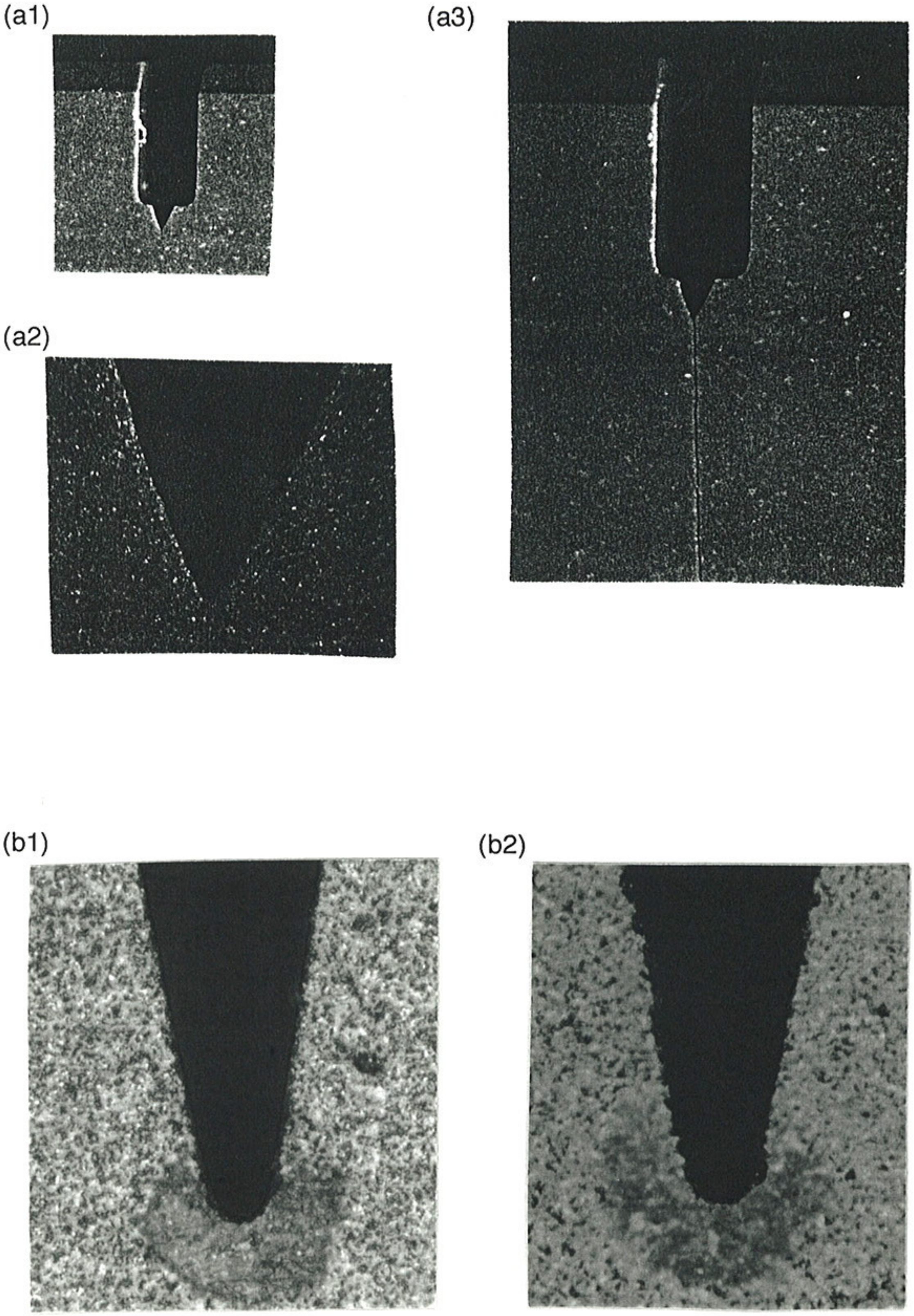


Fig. 8a,b. Profiles of V-notch from Participants.

(a1-3) Lab 3; 14 μm(tip radius), No.45-7, 6.3 MPa$\cdot$m$^{0.5}$,

(b) Lab 6; (b1)9 μm(tip radius), No.3-2, 6.3 MPa$\cdot$m$^{0.5}$, 3p-40 mm span,
(b2)9 μm(tip radius), No.44-9, 6.1 MPa$\cdot$m$^{0.5}$, 4p-40 & 20 mm spans

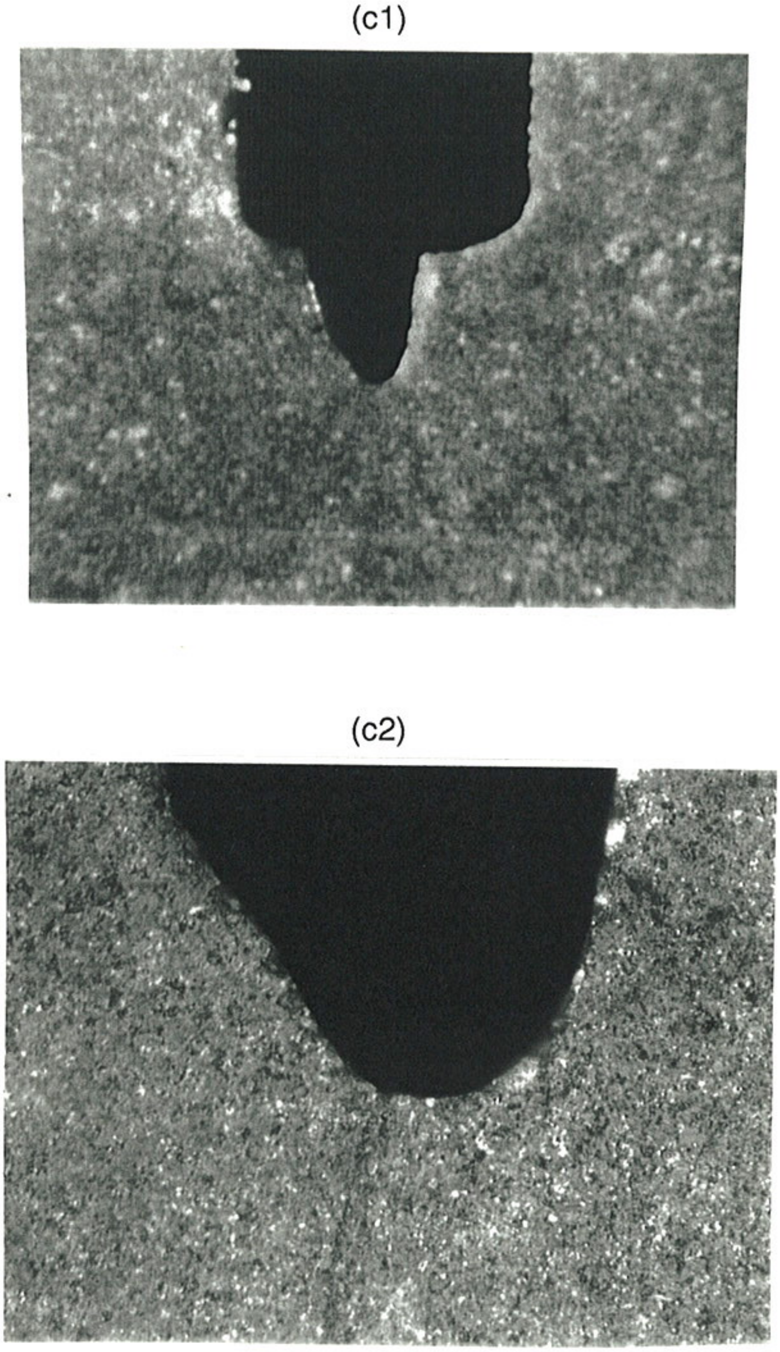


Fig. 8c. Profiles of V-notch from Participants.
Lab 7; (c1,2) 35 μm (tip radius), No.44-3, 7.7 MPa·m$^{0.5}$

Table 7. Kc,n Values by SENB and SEVNB Methods.

