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JAPAN FINE CERAMICS CENTER
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VAMAS Round Robin on Fracture Toughness of Silicon Nitride at High Temperature.

by

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SUMMARY

Eight laboratories in Germany, Japan, UK and USA participated in the VAMAS Round Robin on high temperature fracture toughness of silicon nitride. This report describes the results of the round robin. The fracture toughness at room temperature and at 1200°C were measured by three methods; the single edge V-notched beam(SEVNB), single edge precracked beam(SEPB) and chevron notched beam(CNB). The obtained values do not show crosshead speed dependence, irrespective of ambient temperature and atmosphere. The values at 1200°C in N_2 can be measured by the SEVNB and SEPB methods with small scatters. The oxidation of silicon nitride, caused by heating in air, increases the SEVNB and SEPB values. The CNB values are free from the effects of ambient temperature and atmosphere but they show a large scatter between laboratories. However, the chevron V-notched beam(CVNB) method, which is an improved CNB method, shows values with a small scatter, irrespective of the measurement conditions. The SEVNB and SEPB measurements in N_2 are recommended for the measurement of high temperature fracture toughness, and the CVNB method can measure the fracture toughness with a small scatter under any conditions.

Keywords: Silicon nitride, Fracture toughness, SEVNB, SEPB, CNB, CVNB, Round robin.

CONTENTS

1. INTRODUCTION

Several round robin tests for mechanical properties of advanced ceramics have been carried out since 1986 as TWA#3 "Ceramics" within the Versailles Project on Advanced Materials and Standards(VAMAS). The international collaboration is aimed at doing prestandardization research[1].

The Japan Fine Ceramics Center organized a room temperature fracture toughness round robin in 1989. Three methods were adopted; single edge precracked beam(SEPB), indentation fracture(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[2] and a reference[3]. Subsequently, Quinn[4,5] compiled the results from 18 labs including additional 5 labs in the U.S.A.

JFCC organized another round robin on high temperature fracture toughness by three methods, single edge V-notched beam(SEVNB), SEPB and chevron notched beam(CNB). Instructions and silicon nitride specimen were sent to 12 labs in December, 1990. Eight labs out of 12 returned their results to JFCC. The last data set was obtained in June in 1993. This report is based on the results from the eight labs in Germany, Japan, U.K., and U.S.A.(Table 1-1)[Appendix A-1].

Several labs were delayed in submitting the data, probably because the fracture toughness tests require high temperature measurement apparatus which includes ceramic jig for 1200°C. Moreover, the SEPB method requires an accurate jigs for precracking and some experience to get reliable data. There must have been some trouble in performing the experiments, resulting in such a long delay.

There are several methods proposed for measuring fracture toughness of advanced ceramics[6]. Among these methods, the SEPB and IF methods became standard methods in Japan[7] in 1990. The SEVNB and CNB are methods under consideration for standardization.

The objective of this round robin is to assess the methods for fracture toughness of silicon nitride, which is a popular high temperature structural material.

2. MATERIALS

Sintered silicon nitride, SSN-H, manufactured by Kyocera Corporation was used for this round robin test. As shown in Table 2-1, SSN-H contains ytterbia and small amounts of alumina as the sintering additives. The sintering process is controlled to promote the growth of β-silicon nitride elongated crystals in the silicon nitride matrix. The largest crystals are sometimes up to 200μm in length.

JFCC measured the fracture toughness by the IF with the indentation load of 98N. The obtained value and the standard deviation at room temperature were 4.9 and $0.3 MPa\cdot m^{0.5}$, respectively.

Forty-five specimens were sent to each participant from JFCC(Table 2-2). Specimens dimensions were (4.0±0.1)×(3.0±0.1)×50mm. The parallelism[8] of upper and lower surfaces was better than 0.01mm. The roughness of top, bottom and side surfaces was not more than 0.2μm Ra specified in JIS B 0601[9]. The specimens for the SEVNB method were V-notched and those for the SEPB method were chamfered by JFCC.

3. EXPERIMENTAL PROCEDURE

The experimental procedures were specified in detail in the instructions [Appendix A-2], which were sent to the participants with 45 specimens. Approximate 5 specimens were used per condition.

3.1 Single Edge V-Notched Beam Method

The SEVNB is an improved version of single edge notched beam(SENB) method. The SENB method generally uses a straight through notch usually with the width of more than 150μm, while the SEVNB uses a V-shaped notch with very small root radius from 10 to 40μm. The fracture occurs from the V-notch tip with a controlled shape so that the fracture toughness shows quite small scatter[10].

The instructions specified that V-notched specimens had to be fractured in 3-point loading with a span of 30mm at both room temperature and 1200°C. Lab 8 adopted 4-point flexure with spans of 40 and 20mm for the 1200°C measurement, and Labs 7 and 8 adopted 3-point flexure with a span of 40mm for the room temperature measurement(Table 3-1).

The $K_{c,n}$ for the 4-point flexure was calculated using equation(1,2)[11].

$$K = \frac{P}{B\sqrt{W}} \frac{S_1 - S_2}{W} \frac{3F(\alpha)\sqrt{\alpha}}{2(1-\alpha)^{1.5}} \quad \cdots\cdots \quad (1)$$

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

where $\alpha = a/W$, K is $K_{c,n}$ or K_{IC}, a is notch depth or precrack length, P is fracture load, B is thickness, W is width, S_1 is outer span and S_2 is inner span. The $K_{c,n}$ for 3-point flexure with 40mm span was calculated using the equation for 3-point flexure with 30mm shown in the instructions[A-2]. This was done because the $F(\alpha)$ vs α curve for a span to width ratio of 7.5 is almost the same as that for a ratio of 10 [12].

The crosshead speeds were 0.005 and 0.5mm/min in all cases.

The fracture toughness for a notched specimen is termed $K_{c,n}$, which is distinguished from K_{IC} as measured with a precracked specimen in this report.

Although introducing a V-notch into a specimen requires high performance machining apparatus and techniques, the SEVNB method is easy to perform.

According to the original instructions, $K_{c,n}$ should be corrected using an equation which includes factors of V-notch root radius and critical distance from the notch tip in order to obtain $K_{c,n}$(corrected). However, the correction of $K_{c,n}$ is not adopted in this report because further examination of the validity of the correction is considered to be necessary.

3.2 Single Edge Precracked Beam Method

The SEPB method uses a pop-in crack. Specimens were precracked from Vickers indent or saw cut on the 3mm wide face. A bridge-anvil was used for precracking. The precrack was dye-penetrated and the specimen fractured at 1200°C in 3-point loading. Lab 7 adopted 3-point flexure with a span of 40mm, and Lab 8 did 4-point with spans of 40 and 20mm. The K_{IC} for the 4-point flexure was calculated using equation(1,2) which is the same as that for the SEVNB method. The crosshead speeds were 0.005 and 0.5mm/min.

This procedure is based on JIS R 1607. The similar specifications are shown in DIN 51 109 [13].

3.3 Chevron Notched Beam Method

The CNB method was also applied to measure the fracture toughness [14].

The specimen was chevron-notched and then fractured at 1200°C in 4-point loading. The spans were either 30 and 10mm or 40 and 20mm. The chevron was triangular with the angle of 60 degree. The notch was straight-through for all the labs except Lab 1 which used a V-notch (Fig.3-1). The crosshead speeds were 0.005 and 0.5mm/min.

The crack is expected to extend stably from the notch tip as load is increased, and then propagate instantaneously. It is unnecessary to measure the crack length at peak load. It is rather difficult to introduce a chevron-notch properly into a specimen.

4. RESULTS AND DISCUSSION

4.1 Single Edge V-Notched Beam Method

Each V-notch tip radius was measured by each lab(Appendix A-3). Cross-sections of a V-notch and the tip are shown in Fig.4-1. Some notch roots in SSN-H specimens did not show a single smooth radius(Fig.4-1c), so the tip radius measurement was difficult and the obtained values seemed to have errors. Therefore V-notch tip radius dependence of $K_{c,n}$ is not discussed in this report, although there seems to be a strong correlation between the $K_{c,n}$ and tip radius.

The average $K_{c,n}$ and the standard deviation(σ_{n-1}) are compiled in Table 4-1 and Figs.4-2a and 2b. The σ_{n-1} for room temperature $K_{c,n}$ shows a small scatter of less than

0.5MPa•$m^{0.5}$. The difference between the maximum and minimum average $K_{c,n}$ values is 1.2MPa•$m^{0.5}$ at a crosshead speed of 0.005 or 0.5mm/min. The values from Lab 5 are the highest and seem to be systematically high. All labs fractured V-notched specimens which had been machined by JFCC. There seems to be little difference among the apparatuses for room temperature measurement.

The $K_{c,n}$ at room temperature shows no crosshead speed dependence in the range from 0.005 to 0.5mm/min (Fig.4-3a). Therefore environmentally-assisted crack growth did not effect the measured toughness of SSN-H.

The average $K_{c,n}$ and σ_{n-1} at 1200°C measured by each lab show larger scatters than do those at room temperature(Figs.4-4a and 4b). The crosshead speed dependence of the $K_{c,n}$ is not as clear as for that at room temperature(Fig.4-3b).

High temperature $K_{c,n}$ values measured in air are larger than those in N_2. Lab 1 measured $K_{c,n}$ both in air and in N_2. The results show that $K_{c,n}$ in air is higher than that in N_2(Table 4-1). As shown in Fig.4-5, an oxide layer was observed around a V-notch after 1200°C for 15 min exposure.

Temperature dependence of $K_{c,n}$ is shown in Figs.4-6a and 6b. The $K_{c,n}$ at 1200°C in air from each lab is higher than that at room temperature, while $K_{c,n}$ at 1200°C in N_2 is almost the same as that at room temperature. The data from Lab 1 show no significant difference between the values at room temperature in air and at 1200°C in N_2. Machining damage is considered to be healed by the formation of oxide layer, resulting in the increasing $K_{c,n}$. Therefore, environmental atmosphere affects $K_{c,n}$ at 1200°C when measured by the SEVNB method.

4.2 Single Edge Precracked Beam Method

Compared with the EC-141 silicon nitride(NGK Spark Plug Co.Ltd.) which was used for VAMAS round robin on room temperature fracture toughness in 1989[3,4], the SSN-H specimens were more difficult to precrack. In some cases, the precracks were outside the limit in the specification. Fig.4-7a shows a crack front on the fracture surface which is valid for calculation of toughness, while an angled crack front in Fig.4-7b is invalid. Lab 6 reported that precracking the SEPB specimens was a big problem and that the success rate was about 30%. In order to introduce a valid precrack, precision jigs and careful operations are necessary.

Lab 5, using closed loop, electromechanical machine (Instron 8562), suggested slow crack growth during heating in air or during the loading ramp. Lab 5 used some preload on the specimens when the furnace was heating up to 1200°C, and observed the area (Δa) where slow crack growth seemed to have occurred(Fig.4-7c). The other labs did not report about the point. The preload is considered to cause some slow crack growth during loading, or to open the crack up and allow more oxidation or creep to change the creep tip. The precrack length should include the length due to the slow crack growth.

Lab 1 did not observed the slow crack growth area. However, the load vs. displacement curves, measured at a crosshead speed of 0.005mm/min and at both room temperature and 1200°C, suggest that the slow crack growth occurred for an instant just before the breakage(Fig.4-8). Micrographs show a fracture surface of SSN-H after the SEPB test at 1200°C(Fig.4-9). Area 1, located near a precrack front, formed instantly after a crack propagated from the precrack front. Area 2 is located in the precracked region far from the front. Intergranular fracture seems to have occurred more frequently at area 1 than at area 2, suggesting that same slow crack growth occurred after crack propagation from the precrack front.

Considering the definition of K_{IC}, the values from Lab 5 were calculated using the crack length at the point of breakage(after some slow crack growth had occurred).

The results obtained from the SEPB method are shown in Table 4-2 and Figs.4-10a and 10b. The standard deviations for the data measured in N_2 are small.(<0.6MPa• $m^{0.5}$), while those in air are large.

As shown in Fig.4-11 and the data from Lab 1, the values measured at 1200°C in air are much higher than those in N_2. The difference is 3MPa• $m^{0.5}$ and higher. The increase in the K_{IC} in air is considered to be due to the healing of the precrack caused by the formation of an oxide layer. The crack tip would become blunt and some adhesion would occur between the precracked surfaces. The obtained values are no longer the true fracture toughness. The SEPB method is therefore not appropriate to measure the high temperature fracture toughness in air, if the material suffers oxidation.

No crosshead speed dependence is shown from the data of Lab 1 at room temperature and from all the data at 1200°C. No effect of span configuration was apparent.

4.3 Chevron Notched Beam Method

The fracture toughness values measured by the CNB method differed with laboratory in sereral earlier studies. This scatter of the values was considered to be due to the difference in machining conditions for the specimens. In this round robin, Lab 1 used a V-notch with a tip radius of about 30μm, machined by a highly controlled diamond wheel. The other labs used straight-through notches with widths of 300, 150 and 200μm for Labs 4, 5 and 6, respectively, and their notch tip profiles were different among the various machining shops.

Labs 1 and 5 observed slow crack growth just before the breakage of specimens (Fig.4-12). Lab.6 did not observe the slow crack growth, probably due to the lack of stiffness in the test machine which has a long train for use at 1200°C. The participants in this round robin had different fixtures, silicon carbide, alumina and carbon, as shown in Table 3-1. The details of the pushrods and other parts of their equipment are not clear. The test equipment stiffness must relate to the observation of slow crack growth.

The micrographs in areas 1 and 2 [A-2] were taken by 5 labs. As shown in Fig.4-13, fracture surface observation suggests that intergranular fracture is dominant both at the apex of triangular ligament and in area 1, and that intragranular fracture occurred more frequently in area 2 than in area 1. Therefore, slow crack growth seems to have occurred in area 1 and subsequently the crack propagated unstably through area 2.

Results consistent with this were observed from the micrographs from Lab 4. However, the micrographs from Labs 5, 6 and 8 did not suggest the slow crack growth clearly. The average K_{IC} values and the standard deviations are shown in Table 4-3 and Figs.4-14a, 14b and 15. The values show a large scatter among laboratories. The standard deviations at a crosshead speed of 0.5mm/min from Lab 2 is extremely high, compared with the others. Lab 2 reports that they could not find out what caused such a high value.

No crosshead dependence of the K_{IC} was shown either in air or in N_2(Fig.4-15). Within the scatter, no effect of environment was apparent.

Lab 1, applying a V-notch for the CNB method(i.e.CVNB method), measured the K_{IC} under several conditions. These results show the similar values from 5.2 to 5.7 $MPa \cdot m^{0.5}$ and small standard deviations from 0.1 to 0.7$MPa \cdot m^{0.5}$, irrespective of crosshead speed, temperature(room temperature or 1200°C), or atmosphere(air or N_2). Among the methods adopted in this round robin, the CVNB seems to be the only method which can measure K_{IC} without the influence of measuring conditions. The scatter is also small, probably due to the combination of a chevron-shaped ligament and highly-controlled V-notch machining.

Himsolt et al.[15] reported the improved CNB method. The chevron tip was effectively outside the specimen, i.e. there was a flat tip to the chevron. And a Knoop indent was indented in line with the chevron to act as a precrack. The modified chevron specimens gave the fracture toughness values with a small scatter. This method is considered to make the scatter among labs smaller and is expected to be one of the improved CNB methods.

4.4 Comparison among Three Methods

In order to compare the fracture toughness values by the SEVNB, SEPB and CNB methods, the average values and the standard deviations are calculated using the data from all the labs(Table 4-4 and Figs. 4-16a and 16b). The fracture toughness values by the three methods are schematically compared in Tables 4-5 and 4-6.

There does not appear to be a dependence of fracture toughness on crosshead speed in all the methods.

Oxidation of silicon nitride, caused by heating at 1200°C in air, affects the values variously. The values by the CNB method are free from oxidation, while those by the SEVNB method increase by 1.1 and 1.5MPa•$m^{0.5}$ for crosshead speeds of 0.005 and 0.5mm/min, respectively. The SEPB values increase markedly by 3.3 and 3.4MPa•$m^{0.5}$ for 0.005 and 0.5mm/min, respectively. The SEPB results in air are the highest in this round robin. It is probably due to the precrack healing in the SEPB specimens.

The SEVNB and SEPB values measured in N_2 are almost the same, and a little lower than the CNB value. As for the values measured in air, the SEVNB value is almost the same as or slightly higher than the CNB value, but the both are much lower than the SEPB value. The CVNB values are similar to the average values measured in N_2 by the SEVNB, SEPB and CNB methods.

5. CONCLUSIONS AND RECOMMENDATIONS

Eight labs in four countries participated in the high temperature fracture toughness round robin. The material used was silicon nitride, SSN-H. The values of fracture toughnesses, $K_{c,n}$(obtained by the SEVNB method) and K_{IC} (by the SEPB and CNB methods), were measured as a function of crosshead speed, ambient temperature or atmosphere. The analyzed results are as follows;

1. The SSN-H does not show crosshead speed dependence, suggesting that environmentally-assisted crack growth is inactive for the material.
2. Oxidation of SSN-H by heating at 1200°C in air increases the apparent fracture toughness values by the SEVNB and SEPB methods.
3. The SEVNB values in N_2 are almost the same, irrespective of ambient temperature or crosshead speed, while those at 1200°C in air are a little higher than those at room temperature. This is probably due to the healing of machining damage.
4. The SEPB values at 1200°C in N_2 are slightly lower than those at room temperature, while those at 1200°C in air are much higher than those both at 1200°C in N_2 and at room temperature. This is probably due to the healing, resulting in partial adhesion on precrack surfaces and precrack tip blunting.
5. It requires accurate jigs and some experience to precrack the SEPB specimens properly. One lab observed slow crack growth during the 1200°C SEPB test. The crack length for calculating K_{IC} should include the length caused by the stable crack propagation.
6. The CNB values vary according to laboratories, due to the difference in machining conditions for introducing chevron notches. However, the CVNB values hardly vary, irrespective of crosshead speed, ambient temperature or atmosphere. This is probably due to the combination of V-notch machining and configuration of a ligament.
7. As for high temperature fracture toughness in N_2, the SEVNB and SEPB values

are almost the same, and are lower than the CNB value. The SEPB value at 1200°C in air is much higher than the SEVNB and CNB values. The CNB method can be used in inert and air atmosphere, but the SEPB and SEVNB are not suitable in air.

Although SSN-H does not show a crosshead speed dependence, other materials might do so, and lead to errors due to subcritical growth. At a speed of 0.5mm/min, the test is over in a few seconds, which should give more reliable results if subcritical crack growth corrections are not made because evidence is difficult to obtain. A crosshead speed of 0.005mm/min is not considered to be practical, because the measurement at that speed takes much longer. The speed of 0.5mm/min is recommended.

Ambient atmosphere appears to be a very important factor for fracture toughness measurement of non-oxides at high temperatures. Silicon nitride ceramics suffers the oxidation to varying degree with the heating conditions in air, which affects the fracture toughness.

The high temperature fracture toughness in non-oxidizing atmosphere can be measured by the SEVNB and SEPB methods with a small scatter, although the absolute values differ from each other. The CVNB method shows a small scatter and is recommended for any measurement conditions. The CVNB values are independent of ambient temperature or atmosphere. However, the method requires precision machining apparatus and some technique for introducing V-notches into specimens.

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APPENDICES

A.1 Lists of Participants.

A.2 Instructions for the VAMAS High Temperature Fracture Toughness Round Robin.

A.3 Data Sheets from Laboratories.

(1) SEVNB, (2) SEPB, (3) CNB.

Table 1-1 List of Participating Laboratory.
(High Temperature Fracture Toughness Round Robin)

COUNTRIES	LABORATORY
Germany	BAM (Bundesanstalt für Materialforschung und -prüfung) Universität Karlsruhe
Japan	GIRIN (Government Industrial Research Institute, NAGOYA) JFCC (Japan Fine Ceramics Center) NIRIM (National Institute for Research in Inorganic Materials) NRLM (National Research Laboratory of Metrology)
U.K.	NPL (National Physical Laboratory)
U.S.A.	NASA (National Aeronautics and Space Administration)

Table 2-1 Properties of Silicon Nitride, SSN-H.

Manufacturer	Kyocera Co.
Main component	Silicon Nitride
Additive	Yb_2O_3, Al_2O_3
Flexural Strength	
RT	650 MPa
1372°C (2500F)	500 MPa
Fracture Toughness* (IF Method)	4.9 $MPa\cdot m^{0.5}$
Vickers Hardness	1,620 (10kgf)
Weight Gain after Oxidation(1371°C·24h)	0.2 mg/cm^2

* Measured by JFCC.

Table 2-2 Numbers of Specimens for Fracture Toughness Measurement.

		Crosshead Speed	
		0.5 mm/min	0.005 mm/min
SEVNB	R.T.	5 "	5 "
	1200°C	5 "	5 "
SEPB	1200°C	5 ""	5 ""
CNB	1200°C	5	5
spare pieces		5	

" V-notched , "" Chamfered

* JFCC V-notched all the specimens for the SEVNB method.

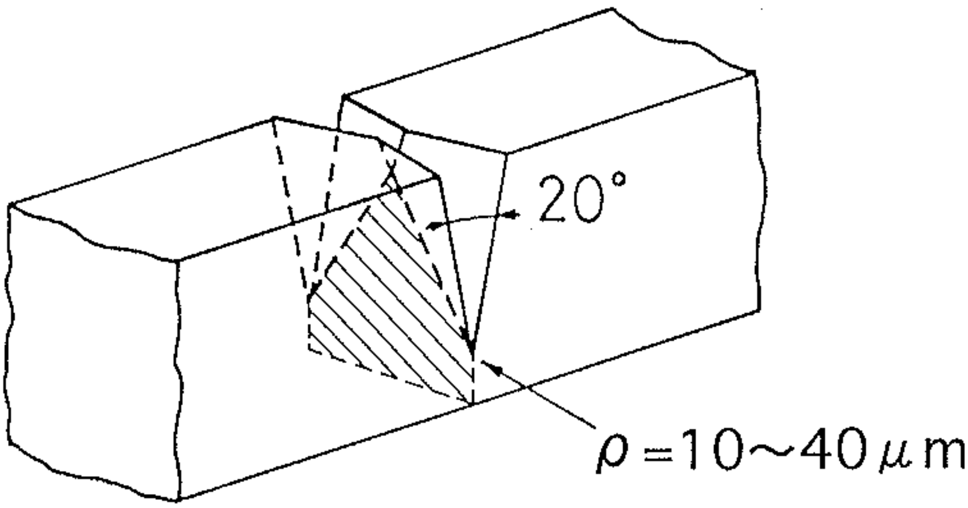


Fig. 3-1 Schematic Illustration of Chevron V-Notched Specimen.

Table 3-1 Measurement Conditions for Fracture Toughness.

Labo No.	SEVNB Room Temperature 3-p or 4-p Span(mm)	SEVNB Room Temperature Jig	SEVNB 1200° C 3-p or 4-p Span(mm)	SEVNB 1200° C Atmos-phere	SEVNB 1200° C Jig	SEPB 1200° C 3-p or 4-p Span(mm)	SEPB 1200° C Atmos-phere	SEPB 1200° C Jig	CNB 1200° C 3-p or 4-p Span(mm)	CNB 1200° C Atmos-phere	CNB 1200° C Jig	CNB 1200° C Notch
1	3-p 30mm	Steel, SiC pin	3-p 30mm	N_2	Carbon	3-p 30mm	N_2	Carbon	4-p 30&10mm	N_2	Carbon	V-notch
			3-p 30mm	Air	SiC	3-p 30mm	Air	SiC	4-p 30&10mm	Air	SiC	V-notch
									4-p 40&20mm	N_2	Carbon	V-notch
2	3-p 30mm	SiC	3-p 30mm	N_2	SiC	3-p 30mm	N_2	SiC	4-p 30&10mm	N_2	SiC	Straight
3	3-p 30mm	Carbon, SiC pin	3-p 30mm	N_2	Carbon, SiC pin	3-p 30mm	N_2	Carbon, SiC pin				
4	3-p 30mm	Stainless steel	3-p 30mm	Air	SiC				4-p 30&10mm	Air	SiC	Straight (w:0.3mm)
5	3-p 30mm	Steel, SiC pin	3-p 30mm	Air	SiC	3-p 30mm	Air	SiC	4-p 30&10mm	Air	SiC	Straight
6	3-p 30mm	Alumina	3-p 30mm	Air	Alumina	3-p 30mm	Air	Alumina	4-p 40&20mm	Air	Alumina	Straight (w:0.2mm)
7	3-p 40mm	Hardmet-al	3-p 40mm	Air	SiC	3-p 40mm	Air	SiC	4-p 40&20mm	Air	SiC	Straight
8	3-p 40mm	Steel	4-p 40&20mm	Air	SiC	4-p 40&20mm	Air	SiC	4-p 40&20mm	Air	SiC	Straight

3-p: 3-point flexure 4-p: 4-point flexure

w:width

Table 4-1 Results of Fracture Toughness by the SEVNB Method.