SENB SEVNB Participant	Test Method	Flexure Type -Span(s), mm	$K_{c,n}$,MPa√m		V-notch Radius ,μm	Half Width of Saw-cut ,μm	Number of Data
			Average	Standard Deviation			
Lab 1	SENB	4P-40/20	7.9	0.4	-	33-48	9
Lab 2	SENB	3P-30	7.5	0.3	-	105	6
	SENB	3P-30	8.8	0.9	-	145	6
Lab 3	SENB	4P-40/20	9.5	0.4	-	197-200	10
	SENB	4P-40/20	9.1	0.6	-	85-88	8
	SENB	4P-40/20	8.0	-	-	32, 33	2
	SEVNB	4P-40/20	6.3	0.1	12-14	-	10
Lab 5	SENB	4P-40/20	7.8	0.4	-	38-57	6
	SENB	4P-40/20	8.8	0.4	-	65-90	13
	SENB	4P-40/20	9.4	-	-	169	1
Lab 6	SEVNB	3P-16	6.5	0.2	9	-	10
	SEVNB	3P-30	6.4	0.1	9	-	10
	SEVNB	3P-40	6.3	0.1	9	-	10
	SEVNB	4P-40/20	6.2	0.1	9	-	10
Lab 7	SENB	4P-40/20	8.7	0.5	-	88-92	10
	SEVNB	4P-40/20	8.2	0.3	33-38	-	10
Lab 8	SENB	3P-30	10.6	0.4	-	87-89	10
	SEVNB	3P-30	7.4	0.2	30-34	-	10
Lab 9	SENB	3P-30	8.4	0.5	-	48-51	10
	SENB	3P-30	11.8	0.8	-	141-157	10
Lab 10	SEVNB	3P-30	6.3	0.1	15-18	-	10
	SEVNB	3P-30	7.8	0.3	44-47	-	10
Lab 12	SENB	4P-40/20	10.1	1.0	-	175, 200	8
	SENB	4P-40/20	9.1	0.7	-	100, 125	10
	SENB	4P-40/20	8.0	-	-	25	1
Lab 13	SENB	4P-40/20	8.4	0.4	-	110-143	7
	SENB	4P-40/20	11.5	1.2	-	203-221	11
Lab 14	SENB	4P-40/20	8.3	0.5	-	59-85	10
	SENB	4P-40/20	9.7	0.7	-	100-129	10
Lab 15	SENB	4P-40/20	9.3	0.5	-	95	5
	SENB	4P-40/20	9.0	0.3	-	215, 220	4

ranges broadly from 33 to 221 μm, and the V-notch radius ranges from 9 to 47 μm. The average $K_{c,n}$ values are calculated using the values from the similar saw-cut widths.

As well as the SEPB method, Lab 6 conducted the SEVNB measurement with 4 different flexure modes. The measured $K_{c,n}$ values with a 3-p with a span of 16, 30 and 40 mm and a 4-p with spans of 20 and 40 mm are the same each other. They are in the range from 6.2 to 6.5 $MPa\cdot m^{0.5}$ with a standard deviation not more than 0.2 $MPa\cdot m^{0.5}$ (Table 7 and Fig.9). This indicates that there is no flexure mode dependence of the $K_{c,n}$ by the SEVNB method.

Although a long span such as 30 or 40 mm is recommended in the SEPB method, the mode of a 3-p with 16 mm in the SEVNB method is also recommended as well as the modes with a span of 30 or 40 mm. This is probably due that adjusting loading axis to the V-notch tip is not difficult

Figures 10 and 11 show the dependence of Kc,n on half width of saw-cut and V-notch tip radius, respectively. The combined data are shown in Fig.12. The $K_{c,n}$ values by the SENB and SEVNB methods increase with increasing a half width of saw-cut or the tip radius. This tendency is observed in other reports[9,10,16].

The $K_{c,n}$ values by the SENB method show wide scatter ranging from 6 to 13 $MPa\cdot m^{0.5}$. This is considered to be due to the difference in the saw-cut shape and machining conditions among labs. If the half width is specified, the obtained $K_{c,n}$ shows wide scatter. For example, a half width of about 100 μm gives the $K_{c,n}$ values ranging from 7 to 11 $MPa\cdot m^{0.5}$. Therefore the SENB method would not become a standard method without the specification of precise machining conditions for introducing a saw-cut into specimen.

The $K_{c,n}$ values by the SEVNB method show large scatter in case of the tip radius more than 30 μm. However, the values show small scatter in case of the radius less than 20 μm. They are in the range from 6 to 7 $MPa\cdot m^{0.5}$ and similar to the values by the SEPB method (Table 5). According to the data from Lab 6[19], SiC(w)/Si_3N_4 used in this round robin shows almost constant $K_{c,n}$ values in case of the tip radius from 10 to 30 μm, and they were similar to the values by the SEPB. In the previous round robin[10] using silicon nitride, the $K_{c,n}$ values by the SEVNB method were similar to those by the SEPB method in case of the tip radius from 10 to 35 μm. Therefore the notch tip radius less than 30 μm is recommended in the SEVNB method. Under the condition, the $K_{c,n}$ values are expected to be similar to the K_{IC}.

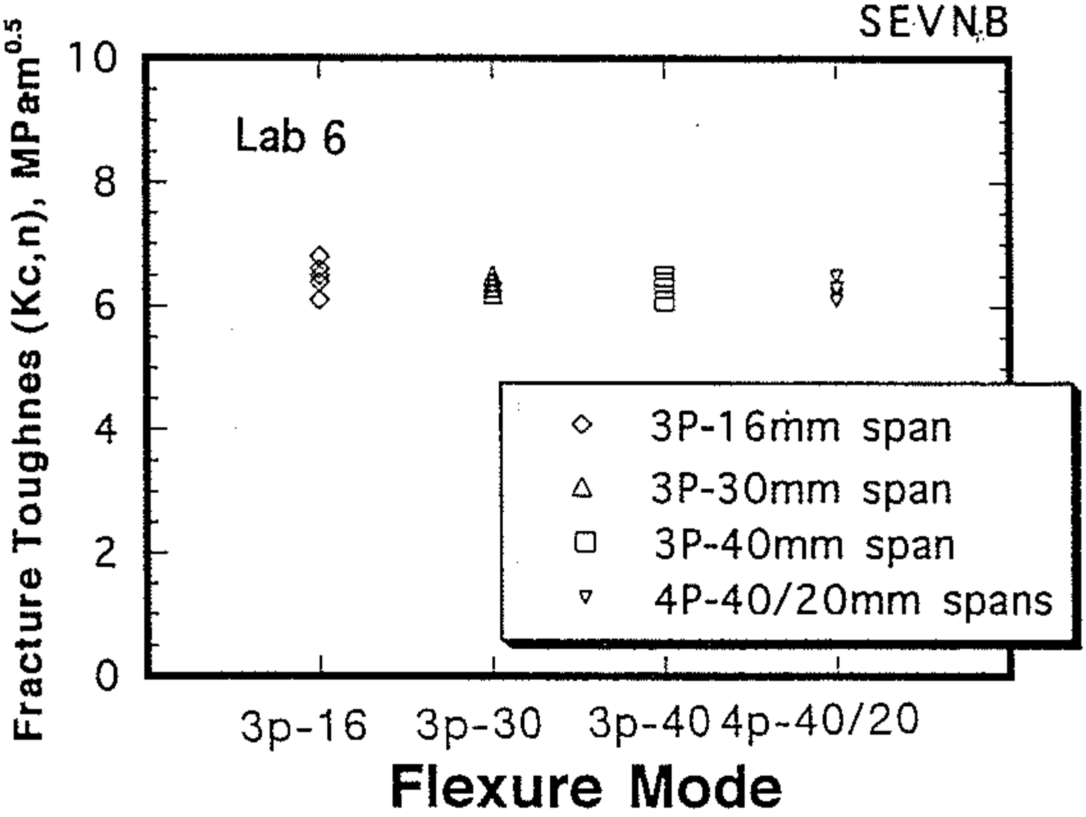


Fig. 9. Flexure Mode dependence of $K_{c,n}$ by SEVNB Method. (Lab 6)

4.3 Comparison among Three Methods

In this round robin, the SEPB, SENB and SEVNB methods are used to measure the fracture toughness of $SiC(w)/Si_3N_4$. Among the three methods, $K_{c,n}$ values by the SENB are much higher and show larger scatter than those by the SEPB and SEVNB. At present, the SENB is not recommended as candidate method for fracture toughness measurement, because the inconstant saw-cut configuration and machining conditions cause large scatter of the data.

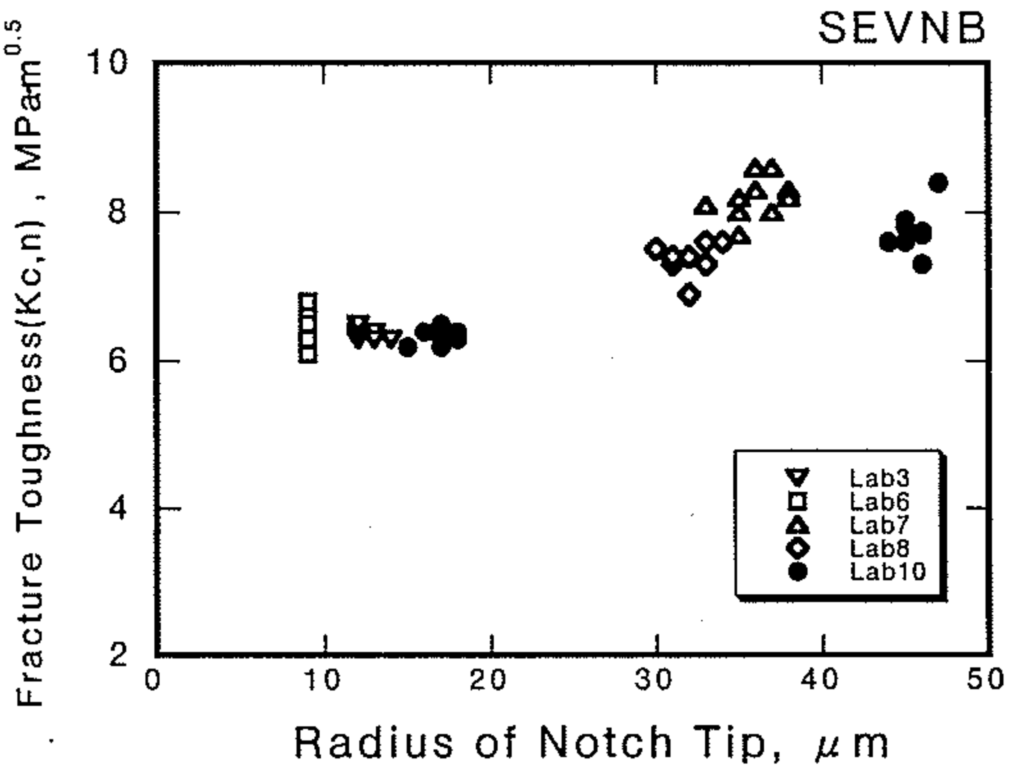


Fig. 10. Half of Saw-cut Width Dependence of Kc,n by SENB Method. (all Labs)