SEVNB Kc,n ($MPa \cdot m^{0.5}$) 3-Point Flexure: Span 30mm in Air or in N_2, Span 40mm in Air
4-Point Flexure: Spans 40 & 20mm in Air

Temperature		Room Temperature				1,200°C			
Crosshead Speed (mm/min)		0.005	0.02	0.05	0.5	0.005	0.02	0.05	0.5
1	Avr. ± σ_{n-1}	5.2±0.5		5.6±0.5	5.1±0.5	4.9±0.3		4.9±0.2	5.0±0.4
						6.2±0.5		5.7±0.6	6.3±0.7
2	Avr. ± σ_{n-1}	4.6±0.3			4.5±0.3	5.9±0.8			5.3±1.0
3	Avr. ± σ_{n-1}	4.9±0.2			4.9±0.2	4.3±0.2			4.4±0.1
4	Avr. ± σ_{n-1}	4.8±0.4			4.7±0.2	6.7±0.3			5.7±0.4
5	Avr. ± σ_{n-1}	5.6±0.3			5.7±0.4	6.9±0.4			6.8±0.3
6	Avr. ± σ_{n-1}		4.6±0.3		4.6±0.1		6.2±0.1		5.3±0.7
7	Avr. ± σ_{n-1}	5.1±0.1			5.2±0.4	6.1±0.4			5.5±1.0
8	Avr. ± σ_{n-1}	5.2±0.4			5.7±0.1	6.4±0.4			6.2±0.5

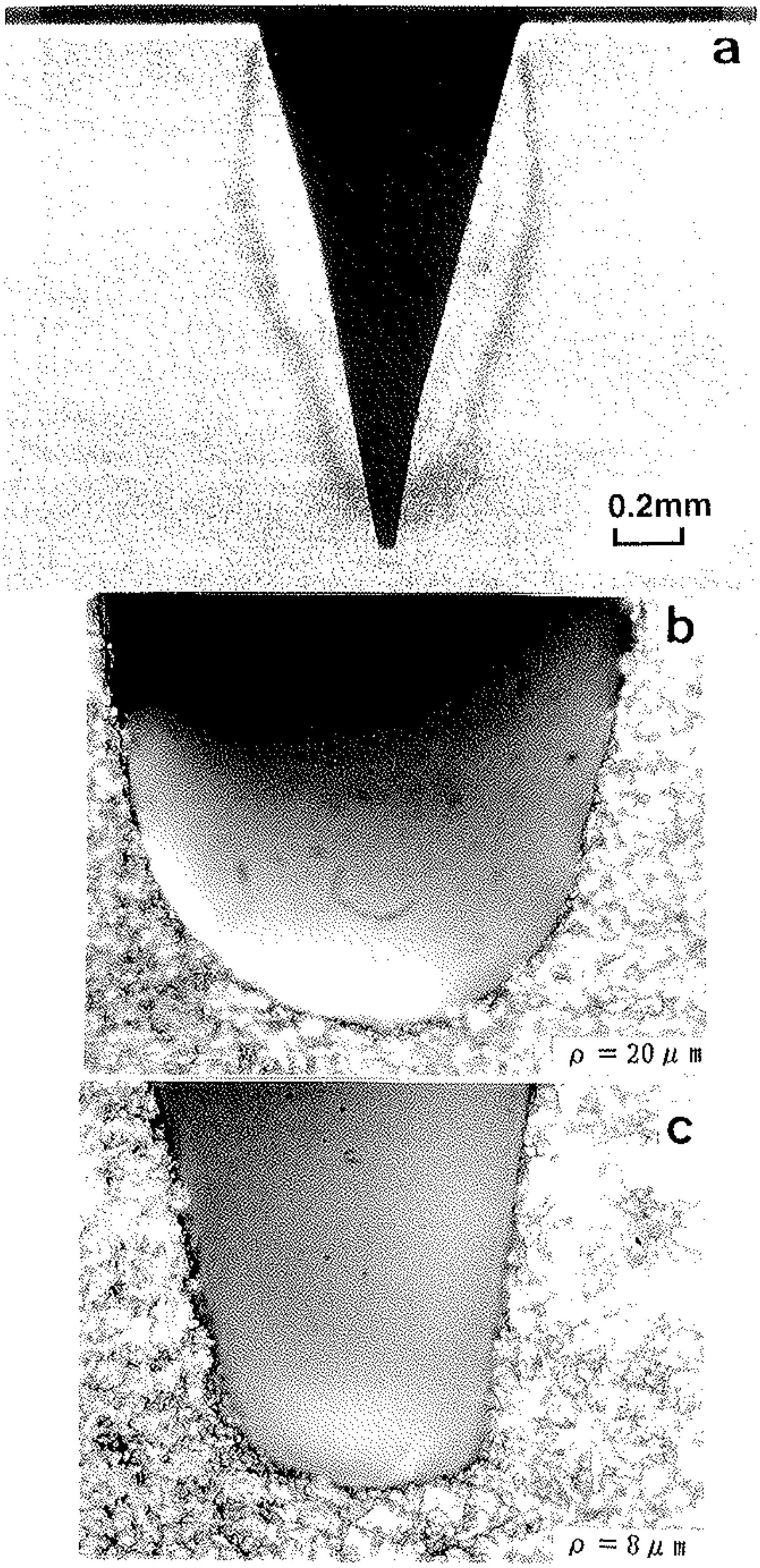


Fig. 4-1 Cross-section of V-Notch. ρ; Tip Radius

a) Overall image of a V-Notch (from Lab.5).

b) Micrograph of V-Notch Tip (from Lab.1). ρ=20μm

c) Micrograph of V-Notch Tip (from Lab.1). ρ=8μm

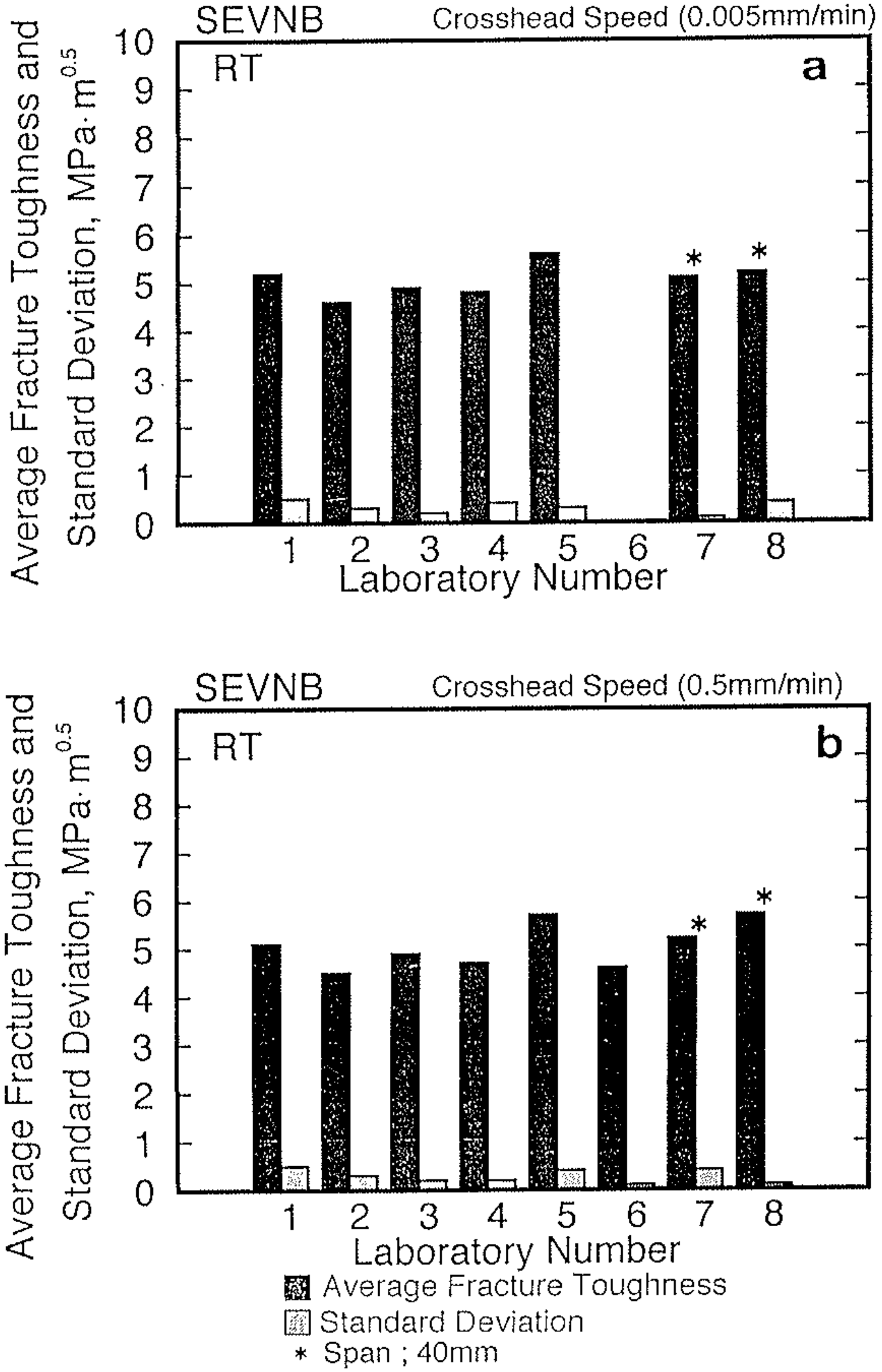


Fig. 4-2 Average Fracture Toughness and the Standard Deviation at Room Temperature(SEVNB).

a) Crosshead Speed: 0.005mm/min, except lab6:0.02mm/min.

b) Crosshead Speed: 0.5 mm/min.

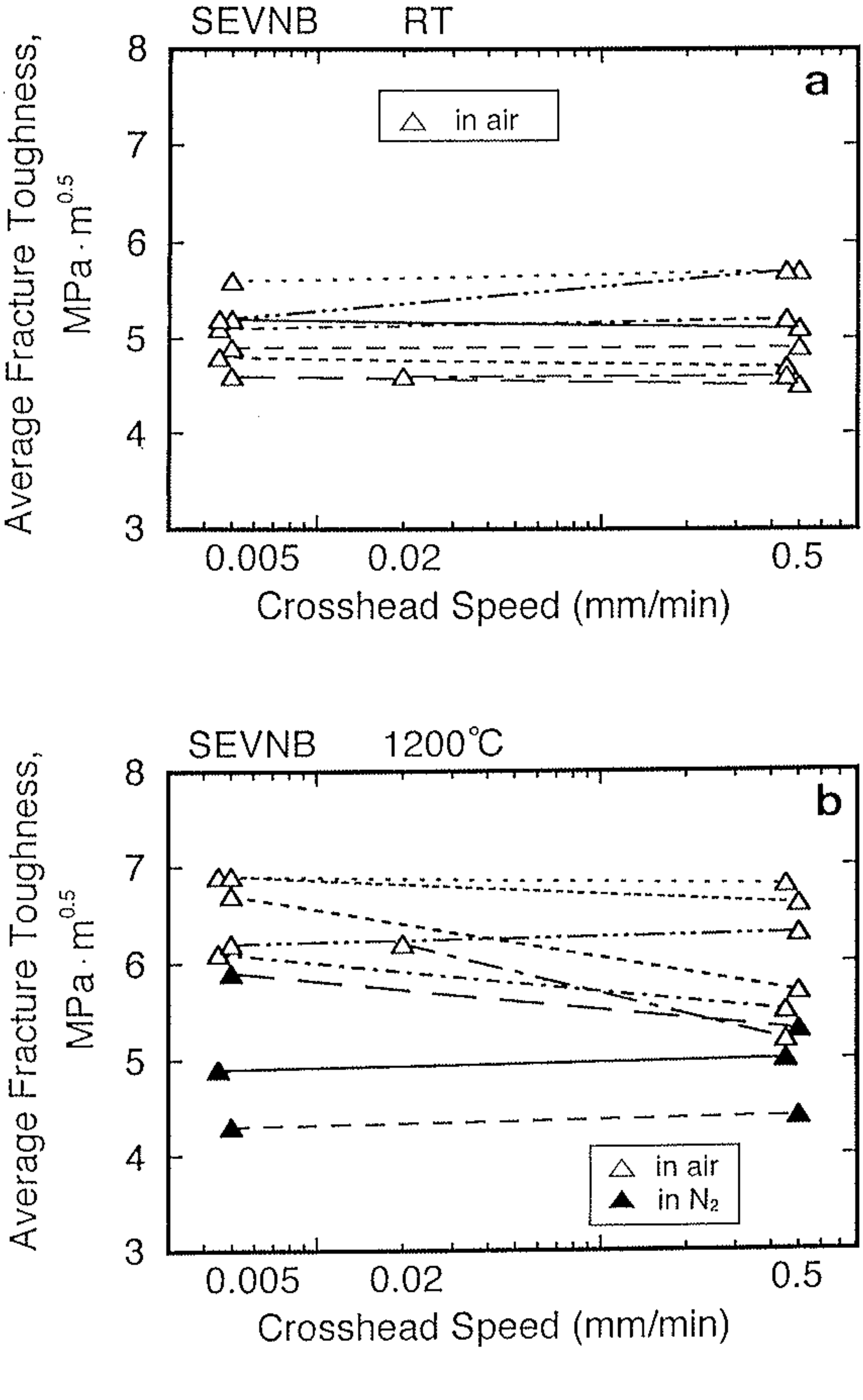
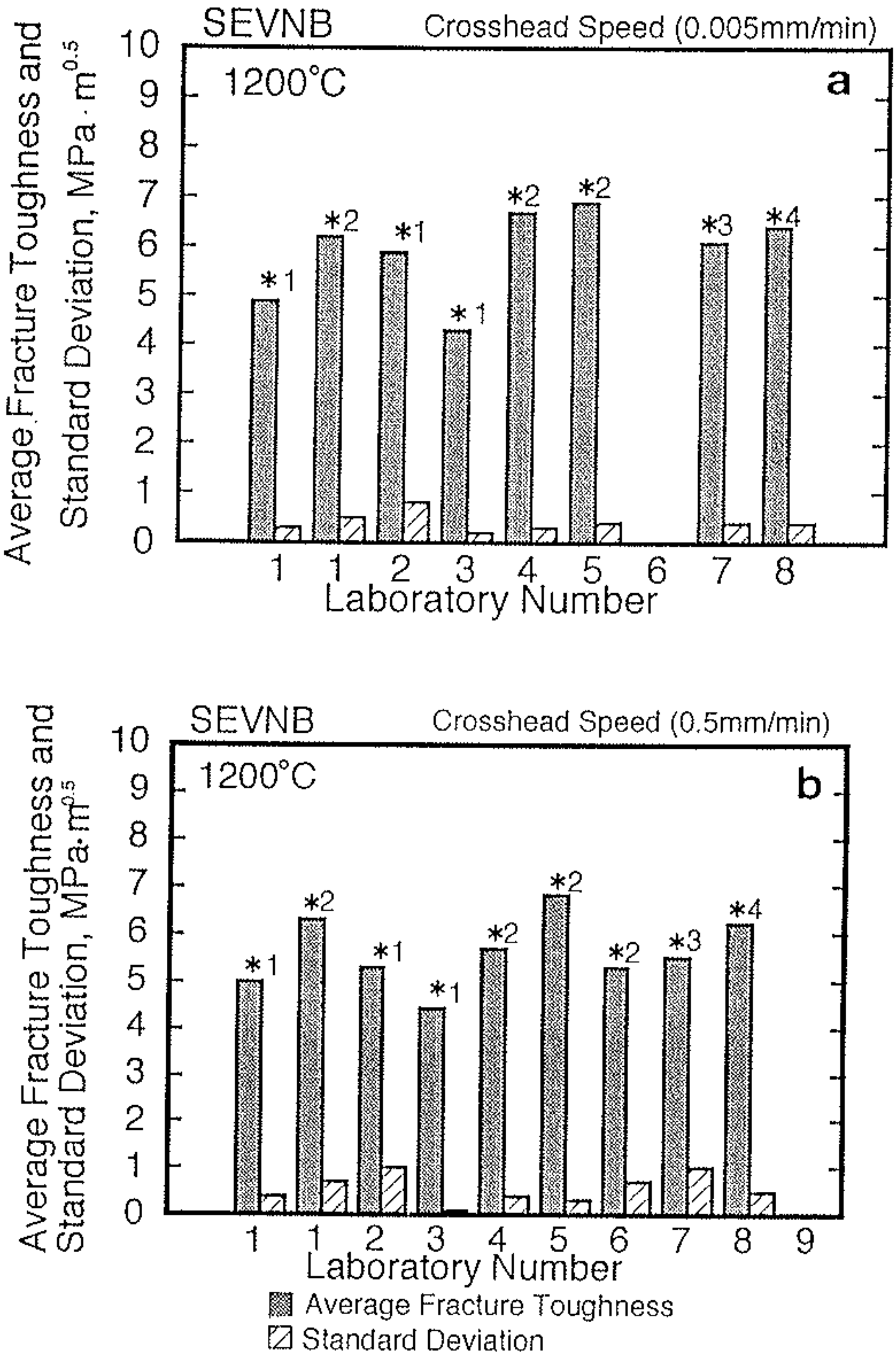


Fig. 4-3 Average Fracture Toughness vs. Crosshead Speed(SEVNB).

a) Room Temperature.

b) 1200°C.



*1 3-Point Flexure in N_2, Span 30mm

*2 3-Point Flexure in Air, Span 30mm

*3 3-Point Flexure in Air, Span 40mm

*4 4-Point Flexure in Air, Spans 40 and 20mm

Fig. 4-4 Average Fracture Toughness and the Standard Deviation at 1200°C(SEVNB).
a) Crosshead Speed: 0.005mm/min.
b) Crosshead Speed: 0.5 mm/min.

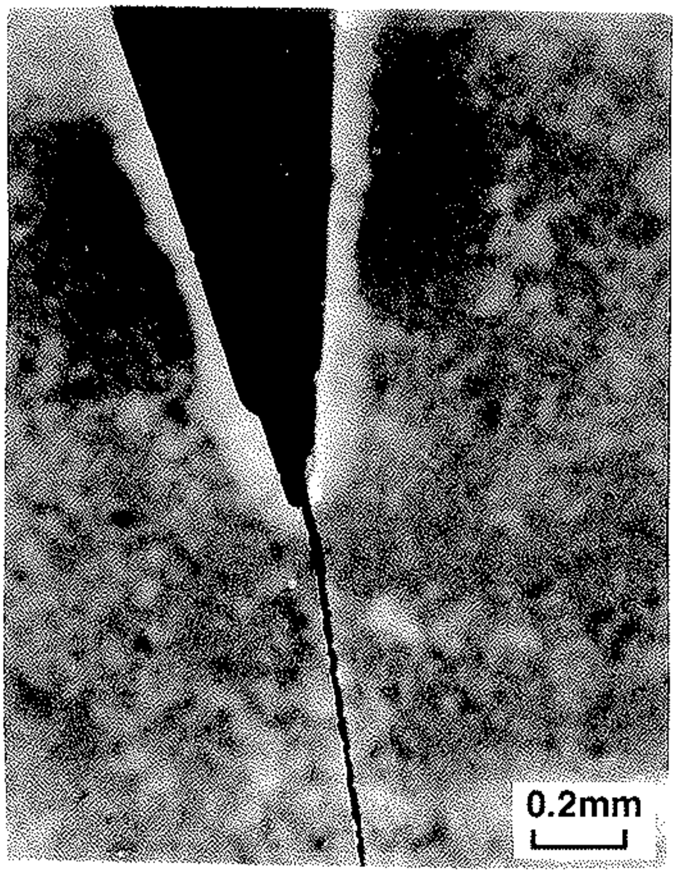
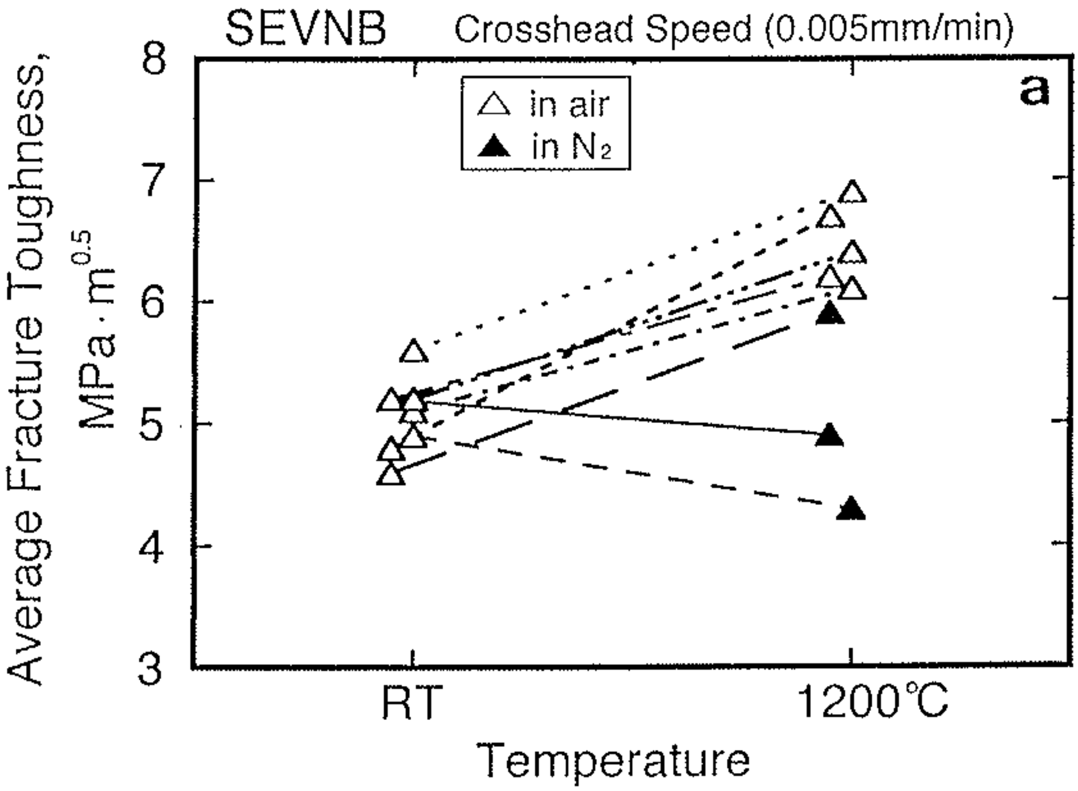


Fig. 4-5 V-Notch Profile Image after Fracture(from Lab.5).
Test Condition: 1200°C for 15min. Note the oxidation.