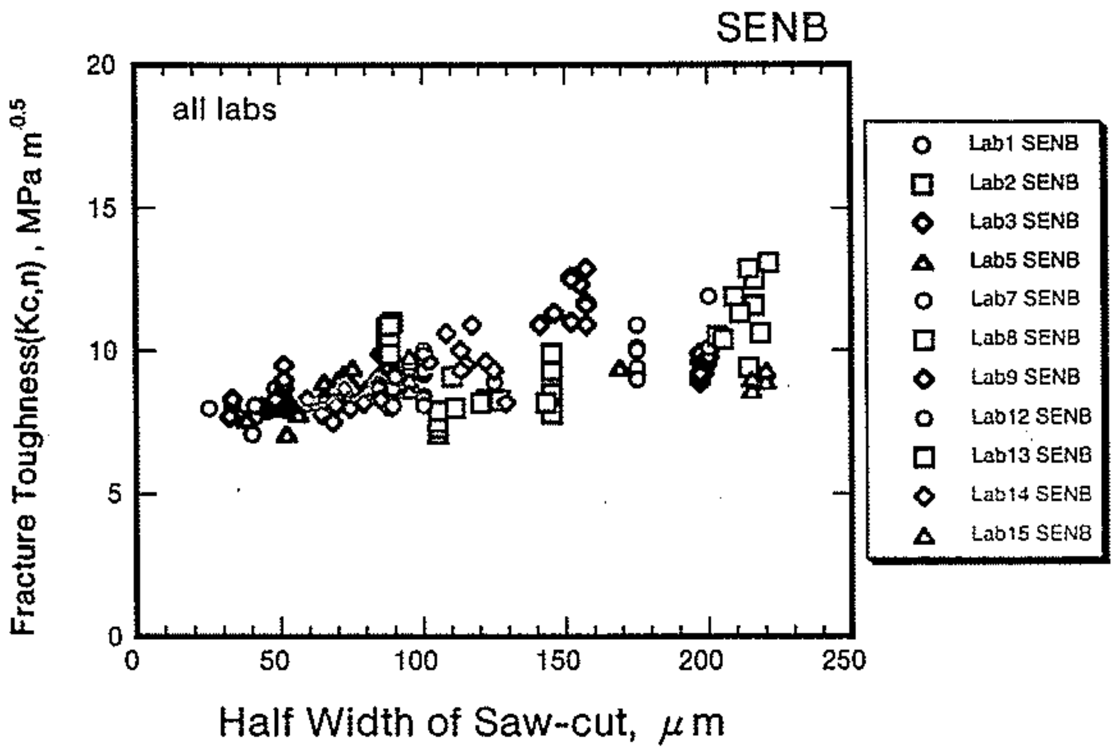


Fig. 11. Notch Tip Radius Dependence of Kc,n by SEVNB Method. (all Labs)

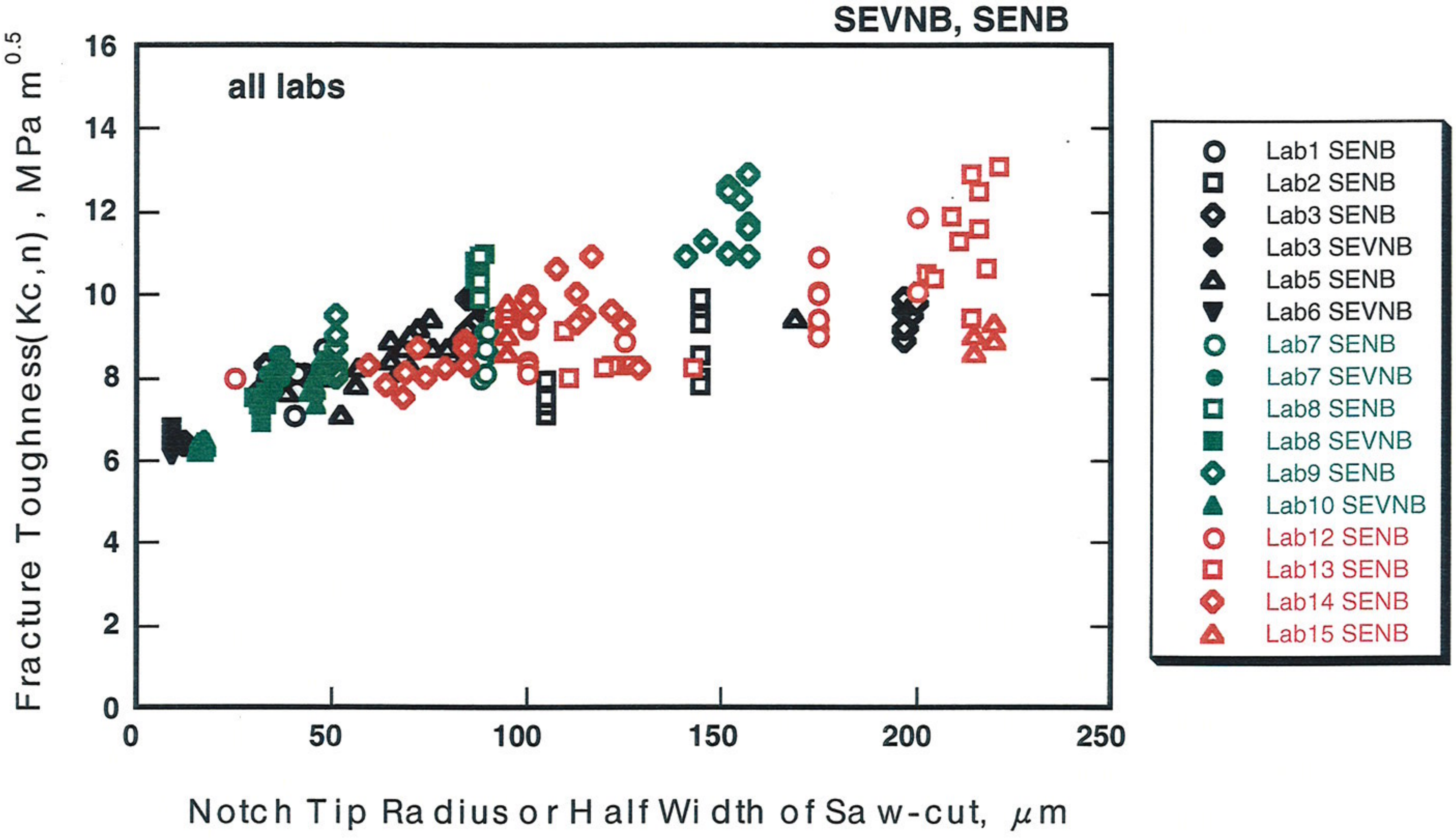


Fig.12. Half of Saw-cut and Notch Tip Radius Dependence of $K_{c,n}$ by SENB and SEVNB Methods. (all labs)

As for the SEPB method, the average from average results from all labs was 6.8 $MPa\cdot m^{0.5}$. On the other hand, the average from average SEVNB results from all labs, whose V-notch tip radius was less than 50 μm, gives 6.8 $MPa\cdot m^{0.5}$. These average values are just the same. However, the SEVNB average from labs, whose V-notch tip radius was less than 20 μm, gives 6.3 $MPa\cdot m^{0.5}$. This is lower than the SEPB data. The SEPB specimen has some whiskers or reinforcing links behind the crack front tip, but the SEVNB has no links behind the notch tip. The force needed to break a precracked SEPB specimen is considered to be higher than that for a notched SEVNB specimen.

Ceramics are very sensitive to the defects and the high strength ceramic parts usually requires precise machining. In the fracture toughness measurement, the introduction of a defect into a specimen is very important and requires the precise technique. The precracking in the SEPB requires some techniques, experience and accurate jigs, and has caused some trouble to some labs in the round robin. However, some loabs have no trouble in precracking. This method has been adopted as JIS standard and will be the ISO standard in the near future. It is the most popular method for fracture toughness measurement at present, but some improvement in precracking process is desirable.