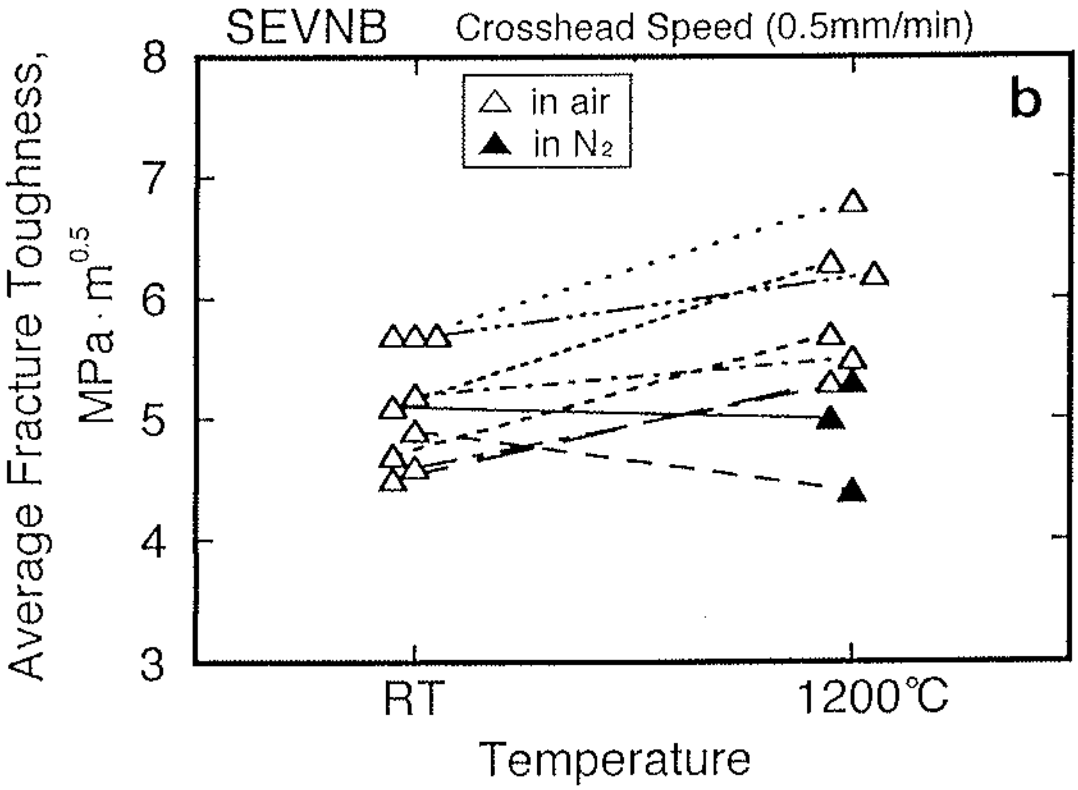


Fig. 4-6 Average Fracture Toughness vs. Temperature(SEVNB).
a) Crosshead Speed: 0.005mm/min.
b) Crosshead Speed: 0.5 mm/min.

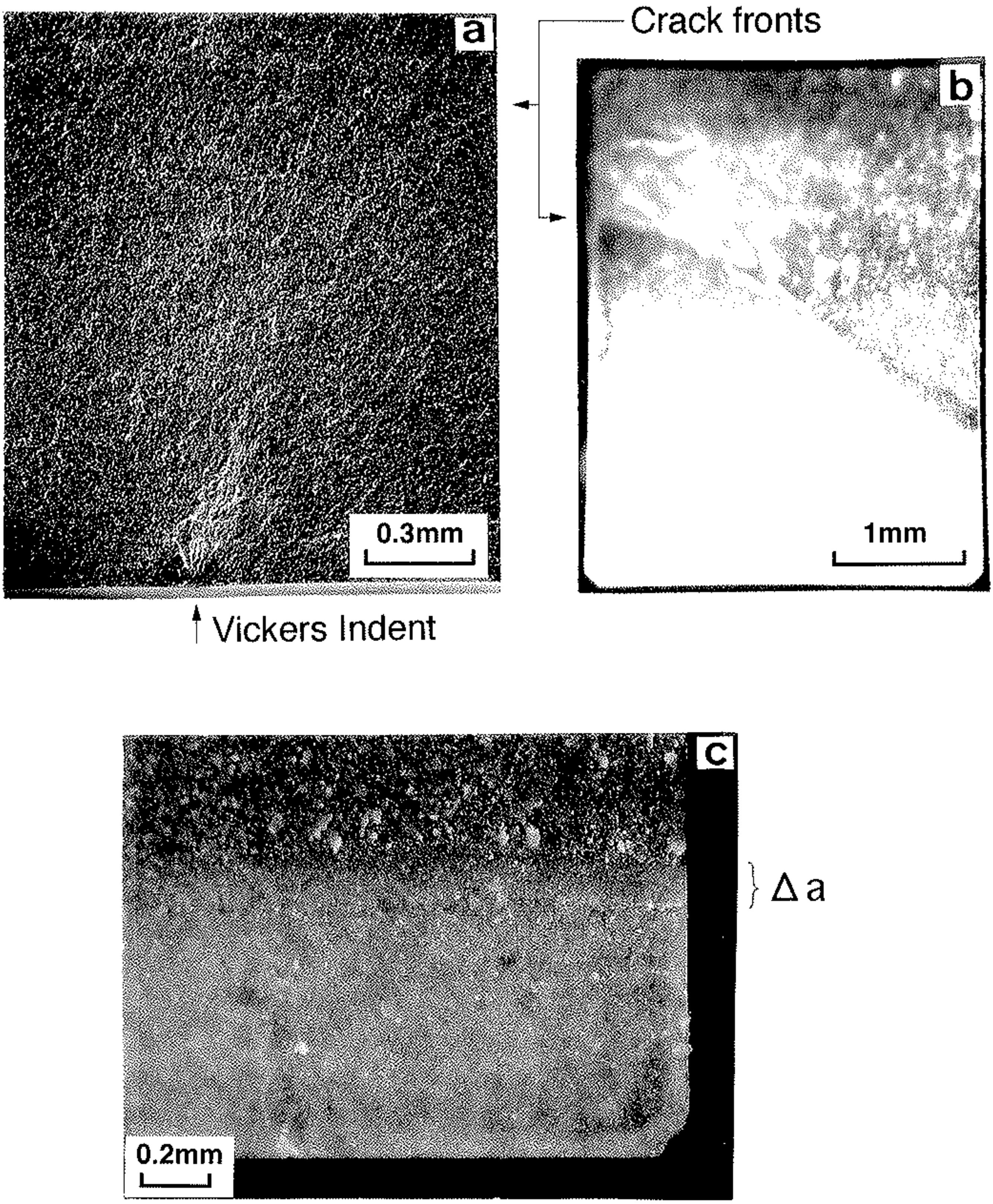


Fig. 4-7 Fracture Surfaces of SEPB Specimens.

a) after Fracture at 1200°C in N_2; Crosshead Speed of 0.005 mm/min.(from Lab.1).

b) after Fracture at 1200°C in air; Crosshead Speed of 0.5 mm/min.(from Lab.6). Crack front outside limits.

c) after Fracture at 1200°C in air; Crosshead Speed of 0.5mm/min.(from Lab.5). Slow crack growth zone(Δa).

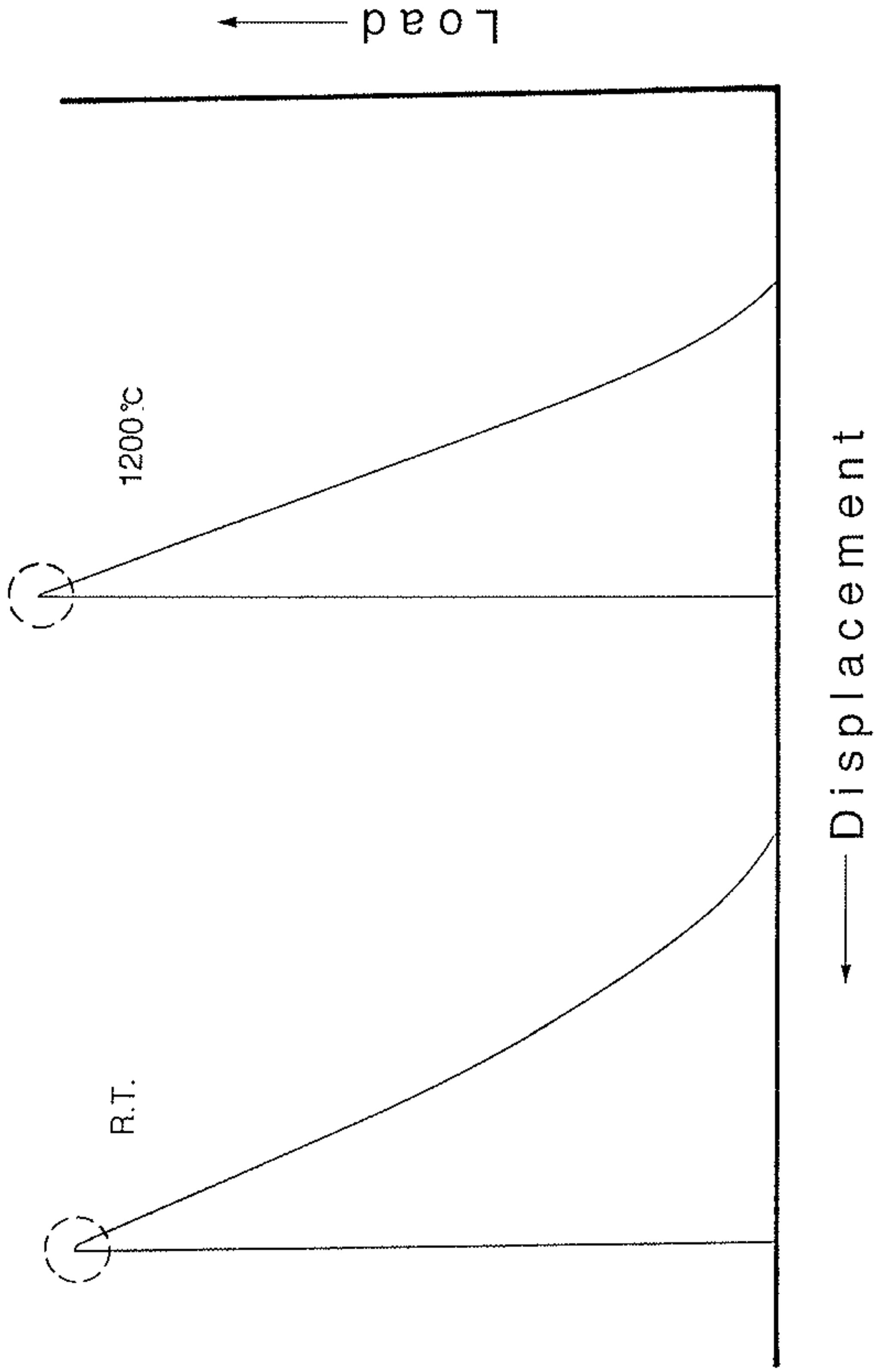


Fig. 4-8 Load vs. Dislocation Curves for the SEPB Measurement(from Lab.1).
Test Condition: Crosshead Speed of 0.005mm/min.