The SEVNB has an advantage because precracking process is not necessary. However the precise V-notch machining is required. The V-notch is introduced by a diamond wheel with a V-shape tip or by hand machining with a razor blade and diamond slurry. With some experience, the tip radius less than 30 mm would be possibly introduced into a specimen. This method will be an good method for fracture toughness measurement.

In the result, the SEPB and SEVNB methods are recommended for the fracture toughness measurement of ceramics.

5. CONCLUSIONS and RECOMMENDATIONS

Fifteen labs in seven countries participated in the room-temperature fracture toughness round robin. The material used was silicon nitride ceramic with 20 vol.% of silicon carbide whisker as reinforcement. The values of fracture toughness, K_{IC} (obtained by the SEPB method) and $K_{c,n}$ (by the SENB and

SEVNB methods), were measured using four different flexure modes; 3-p with a 16 mm span, 3-p with a 30 mm span, 3-p with a 40 mm span, and 4-p with 40 & 20 mm spans. The analyzed results are as follows:

(1) The fracture toughness of whisker reinforced silicon nitride ceramics can be measured by the SEPB and SEVNB methods.

(2) The introduction of precrack into a specimen in the SEPB method caused trouble in several labs. The success rates for precracking were varied from 100 % to 0 %. The accurate jigs and some experience are considered to be required to precrack the SEPB specimens reliably. The detailed specification of the jigs may be necessary.

(3) The profiles of a saw-cut and a V-notch were different among labs.

(4) No effect of flexure mode on the fracture toughness is obviously observed in all the methods. However, in the SEPB method, a short span of 16 mm may cause a larger K_{IC} Value than those from the other modes..

(5) The average K_{IC} values from five labs which measured 5 and more data were in the range from 6.1 to 7.2 $MPa \cdot m^{0.5}$, suggesting the SEPB is the proper method for standard.

(6) $K_{c,n}$ values show saw-cut width or V-notch tip radius dependence. The SENB values range from 6 to 13 $MPa \cdot m^{0.5}$ and show much large scatter. The SEVNB values from the specimens with a V-notch tip radius less than 20 μm are in the range from 6.2 to 6.5 $MPa \cdot m^{0.5}$. The SEVNB measurement with a radius from 10 to 30 μm are recommended as standard method.

(7) As for an average from average results from labs, the SEPB average and the SEVNB average in case of V-notch tip radius less than 50 μm were the same, 6.8 $MPa \cdot m^{0.5}$. However, the SEVNB average in case of the radius less than 20 μm gives 6.3 $MPa \cdot m^{0.5}$, and is lower than the SEPB average.

(8) In this round robin test as the VAMAS project, the results give an important understanding of fracture toughness testing, of the ability of a range of laboratories to make meaningful tests. These results are expected to contribute to future standardization.

These are the recommendations:

(1) The SEPB and SEVNB are the proper methods for fracture toughness measurement of whisker reinforced silicon nitride.

(2) The specification for accurate jigs and some experience are desirable in order to precrack the SEPB specimens reliably.

(3) The flexure mode of 3-point or 4-point with a supporting span of 16, 30 or 40 mm does not affect the fracture toughness values except the SEPB values with a 3-p with 16 mm span.

(4) The V-notch tip radius in the SEVNB should be less than 30 μm. Under this condition, the obtained $K_{c,n}$ shows small scatter.

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APPENDICES

Appendix-1 Instructions for VAMAS Round Robin on Fracture Toughness of Ceramic Matrix Composite.

Appendix-2 Data sheets of K_{IC} by the SEPB method.

Appendix-3 Data sheets of $K_{c,n}$ by the SENB and SEVNB methods.

INSTRUCTIONS FOR VAMAS ROUND ROBIN ON FRACTURE TOUGHNESS OF CERAMIC MATRIX COMPOSITE

CONTENTS

1. Introduction

The purpose of the '94 VAMAS round robin is to assess methods to measure room temperature fracture toughness of ceramics matrix composite. To evaluate the fracture toughness, three methods adopted for the round robin are as follows :

Single Edge Pre-cracked Beam (SEPB) method.
Single Edge Notched Beam (SENB) method.
Single Edge V-Notched Beam (SEVNB) method.

As a standard method, Japan Industrial Standard adopts the SEPB method[1] and German Standard does the SENB and the similar method to the SEPB [2].

The SEPB method uses a natural crack that is introduced into a specimen by a bridge-indentation fixture. This technique was already adopted twice in the '89 and '90 VAMAS round robin on fracture toughness of advanced ceramics proposed by Japan Fine Ceramics Center[3,4].

The SENB and SEVNB methods use specimens with a saw-cut and V-notch, respectively. The SEVNB is an improved method of the SENB method and uses a V-shaped notch with a very small tip radius from 6 to 40μm[5].

The fracture toughness is considered to show saw-cut width or notch tip radius dependence due to difference in stress concentration and machining damage at the bottom of a saw-cut or notch tip[6,7]. Investigation of the notch shape dependence of fracture toughness is the one of the objectives of this round robin.

All the participants are required to measure fracture toughness both by the SEPB method with one condition and by the SENB and/or SEVNB method with two conditions.

Specimens for each method are prepared by JFCC. Each participant introduces a precrack and a saw-cut and/or V-notch into the specimens and then measures the fracture toughness by the two or three methods.

The results will be compiled and analyzed in the VAMAS Report. Obtained data and findings are considered to be useful for the standardization of advanced ceramics evaluation.

2. Common Test Conditions in Three Methods

2.1 Specimen

2.1.1 Material

The material used for this round robin is silicon carbide whisker reinforced silicon nitride, Kryptonite[8], which is manufactured by Japan Metals and Chemicals Co.Ltd., Tokyo, Japan.

Properties of the material are shown in Table 1.

Table 1. Properties of $SiC(w)/Si_3N_4$, Kryptonite

Manufacturer	Japan Metals & Chemicals Co.,Ltd.
Main Component	Silicon Nitride
SiC-whisker	20 vol%
Density	3.20 g/cm^3
Porosity	less than 0.1%
Flexural Strength	880 MPa (RT)
	880 MPa (1000°C)
	490 MPa (1100°C)
Fracture Toughness	6.2 MPa·m$^{0.5}$
Thermal Expansion Coefficient	4.1x 10^{-6} °C^{-1} (RT to 1200°C)
Thermal Conductivity	19.2 W/m·K (RT)
	14.0 W/m·K (1200°C)
Thermal Shock	950 °C

2.1.2 Shape and Dimensions of Specimen

The Specimens distributed to participants are of prism shape in rectangular cross section with dimensions complying with Fig.1.

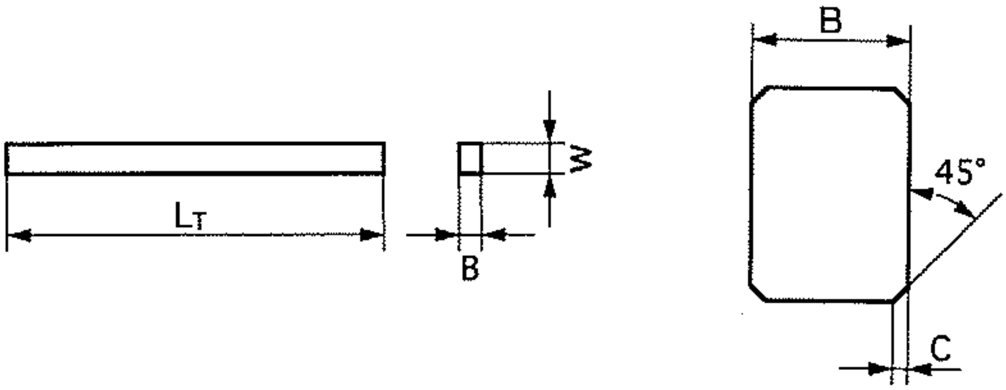


Fig.1. Dimensions of Specimen.

Length overall (L_T) : 48.0 mm, Width(W) : 4.0±0.1 mm

Thickness(B) : 3.0±0.1 mm, c : 0.1 ~ 0.3 mm

Four edges of all specimens for the three methods are chamfered. The parallelism[9] of upper and lower faces is better than 0.01 mm. The roughness of top, bottom, and side surfaces of a test piece is better than 0.2 μm Ra specified in JIS B 0601[10].

2.2 Apparatus and Support Devices

2.2.1 Testing Machine

A suitable testing machine, capable of keeping a uniform crosshead speed should be used. The machine should be capable of measuring to ±1% of the true load for the accuracy on load indication.

Fracture toughness at room temperature is measured in air. If the toughness is measured under special conditions, these conditions should be noted with the result.

2.2.2 Flexure fixture

The material of flexure fixtures at supporting and loading points should have elastic modulus of 147 GPa($1.5\text{x}10^4$ kgf/mm^2) or more, without plastic deformation and rupture during the test.

The devices for 3-point flexure or 4-point flexure are used for the SEPB, SENB and SEVNB methods. Participants choose one of the following conditions:

3-point flexure with a span of 30 mm.
3-point flexure with a span of 40 mm.
4-point flexure with spans of 40 and 20 mm.