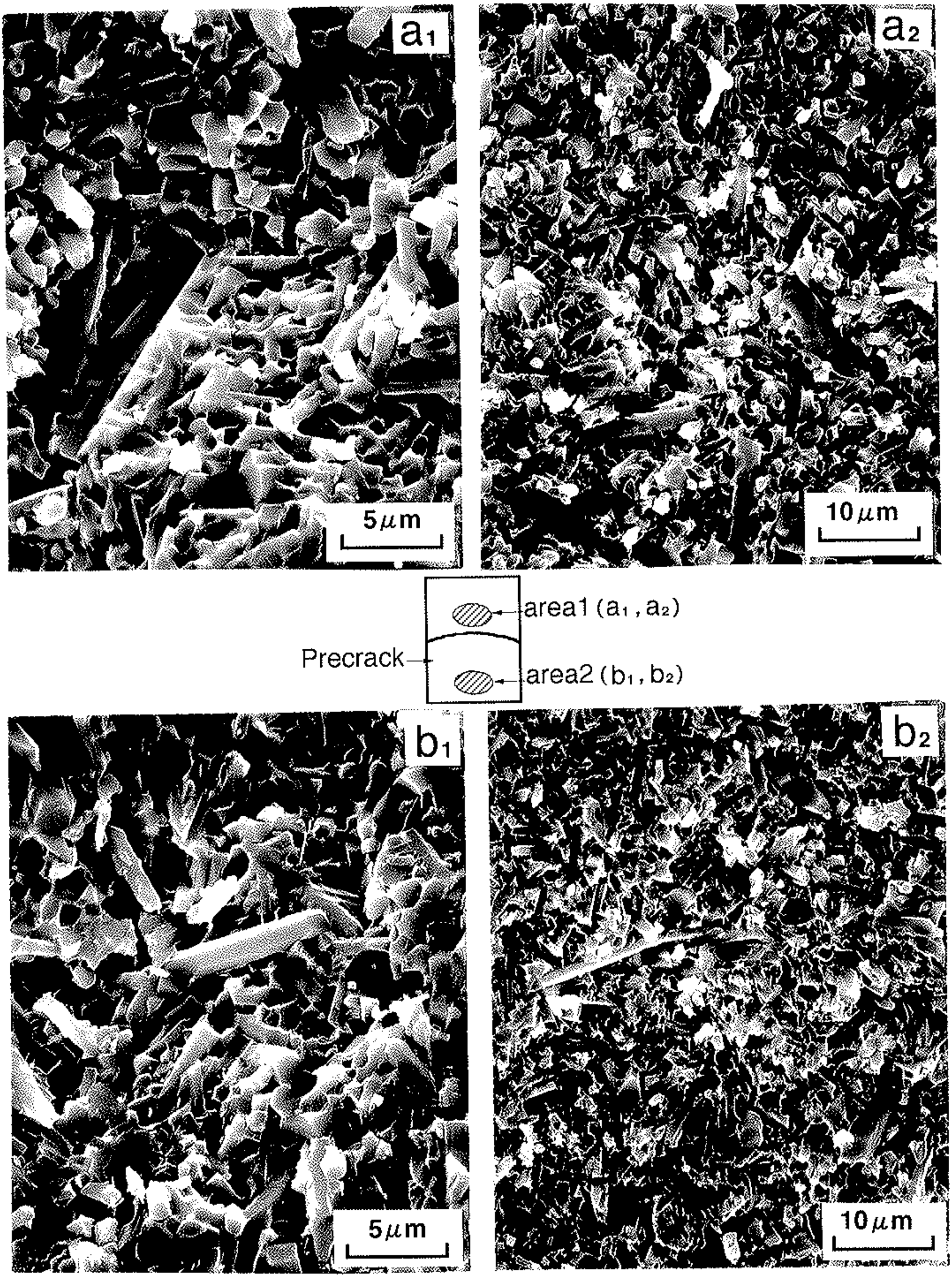


Fig. 4-9 Microphotographs of Fracture Surface of the SEPB Specimens(from Lab.1).
Test Condition: 1200°C in N_2, Crosshead Speed of 0.005mm/min.).
a1 and a2) area 1.
b1 and b2) area 2.

Table 4-2 Results of Fracture Toughness by the SEPB Method.

SEPB 3-P. Flexure:Span 30mm in Air or in N_2, Span 40mm in Air
K_{IC} (MPa·$m^{0.5}$) 4-P. Flexure: Spans 40 & 20mm in Air

Temperature		Room Temperature		1,200°C		
Crosshead Speed (mm/min)		0.005	0.5	0.005	0.02	0.5
1	Avr. ± σ_{n-1}	5.9±0.1	5.9±0.2	5.2±0.1		5.4±0.1
				8.6±1.1		8.6±1.1
2	Avr. ± σ_{n-1}			5.3±0.4		5.2±0.6
3	Avr. ± σ_{n-1}			4.7±0.3		4.8±0.2
5	Avr. ± σ_{n-1}			8.1±0.8		8.0±0.9
6	Avr. ± σ_{n-1}				8.6±1.0	8.6±0.5
7	Avr. ± σ_{n-1}			8.6±1.1		8.5±0.6
8	Avr. ± σ_{n-1}			8.3±1.0		9.0±1.2

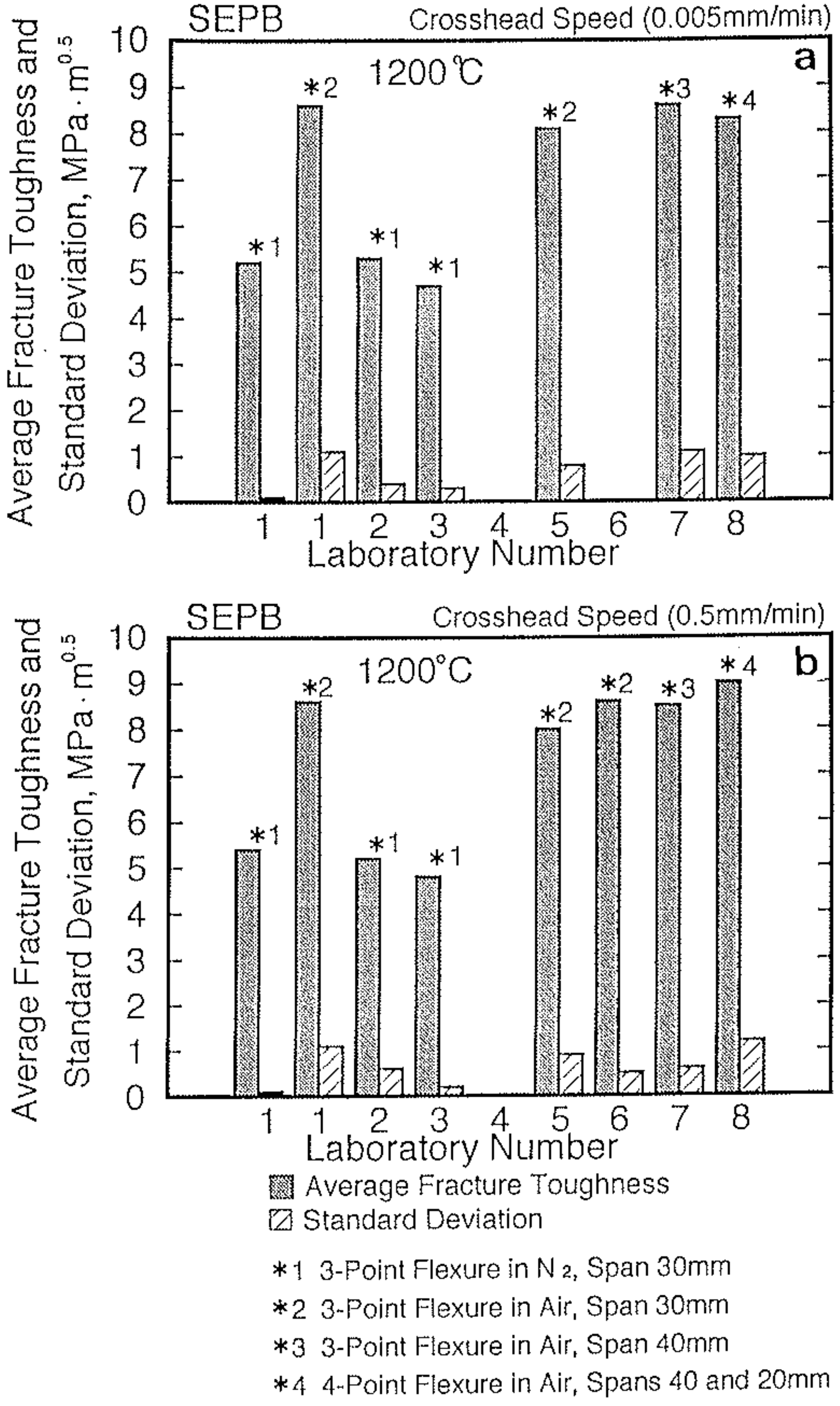


Fig. 4-10 Average Fracture Toughness and the Standard Deviation at 1200°C(SEPB).

a) Crosshead Speed: 0.005mm/min.

b) Crosshead Speed: 0.5 mm/min.

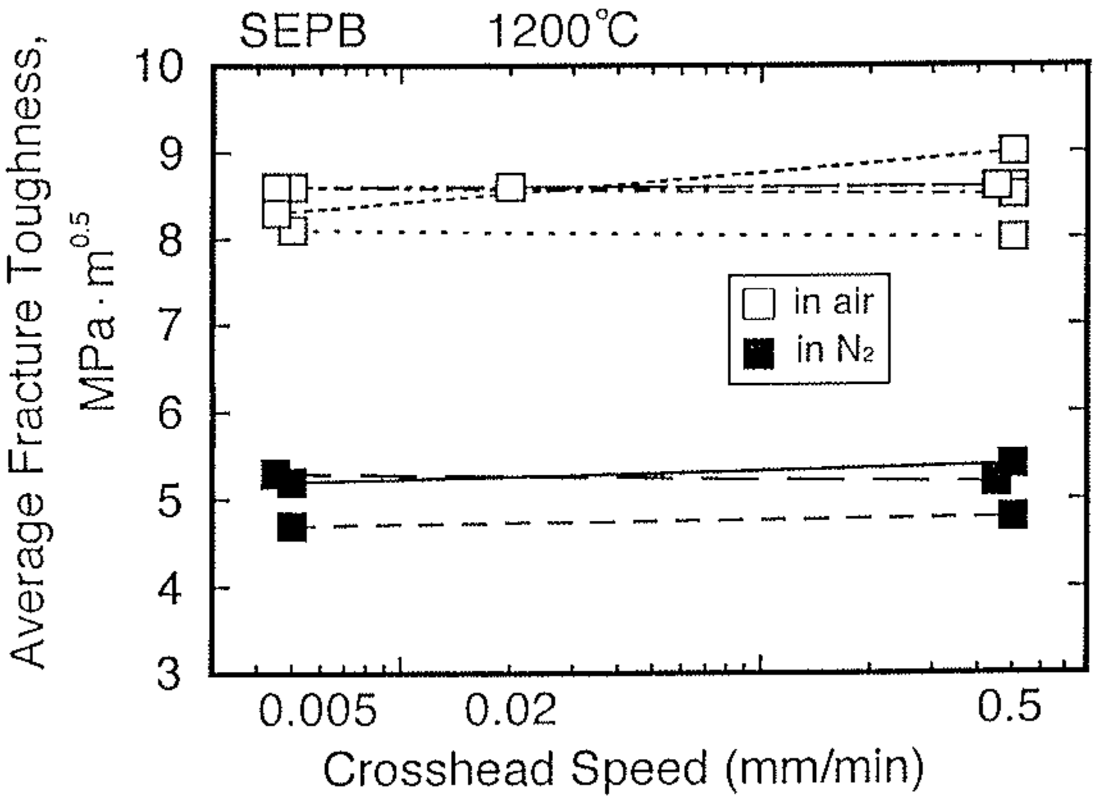


Fig. 4-11 Average Fracture Toughness vs. Crosshead Speed(SEPB).

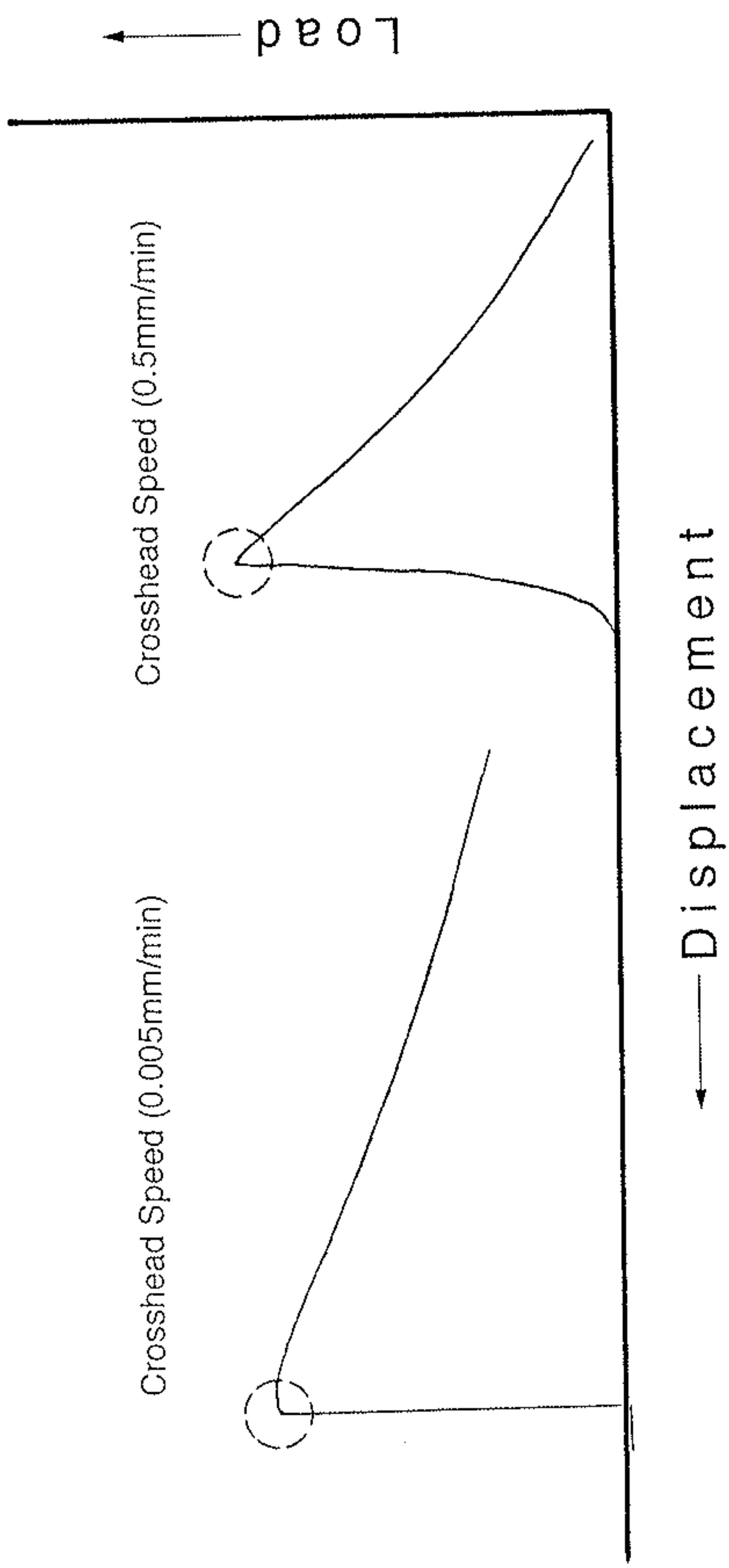


Fig. 4-12 Load vs. Dislocation Curves for the CNB Measurement(from Lab.1).
Test Condition: 1200°C in N_2.

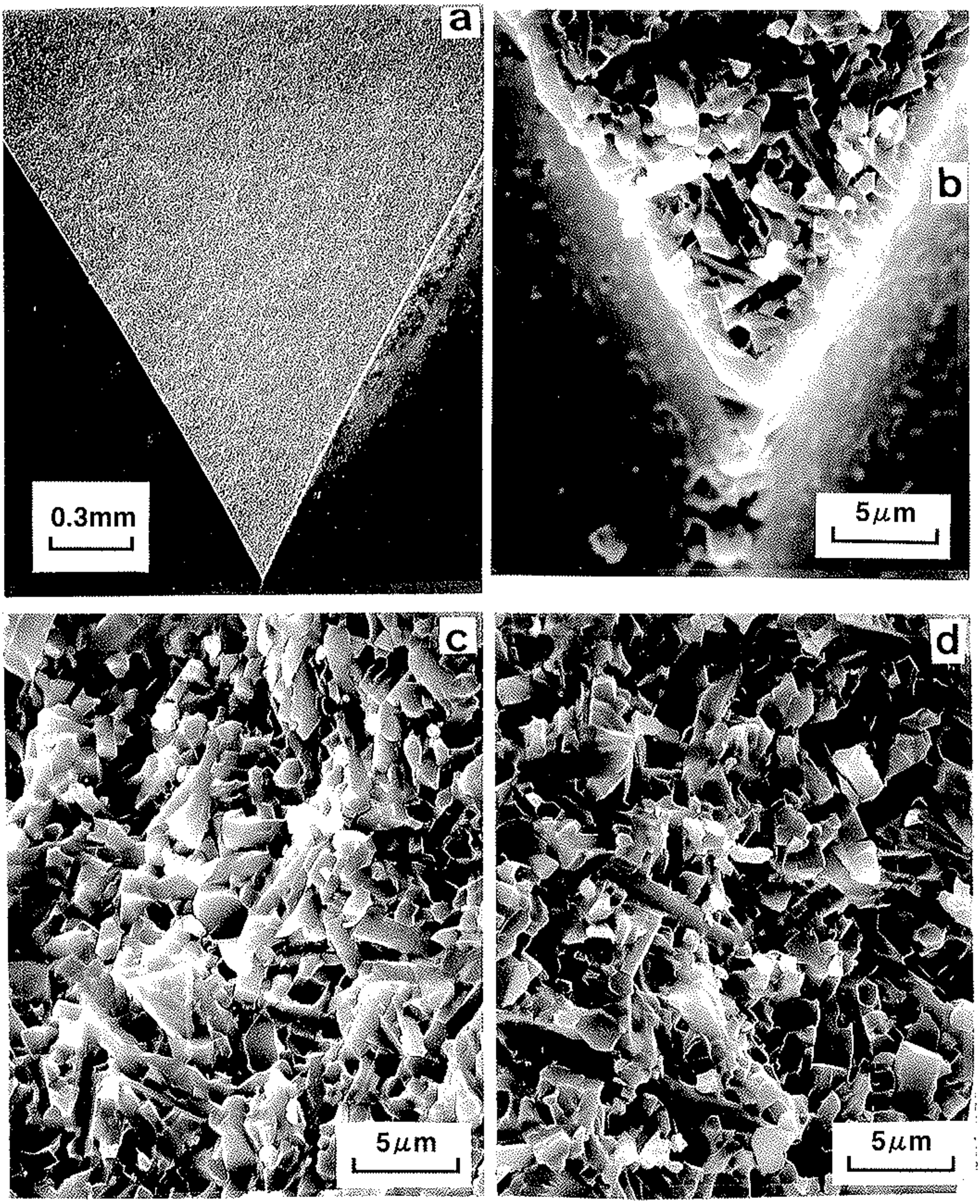


Fig. 4-13 Microphotographs of Fracture Surface of the CNB Specimens (from Lab.1).
Test Condition: 1200°C in N_2, Crosshead Speed of 0.005mm/min.).

a) Overall image of a ligament.

b) Apex of a ligament.

c) area 1.

d) area 2.

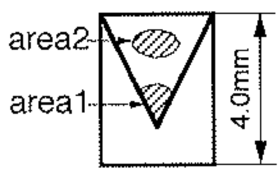


Table 4-3 Results of Fracture Toughness by the CNB Method.

CNB K_{Ic} ($MPa \cdot m^{0.5}$)		4-Point Flexure : Spans 30 & 10 mm in Air or in N_2; Spans 40 & 20 mm in Air or in N_2						
Temperature		Room Temperature			1,200°C			
Crosshead Speed (mm/min)		0.005	0.05	0.5	0.005	0.02	0.05	0.5
1 *1	Avr. ± σ_{n-1}	5.3±0.3	5.6±0.7	5.4±0.5	5.2±0.1		5.4±0.2	5.3±0.3
				5.6±0.3				5.5±0.2
					5.4±0.2		5.4±0.1	5.7±0.2
2 *2	Avr. ± σ_{n-1}				6.7±0.6			6.6±2.1
4 *2	Avr. ± σ_{n-1}				5.6±0.2			6.7±0.4
5 *2	Avr. ± σ_{n-1}				5.6±0.6			5.9±0.3
6 *2	Avr. ± σ_{n-1}				6.4±0.7	5.7±0.7		6.8±1.0
7 *2	Avr. ± σ_{n-1}				4.5±0.4			4.7±0.3
8 *2	Avr. ± σ_{n-1}				5.5±0.4			5.8±0.3

*1: V-notched *2: Straight-notched

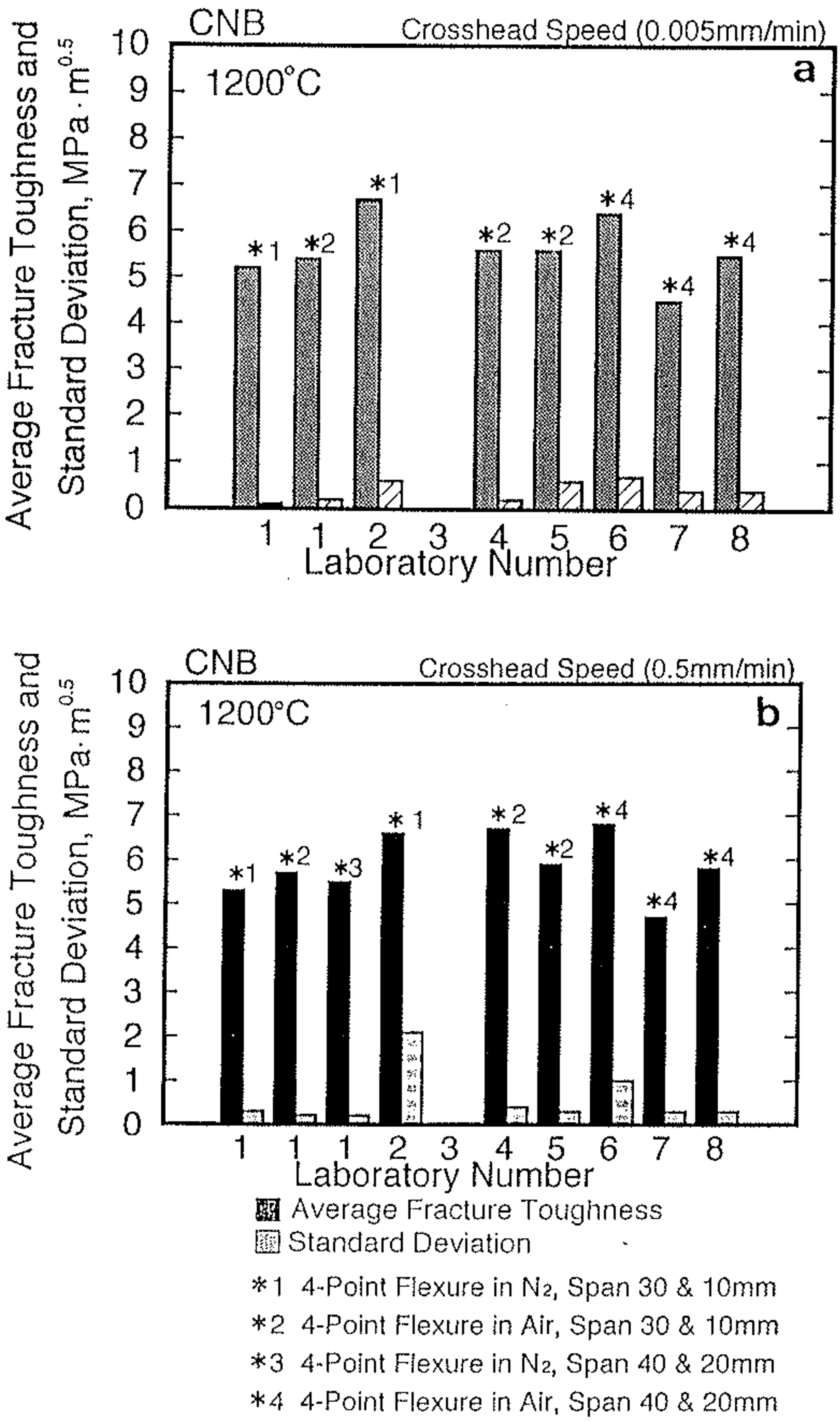


Fig. 4-14 Average Fracture Toughness and the Standard Deviation at 1200°C(CNB).
a) Crosshead Speed: 0.005mm/min.
b) Crosshead Speed: 0.5 mm/min.