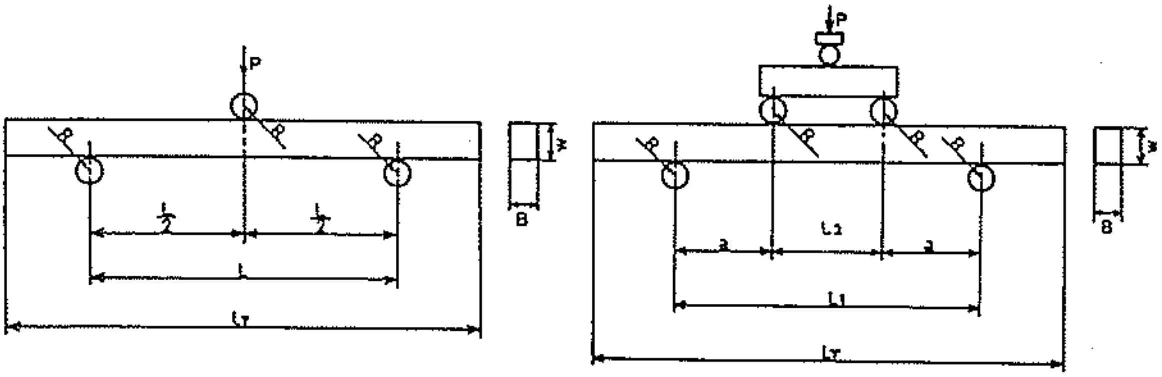
The radii of curvature of both supporting and loading rollers are shown in Fig.2.

2.3 Distribution of Specimens

The number of specimens per condition is ten. Thirty specimens in total, meeting the requirements mentioned above, are distributed to each participant from Japan Fine Ceramics Center (JFCC). Each participant should introduce a precrack into 10 specimens and a saw-cut and/or V-notch with two different conditions into 20 specimens.

2.4 Deadline for Returning Data

All the data sheets and other data should be sent back to JFCC by the end of January 1995.



(a) Three-point Flexure

Radius of curvature (R) : 2.0 to 4.0 mm

Support span (L) : 30±0.5 mm, or 40±0.5 mm

(b) Four-point Flexure

Radius of curvature (R) : 2.0 to 4.0 mm

Support span (L_1) : 40±0.5 mm

Load span (L_2) : 20±0.5 mm

Fig.2. Dimensions and Radius of Curvature of Flexure Fixture.

Testing Procedure

3.1 SEPB Method

3.1.1 Measuring the Dimensions of Specimen

Give a specific number to ten specimens. The specimens should be cleaned of adhesive materials in acetone.

Measure the thickness(B) and width(W) of the specimen by micrometer calipers. Read the values in mm down to three decimal places.

3.1.2 Introducing a Precrack

Introduce a Vickers impression, as shown in Fig.3, into the center region of the 3 mm-width surface. Three indentations may be used if precracking using one is unsuccessful.

Fig.4 shows an example of the precracking fixture consisting of a pusher and an anvil with a deflection groove.

Clean the fixture and the specimen surfaces with acetone. Place the specimen on the support groove with the crack starter side down. The precrack starter should be located just above the center of the deflection groove.

Attach a sonic sensor on one face of the framework of the pusher to detect the pop-in sound.

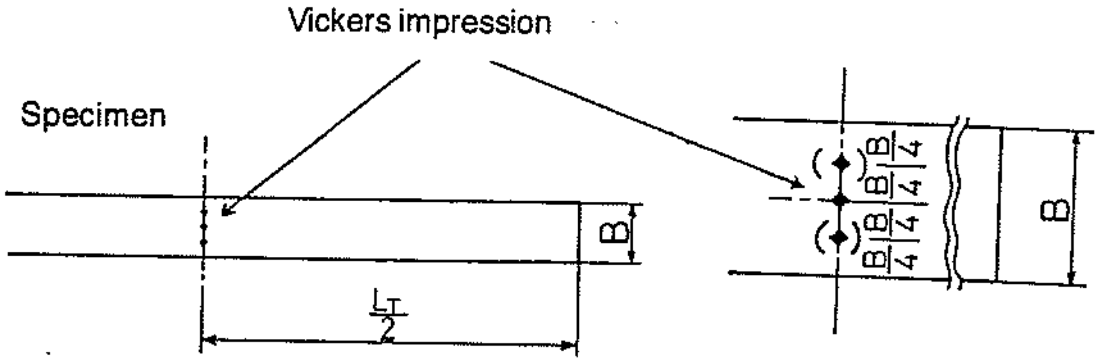


Fig.3 Precrack starter (Vickers Impression(s)) and its site.

Number of Impression	: 1 (or 3, if necessary)
Indent direction	: One diagonal of Vickers impression(s) should be parallel to the specimen thickness direction.
Indent load	: 98 N (10 kgf)

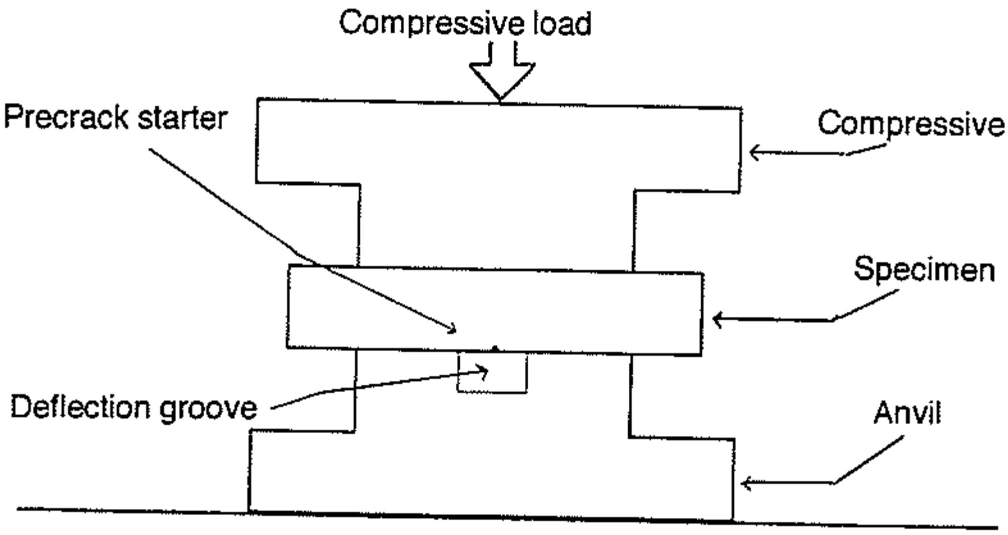


Fig.4 Precracking fixture

Width of the groove(G) : 4 mm or 5 mm.

Increase the compressive load gradually until a pop-in sound is detected by the sonic sensor. The load should not be increased further after a pop-in crack is introduced.

Adjust the precracking conditions to obtain a valid precrack length ranging from 1.2 to 2.4 mm.

EXAMPLE : 1 impression, 5 mm-width groove,
Load for precracking ; about 1.5~2.0 x 10^4N.

3.1.3 Measuring Fracture Load and Precrack Length

Before fracturing a precracked specimen, dye penetrant, e.g., oil paint, mixed with acetone should be used to improve the visibility of the precrack region. After drying the penetrant , measure the fracture load at a crosshead speed is 0.5 mm/min. The fracture load is defined as the maximum load when a specimen is loaded. Record load to 3 significant figures.

The precrack length is measured by observing the fracture surface of a specimen, using a microscope of more than 20x magnification.

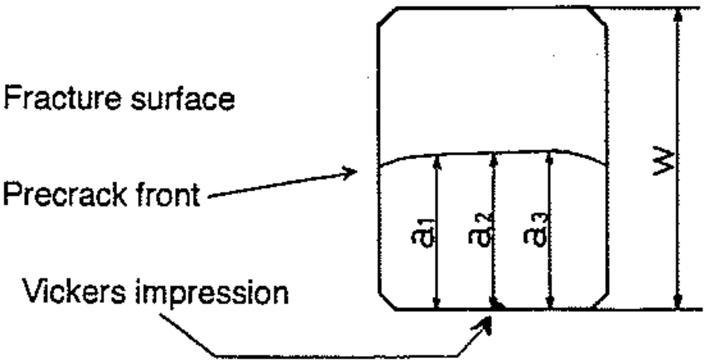


Fig.5 Measurement of precrack length.

Lengths a_1,a_2 and a_3 are shown in Fig.5. Read them down to a decimal place. An average length of a precrack (a) is calculated using eqn.1. These values shall be in the range from 1.2 to 2.4 mm and satisfy eqn.2 to obtain valid K_{IC} values.

$$a=(a_1+a_2+a_3)/\ 3 \text{---------} \ (1)$$

$$(a_{max}-a_{min})/\ a \leq 0.1 \text{---------} \ (2)$$

a_{max} : maximum among a_1, a_2 and a_3 (mm)
a_{min} : minimum among a_1, a_2, and a_3 (mm)

A precrack must be perpendicular to the specimen surfaces within 10° as shown in Fig.6.

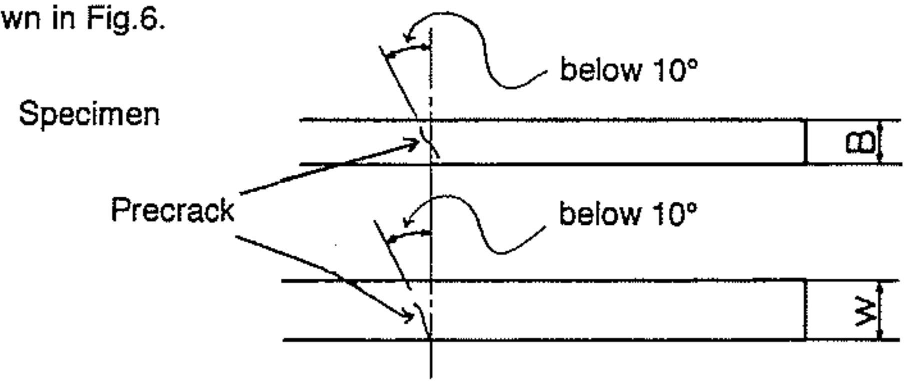


Fig.6 Angle allowance of precrack extension.

3.1.4 Calculation of K_{IC}

In case of 3-point flexure with a span of 30 mm, calculate K_{IC} according to eqns.3, 4 and 5[1].

$$K = \frac{PS}{BW^{1.5}} \cdot \frac{3F(\alpha)\,\alpha^{0.5}}{2} \quad \text{--------- (3)}$$

$$F(\alpha) = 1.964 - 2.837\alpha + 13.71\,\alpha^2 - 23.25\,\alpha^3 + 24.13\,\alpha^4 \quad \text{------ (4)}$$

$$\alpha = a/W \quad \text{--------- (5)}$$

In case of 3-point flexure with a span of 40 mm, calculate K_{IC} according to qns.3, 6 and 5 [11].

$$F(\alpha) = 1.972 - 2.746\,\alpha + 13.44\,\alpha^2 - 22.84\,\alpha^3 + 23.86\,\alpha^4 \quad \text{------- (6)}$$

In case of 4-point flexure with spans of 40 and 20 mm, calculate K_{IC} according to eqns.7, 8 and 5 [12].

$$K = \frac{P(S_1 - S_2)}{BW^{1.5}} \cdot \frac{3F(\alpha)\,\alpha^{0.5}}{2(1-\alpha)^{1.5}} \quad \text{--------- (7)}$$

$$F(\alpha) = 1.9887 - 1.326\alpha - \frac{(3.49 - 0.68\alpha + 1.35\alpha^2)\alpha(1-\alpha)}{(1+\alpha^2)} \quad \text{-------(8)}$$

K_{IC} : Fracture toughness ($MPa{\cdot}m^{0.5}$)

P	: Fracture load (MN),	S, S_1, S_2	: Support span (m)
B	: Thickness of specimen (m),	α	: Initial precrack relative length (m)
a	: Precrack length (m),	W	: Width of specimen (m)

Perform all calculations to three significant figures. Calculate the average K_{IC} value, and round it off to one decimal place.

3.1.5 Data on K_{IC} by SEPB Method

Data sheet(A) should be filled with the experimental and calculated data, and should be returned to JFCC.

3.2 SENB and SEVNB Method

3.2.1 Specimen

Participants do the fracture toughness measurement with two different conditions by the SENB and/or SEVNB method. The number of specimens per condition is 10. Participants should choose two sizes of saw-cut widths and/or V-notch radii and introduce them into one of the 3 mm-width face of a specimen using a diamond wheel cutter.

The width and radius are recommended to be 50 to 500 μm and 6 to 40 μm, respectively. The depth of a saw-cut and/or V-notch depth is recommended to be 1.5 mm.

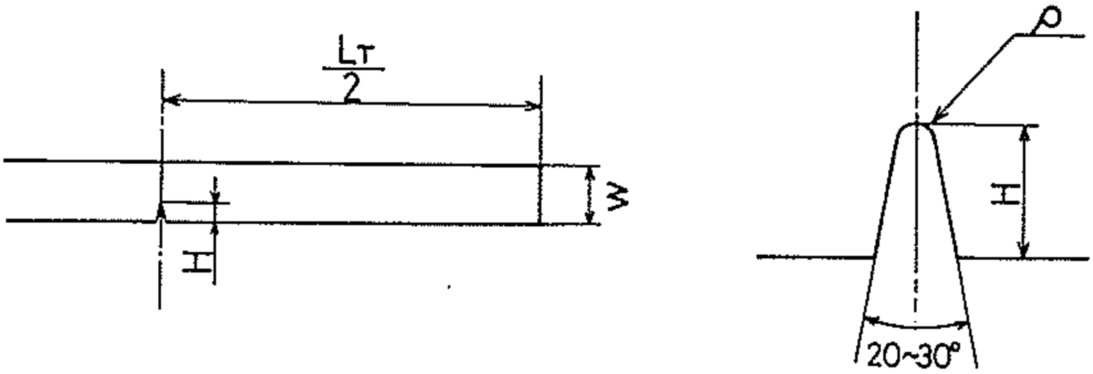


Fig.7 Shape of V-notch

Depth (H) : 1.5 mm, Tip radius (ρ) : 6~40μm

3.2.2 Measuring Dimensions of Specimen

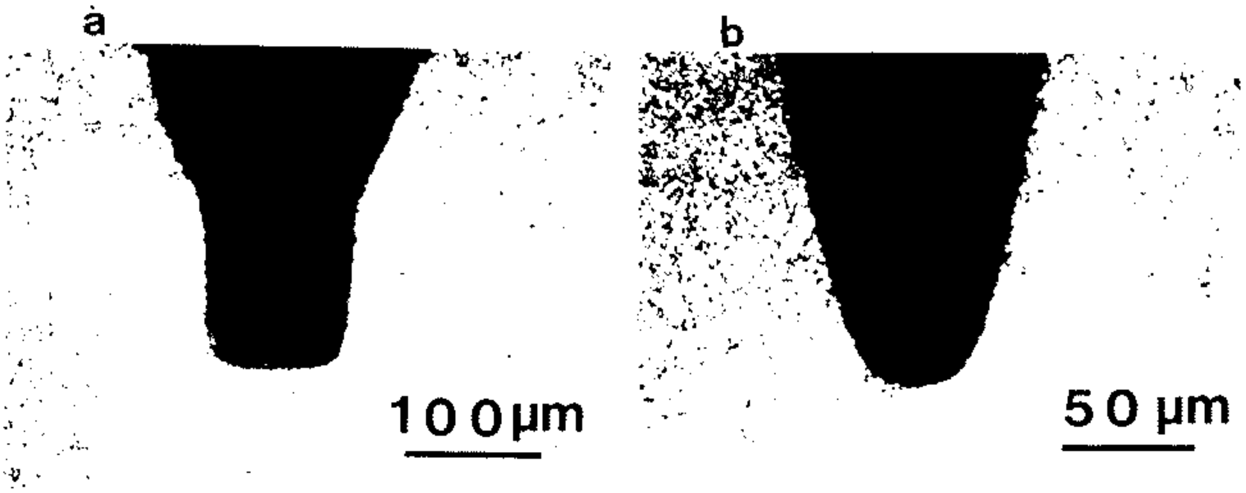
Give a specific number to each of the 20 specimens. After cleaning the specimens in acetone, be sure to take photographs of all specimens showing the tip of a saw-cut and/or V-notch.

Measure the thickness and width of the specimen by micrometer calipers. Read the values down to three decimal places.

Measure the width of SENB notches and the tip radius of SEVNB notches on a microscope of 20 to 400x magnification. Calibrate the microscope magnification using a graticule. The photographs of the notch are convenient for reading the value (Fig.8). In case of a V-notch tip showing an asymmetric or non-uniform shape, measure both the maximum and minimum radii.

3.2.3 Measuring Fracture Load and Average Depth

Place the 3 mm-width face with a saw-cut and/or V-notch down. The fracture load is defined as the maximum load when a specimen is loaded. Record load to 3 significant figures. Crosshead speed is 0.5 mm/min.



(a) Saw-cut width=100 μm (b) Tip radius=30 μm

Fig. 8. Photographs of a Saw-cut and V-notch.

The depth of a saw-cut and/or V-notch is measured by observing fracture surface (Fig.9), using a microscope of more than 20x magnification. Photograph of the surface is convenient for reading the values but should be of calibrated magnification using a graticule. Read the depth, a_1, a_2 and a_3, in mm down to 2 decimal places (i.e.±0.01 mm). An average depth of a saw-cut and/or V-notch is calculated in the same way as an average precrack length, using eqn.1 (see 3.1.3).

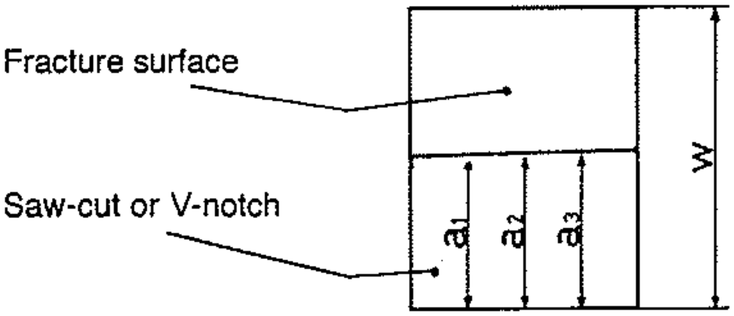


Fig.9. Measurement of Saw-cut or V-notch depth.

3.2.4 Calculation of Fracture Toughness(Kc,n)

The fracture toughness, Kc,n, is calculated according to eqns.3~8 as the same as those for the SEPB values. However precrack length in the SEPB should read saw-cut or V-notch depth.

The significant figure of the value is three.

Calculate the average Kc,n value and round it off the fractions to a decimal place.

3.2.5 Data Sheets on Kc,n by the SENB and SEVNB Methods

Data sheets (B) and (C) for the SENB and SEVNB data, respectively, should be filled with the experimental and calculated data. They should be returned to JFCC with photographs of all the specimens showing the tip of a saw-cut and/or V-notch.

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Data Sheet (A)---**SEPB** Round Robin Results

Testing Laboratory: Investigator:

1.Precrack Starter

Indentation load; N

Number of indent;

2.Precrack Length Measurement

Dyeing;

Comments;

3.Flexure Test Procedure

Test temperatire; °C Relative humidity; %

Machine; Load cell capacity;

Flexure type; -point flexure Span(s);

Crosshead speed; mm/min Avr. time to failure;

4.Results

Specimen Identity	B	W	a_1	a_2	a_3	**a**	Load P	K_{IC}	K_{IC}(avr)
	(m)	(m)	(m)	(m)	(m)	(m)	(N)	(MPa$m^{0.5}$)	

Comment:

Data Sheet (B)---**SENB** Round Robin Results

Testing Laboratory: Investigator:

1.Saw-cut

Machine;

Machining condition;

2..Flexure Test Procedure

Test temperature; °C Relative humidity; %

Machine; Load cell capacity;

Flexure type; -point flexure Span(s);

Crosshead speed; mm/min Avr. time to failure;

3.Results

Specimen Identity	B (m)	W (m)	a_1 (m)	a_2 (m)	a_3 (m)	**a** (m)	Saw-cut (μm)	Load P (N)	K_{cn} ($\mathrm{MPam}^{\cdot 0.5}$)	K_{cn}(avr)

Comment:

Data Sheet (C)---**SEVNB** Round Robin Results

Testing Laboratory:

Investigator:

1.V-notch

Machine;

Machining condition;

2..Flexure Test Procedure

Test temperature; °C

Relative humidity; %

Machine;

Load cell capacity;

Flexure type; -point flexure

Span(s);

Crosshead speed; mm/min

Avr. time to failure; sec.

3.Results

Specimen Identity	B	W	a_1	a_2	a_3	**a**	ρ	Load P	K_{cn}	K_{cn}(avr)
	(m)	(m)	(m)	(m)	(m)	(m)	(μm)	(N)	(MPa$m^{-0.5}$)	

Comment:

Data sheets of K_{IC} by the SEPB Method.

SEPB	Flexure type & Span(s) ,mm	K_{IC} ,MPa$\sqrt{m}$		Number of Data	Specimen No.	K_{IC} Value ,MPa$\sqrt{m}$
		Average	Standard deviation			
Lab 1	4P-40,20	6.3		2	32–3	6.8
					35–5	5.8
Lab 2	3P-30	5.6	-	1	33– 6	5.6
Lab 3	4P-40,20	7.6	0.1	3	10--2	7.5
					38–6	7.7
					27–9	7.5
Lab 4	4P-40,20	6.7	0.4	6	5	6.6
					6	7.2
					7	6.7
					8	6.1
					9	6.9
					10	6.5
Lab 5	4P-40,20	7.4	0.4	3	1–10	6.9
					14--12	7.8
					6--2	7.6
Lab 6	3P-16	7.2	0.2	10	2–6	7.6
					6--11	7.2
					11--3	7.1
					15--8	7.4
					19--13	7.2
					24--5	7.0
					28–10	7.1
					33–4	7.1
					38--1	7.5
					42--10	7.0
	3P-30	6.7	0.1	10	3–12	6.8
					8--4	6.5
					12–9	6.6
					17–1	7.0
					21–6	6.7
					25--11	6.7
					30--3	6.7
					34–11	6.7
					39–8	6.6
					44--5	6.6

Data sheets of K_{IC} by the SEPB Method.

SEPB	Flexure type & Span(s) ,mm	K_{IC} ,MPa√m		Number of Data	Specimen No.	K_{IC} Value ,MPa√m
		Average	Standard deviation			
	3P-40	6.9	0.2	10	5--5	6.3
					9--10	6.9
					14--2	7.0
					18--7	7.0
					22--12	6.9
					27--4	7.0
					31--9	6.8
					36--6	6.8
					41--3	6.9
					45--12	7.0
	4P-40,20	6.7	0.1	10	1--9	6.7
					6--1	6.9
					10--6	6.8
					14--11	6.6
					19--3	6.9
					23--8	6.5
					27--13	6.7
					32--6	6.7
					37--3	6.6
					41--12	6.7
Lab 7	4P-40,20	6.6	0.2	8	11--1	6.5
					12--7	6.6
					13--13	6.8
					15--6	6.8
					18--5	6.8
					31--7	6.6
					3--10	6.6
					5--3	6.3
Lab 8	3P-30	6.7	0.2	10	2--2	6.8
					3--8	6.5
					5--1	6.6
					6--7	6.8
					7--13	6.6
					9--6	6.8
					10--12	6.8
					12--5	6.5
					13--11	6.8
					29--12	6.9

Data sheets of K_{IC} by the SEPB Method.

SEPB	Flexure type & Span(s) ,mm	K_{IC} ,MPa$\sqrt{m}$		Number of Data	Specimen No.	K_{IC} Value ,MPa$\sqrt{m}$
		Average	Standard deviation			
Lab 9	3P-30	7.2	0.4	7	40--1	7.9
					50--5	6.9
					30--12	7.2
					43--12	7.3
					50--11	6.9
					29--10	7.0
					7--11	6.9
Lab 10	3P-16	7.0	0.3	10	1--12	6.8
					3--5	6.5
					4--11	7.0
					6--4	7.1
					7--10	6.8
					9--3	6.8
					10--9	7.1
					12--2	6.9
					13--8	6.9
					15--1	7.4
Lab 11	4P-40,20	6.1	0.2	5	4	5.8
					5	5.9
					7	6.3
					8	6.3
					9	6.2
Lab 12	4P-40,20	7.1	0.4	9	17--9	7.5
					38--9	7.0
					13--4	7.6
					26--6	6.5
					30--11	6.8
					27--12	6.7
					1--8	7.1
					3--1	7.3
					4--7	7.1
Lab 15	4P-40,20	6.3	-	2	33-- 3	6.4
					34--10	6.1

Data sheets of $K_{c,n}$ by the SENB and SEVNB methods.

	Test Method	Flexure type & Span(s)-mm	$K_{c,n}$,MPa√m		Number of Data	Specimen No.	V-notch Radius , μm	Half Width of Saw-cut , μm	$K_{c,n}$ Value ,MPa√m
			Average	Standard Deviation					
Lab 1	SENB	4P-40/20	7.9	0.4	9	2–12		33	8.0
						1– 6		41	7.7
						10– 3		43	8.1
						11–9		33	8.0
						13– 2		48	8.7
						5–11		40	7.1
						7– 4		45	7.9
						8–10		35	7.7
						18–13		41	8.1
Lab 2	SENB	3P-30	7.5	0.3	6	38–30		105	7.9
						41–50		105	7.5
						44–70		105	7.1
						36–80		105	7.3
						42–12		105	7.5
						2– 8		105	7.9
	SENB	3P-30	8.8	0.9	6	14–40		145	9.5
						15–10		145	9.3
						17–30		145	8.5
						23–10		145	8.0
						24–70		145	7.8
						12–11		145	9.9
Lab 3	SENB	4-P40/20	9.5	0.4	10	32–2		197	9.6
						4–4		199	9.5
						23–4		198	9.7
						1–5		198	9.2
						17–6		200	10
						6–8		197	9.1
						5–10		197	8.9
						2–11		197	9.2
						21–11		197	9.9
						15–13		200	9.8
	SENB	4P-40/20	9.1	0.6	8	26–3		85	9.9
						43–3		88	9.4
						20–5		86	8.4
						14–7		86	8.3
						41–8		87	9.3
						24–10		85	9.1
						44–10		87	9.9
						18–12		86	8.5
	SENB	4P-40/20	8.0		2	7– 3		32	7.7
						36–11		33	8.3

Data sheets of $K_{c,n}$ by the SENB and SEVNB methods.

	Test Method	Flexure type & Span(s)-mm	$K_{c,n}$,MPa√m		Number of Data	Specimen No.	V-notch Radius , μm	Half Width of Saw-cut , μm	$K_{c,n}$ Value ,MPa√m
			Average	Standard Deviation					
	SEVNB	4P-40/20	6.3	0.1	10	45–7	14		6.3
						46–3	12		6.3
						48–8	12		6.4
						4–13	12		6.5
						9–5	13		6.4
						49–5	14		6.3
						17–10	14		6.3
						40–10	13		6.3
						47–10	13		6.3
						40–4	14		6.3
Lab 5	SENB	4P-40/20	7.8	0.4	6	23–7		52	7.1
						32–7		57	8.2
						34–2		38	7.6
						35–9		56	7.8
						40–6		46	8.1
						45–3		48	8.0
	SENB	4P-40/20	8.8	0.4	13	37–4		70	8.2
						38–11		72	9.2
						42–1		67	8.1
						43–8		65	8.4
						30–13		90	9.2
						17–11		75	9.4
						19–4		76	8.7
						20–10		81	8.5
						28–1		70	9.0
						22–3		83	9.0
						23–9		65	8.9
						25–2		68	8.7
						26–8		80	8.7
	SENB	4P-40/20	9.4		1	16–5		169	9.4
Lab 6	SEVNB	3P-16	6.5	0.2	10	1–4	9		6.6
						5–9	9		6.1
						10–1	9		6.5
						14–6	9		6.4
						18–11	9		6.5
						23–3	9		6.5
						27–8	9		6.4
						32–1	9		6.8
						36–10	9		6.4
						41–7	9		6.4

Data sheets of $K_{c,n}$ by the SENB and SEVNB methods.

	Test Method	Flexure type & Span(s)-mm	Kc,n ,MPa√m		Number of Data	Specimen No.	V-notch Radius , μm	Half Width of Saw-cut , μm	Kc,n Value ,MPa√m
			Average	Standard Deviation					
	SEVNB	3P-30	6.4	0.1	10	2--10	9		6.4
						7--2	9		6.3
						11--7	9		6.4
						15--12	9		6.5
						20--4	9		6.2
						24--9	9		6.3
						29--1	9		6.5
						33--8	9		6.4
						38--5	9		6.5
						43--2	9		6.5
	SEVNB	3P-40	6.3	0.1	10	3--2	9		6.3
						7--7	9		6.1
						11--12	9		6.4
						16--4	9		6.3
						20--9	9		6.3
						25--1	9		6.5
						29--6	9		6.3
						34--1	9		6.4
						38--10	9		6.3
						43--7	9		6.3
	SEVNB	4P-40/20	6.2	0.1	10	4--3	9		6.2
						8--8	9		6.2
						12--13	9		6.3
						17--5	9		6.3
						21--10	9		6.1
						26--2	9		6.5
						30--7	9		6.1
						35--3	9		6.3
						39--12	9		6.1
						44--9	9		6.1
Lab 7	SENB	4P-40/20	8.7	0.5	10	25--9		88	8.8
						28--8		90	8.6
						9--8		88	8.0
						19--11		88	8.4
						21--4		89	8.1
						8--2		92	9.5
						6--9		92	9.2
						22--10		89	9.1
						24--3		89	8.7
						27--2		90	9.1

Data sheets of $K_{c,n}$ by the SENB and SEVNB methods.

	Test Method	Flexure type & Span(s)-mm	Kc,n ,MPa√m		Number of Data	Specimen No.	V-notch Radius , μm	Half Width of Saw-cut , μm	Kc,n Value ,MPa√m
			Average	Standard Deviation					
	SEVNB	4P-40/20	8.2	0.3	10	37--11	36		8.3
						36--4	37		8.0
						34--9	35		8.2
						30--1	38		8.3
						45--10	36		8.6
						33--2	37		8.6
						41--1	38		8.2
						42--8	35		8.0
						39--6	33		8.1
						44--3	35		7.7
Lab 8	SENB	3P-30	10.6	0.4	10	18--3		87	10.8
						15--4		89	11.0
						25--7		87	10.4
						34--7		87	10.8
						36--2		88	10.8
						24--1		88	10.7
						28--6		88	10.8
						16--10		88	10.3
						32--		88	9.9
						26--		88	10.9
	SEVNB	3P-30	7.4	0.2	10	45--8	31		7.3
						39--4	32		7.4
						40--11	33		7.3
						31--5	33		7.6
						19--9	32		6.9
						37--9	30		7.5
						21--2	33		7.3
						22--8	32		7.4
						42--6	31		7.4
						44--1	34		7.6
Lab 9	SENB	3P-30	8.4	0.5	10	3--1		51	8.3
						4--8		50	8.1
						7--12		48	8.1
						8--13		51	9.5
						12--4		51	8.7
						13--5		51	8.0
						16--9		51	8.1
						17--10		50	8.3
						21--1		48	8.3
						22--2		51	9.0

Data sheets of $K_{c,n}$ by the SENB and SEVNB methods.

	Test Method	Flexure type & Span(s)-mm	$K_{c,n}$,MPa$\sqrt{m}$		Number of Data	Specimen No.	V-notch Radius , μm	Half Width of Saw-cut , μm	$K_{c,n}$ Value ,MPa$\sqrt{m}$
			Average	Standard Deviation					
Lab 10	SENB	3P-30	11.8	0.8	10	26--7		157	11.7
						25--6		157	10.9
						35--8		141	10.9
						39--3		157	11.6
						46--1		155	12.3
						46--7		152	11.0
						47--8		146	11.3
						48--3		152	12.6
						48--9		157	12.9
						49--10		152	12.5
	SEVNB	3P-30	6.3	0.1	10	16--7	18		6.3
						17--13	17		6.2
						19--6	17		6.3
						20--12	18		6.4
						22--5	17		6.5
						23--11	16		6.4
						25--4	17		6.3
						26--10	15		6.2
						28--3	17		6.2
						29--9	17		6.2
	SEVNB	3P-30	7.8	0.3	10	31--2	45		7.8
						32--9	45		7.6
						34--4	44		7.6
						35--11	45		7.9
						37--6	45		7.9
						39--1	47		8.4
						40--8	45		7.9
						42--3	46		7.3
						43--10	46		7.7
						45--5	46		7.7
Lab 12	SENB	4P-40/20	10.1	1	8	32--5		175	9.2
						33--12		175	9.4
						11--11		200	10.1
						41--1		200	11.9
						10--5		175	9.0
						35--7		175	10.1
						23--7		175	10.9
						24--13		175	10.0

Data sheets of $K_{c,n}$ by the SENB and SEVNB methods.

	Test Method	Flexure type & Span(s)-mm	$K_{c,n}$,$\mathrm{MPa}\sqrt{m}$		Number of Data	Specimen No.	V-notch Radius , μm	Half Width of Saw-cut , μm	$K_{c,n}$ Value ,$\mathrm{MPa}\sqrt{m}$
			Average	Standard Deviation					
	SENB	4P-40/20	9.1	0.7	10	14--10		100	8.4
						5--13		100	9.2
						70--8		100	9.3
						22--1		100	9.5
						45--1		125	8.9
						8--12		100	10.0
						7--6		100	9.3
						19--2		100	8.3
						37--2		100	8.1
						43--6		100	10.0
	SENB	4P-40/20	8.0		1	16--3		25	8.0
Lab 13	SENB	4P-40/20	8.4	0.4	7	STN02		143	8.2
						STN04		125	8.3
						STN05		122	8.3
						STN06		111	8.0
						STN08		127	8.5
						STN09		120	8.2
						STN10		110	9.1
	SENB	4P-40/20	11.5	1.1	11	STN01		203	10.5
						STW01		216	11.6
						STW02		205	10.4
						STW03		221	13.1
						STW04		218	10.6
						STW05		214	9.4
						STW06		216	12.5
						STW07		209	11.9
						STW08		214	12.9
						STW09		209	11.9
						STW10		211	11.3

Data sheets of $K_{c,n}$ by the SENB and SEVNB methods.

	Test Method	Flexure type & Span(s)-mm	$K_{c,n}$,MPa$\sqrt{m}$		Number of Data	Specimen No.	V-notch Radius , μm	Half Width of Saw-cut , μm	$K_{c,n}$ Value ,MPa$\sqrt{m}$
			Average	Standard Deviation					
Lab 14	SENB	4P-40/20	8.3	0.5	10	1--2		84	8.9
						2--2		59	8.3
						3--2		74	8.0
						4--2		68	7.5
						5--2		64	7.8
						6--2		84	8.7
						7--2		85	8.3
						8--2		79	8.2
						9--2		72	8.7
						10-- 2		69	8.1
	SENB	4P-40/20	9.7	0.7	10	1--1		113	10.0
						2--1		115	9.5
						3--1		117	10.9
						4--1		108	10.6
						5--1		122	9.6
						6--1		113	9.3
						7--1		102	9.6
						8--1		125	9.3
						9--1		100	9.9
						10--1		129	8.2
Lab 15	SENB	4P-40/20	9.3	0.5	5	16--13		95	8.6
						18-- 6		95	9.0
						19--12		95	9.4
						21-- 5		95	9.6
						22--11		95	9.8
	SENB	4P-40/20	9.0	0.3	4	2-- 5		215	9.0
						3--11		220	8.9
						5-- 4		215	8.6
						6--10		220	9.3