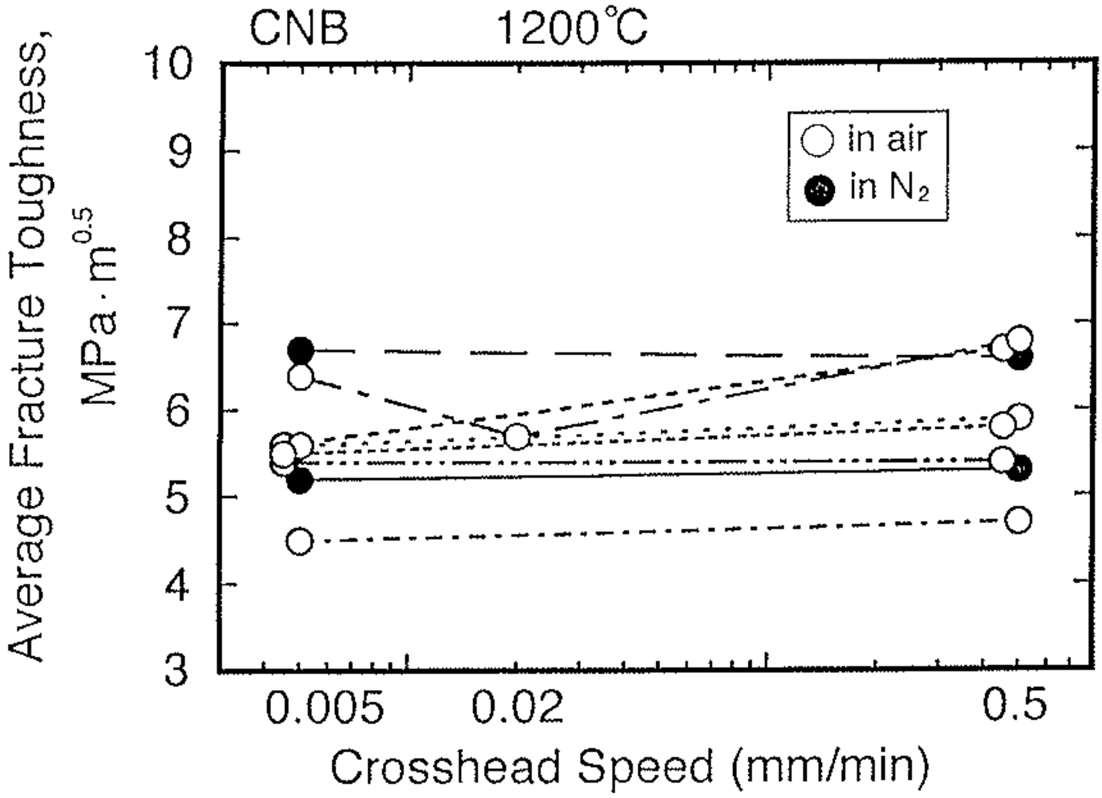


Fig. 4-15 Average Fracture Toughness vs. Crosshead Speed(CNB).

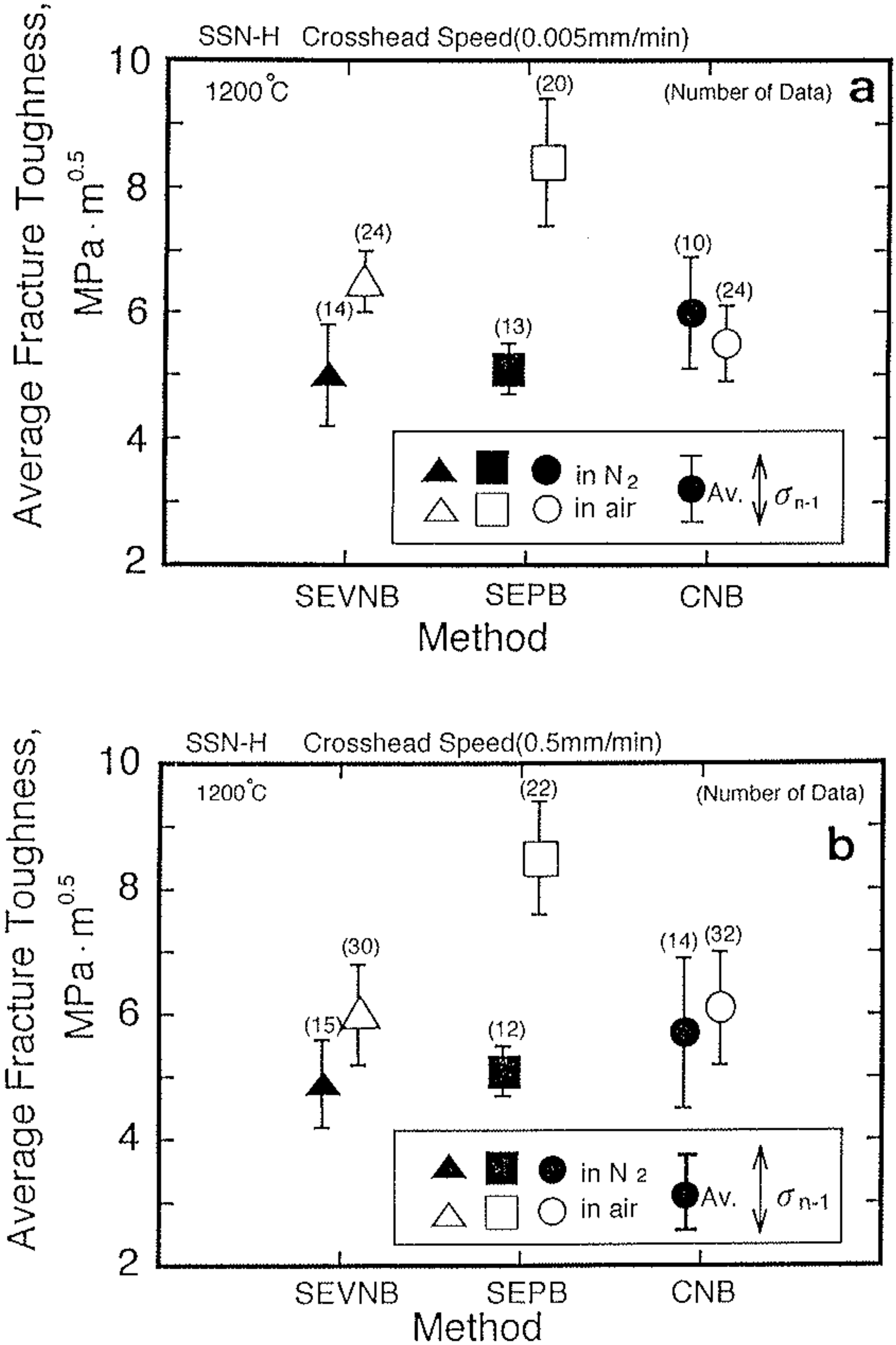


Fig. 4-16 Average Fracture Toughness at 1200°C(SEVNB, SEPB and CNB).

a) Crosshead Speed: 0.005mm/min.

b) Crosshead Speed: 0.5 mm/min.

Table 4-4 Summary of Results of Fracture Toughness Round Robin.

Method	SEVNB $K_{c,n}$						SEPB K_{IC}				CNB K_{IC}			
Temperature	RT		1200° C				1200° C				1200° C			
Crosshead speed	0.005	0.5	0.005		0.5		0.005		0.5		0.005		0.5	
Atmosphere	Air	Air	N_2	Air	N_2	Air	N_2	Air	N_2	Air	N_2	Air	N_2	Air
Range of avr. fracture toughness ($MPa \cdot m^{0.5}$)	4.6 ~ 5.6	4.5 ~ 5.7	4.3 ~ 5.9	6.1 ~ 6.9	4.4 ~ 5.3	5.3 ~ 6.8	4.7 ~ 5.3	8.1 ~ 8.6	4.8 ~ 5.4	8.0 ~ 9.0	5.2 ~ 6.7	4.5 ~ 6.4	5.3 ~ 6.6	4.7 ~ 6.8
Range of std. deviation ($MPa \cdot m^{0.5}$)	0.1 ~ 0.5	0.1 ~ 0.5	0.2 ~ 0.8	0.3 ~ 0.5	0.1 ~ 1.0	0.3 ~ 1.0	0.1 ~ 0.4	0.8 ~ 1.1	0.1 ~ 0.6	0.5 ~ 1.2	0.1 ~ 0.6	0.2 ~ 0.7	0.2 ~ 2.1	0.2 ~ 1.0
Overall avr. fracture toughness (Number of Data)	5.0 (33)	5.0 (40)	5.0 (14)	6.5 (24)	4.9 (15)	6.0 (30)	5.1 (13)	8.4 (20)	5.1 (12)	8.5 (22)	6.0 (10)	5.5 (24)	5.7 (14)	6.1 (32)
Overall avr. std. deviation ($MPa \cdot m^{0.5}$)	0.4	0.5	0.8	0.5	0.7	0.8	0.4	1.0	0.4	0.9	0.9	0.6	1.2	0.9
Number of labs.	7	8	3	5	3	6	3	4	3	5	2	6	2	6

Table 4-5 Schematic Relation of Fracture Toughnesses by the SEVNB, SEPB and CNB Methods.

Condition 1200°C	Condition CHS*1	Fracture Toughness
in N_2	0.005	SEVNB ≒ SEPB < CNB
	0.5	SEVNB ≒ SEPB < CNB
in air	0.005	CNB ≦ SEVNB << SEPB
	0.5	CNB ≒ SEVNB << SEPB

*1 Crosshead speed(mm/min)
A≒B : A is almost equal to B,
C≦D : D is equal to or higher than C,
E<<F: F is much higher than E.

Table 4-6 Schematic Relation of Fracture Toughnesses under different conditions by the SEVNB, SEPB and CNB Methods.

	Temperature	Crosshead Speed (mm/min)		Scatter (1200°C)		Precrack or Notch Machining
		RT	1200°C			
	RT 1200°C	0.005 0.5	0.005 0.5	N_2	air	
SEVNB	≒ N_2 < air	≒ air	≒ N_2 ≒ air	small	small	Skill is required
SEPB	≧ N_2 << air	≒ air	≒ N_2 ≒ air	small	large	not difficult
CNB			≒ N_2 ≒ air	large	large	Skill is required
CVNB	≒ N_2 ≒ air	≒ air	≒ N_2 ≒ air	small	small	Skill is required

A≧B: A is equal to or higher than B, C≒D: C is almost equal to D,
E<F: F is higher than E, G<<H: H is much higher than G.

Appendix A-1. List of Participants.

Person / Laboratory	Address
Dr. Edith RUDOLPH BAM (Bundesanstalt fur Materialforschung und -prufung)	Unter den Eichen 87 1000 BERLIN 45 (GERMANY)
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Appendix A-2.

INSTRUCTIONS FOR THE VAMAS HIGH TEMPERATURE FRACTURE TOUGHNESS ROUND ROBIN

CONTENTS

I. Introduction

The purpose of the '90 VAMAS High Temperature Fracture Toughness Round Robin Test(RRT) is to assess methods to measure high temperature fracture toughness of advanced ceramics.

Three methods adopted to evaluate the fracture toughness at 1200℃ are as follows :

Single Edge V-Notched Beam (SEVNB) method[1).
Single Edge Pre-cracked Beam (SEPB) method[2).
Chevron Notched Beam (CNB) method[3).

Fracture toughness at room temperature is also evaluated by SEVNB method for reference.

SEVNB method is an improved technique using a V-shaped notch with very small root radius. A specimen is V-notched by a sharp V-shaped diamond wheel.

SEPB method uses a natural crack which is made by a bridge-indentation fixture[2). The technique was adopted in the '89 Fracture Toughness RRT.

CNB method is known as one of the reasonable techniques. The most important advantage of CNB method is that it is unnecessary to measure the crack length.

In these three methods, all the participants are required to adopt SEVNB method, and to choose one between SEPB and CNB method. The participants can certainly adopt all three methods.

Test pieces for each method are prepared by JFCC. Fracture toughness values of the participants, measured by the three methods with two cross-head speeds at 1200℃ and at room temperature, will be compiled and analyzed, and the report will be sent back to the participants.

II. Common Test Conditions in Three Methods

2.1 Test Piece

2.1.1 Material

Sintered silicon nitride, SSN-II(Kyocera corp.), is used for this RRT. This material has a stable strength at high temperature.

2.1.2 Shape and Dimensions of Test Piece

The test piece is of prism shape in rectangular cross section with dimensions complying with Fig.1.

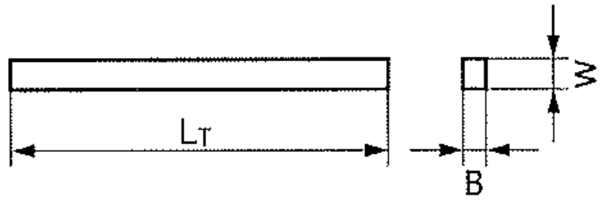


Fig.1. Dimensions of test piece.

Length overall	L_T	:	50 mm
Width	W	:	4.0±0.1 mm
Thickness	B	:	3.0±0.1 mm

The parallelism of upper and lower faces*[1] is not more than 0.01 mm.

*[1] For the definition of parallelism, refer to JIS B 0621.

2.1.3 Chamfering Edges of Test Piece

A test piece for the SEVNB and CNB methods is not chamfered.

Four edges of a test piece for the SEPB method shall be chamfered as shown in Fig.2.

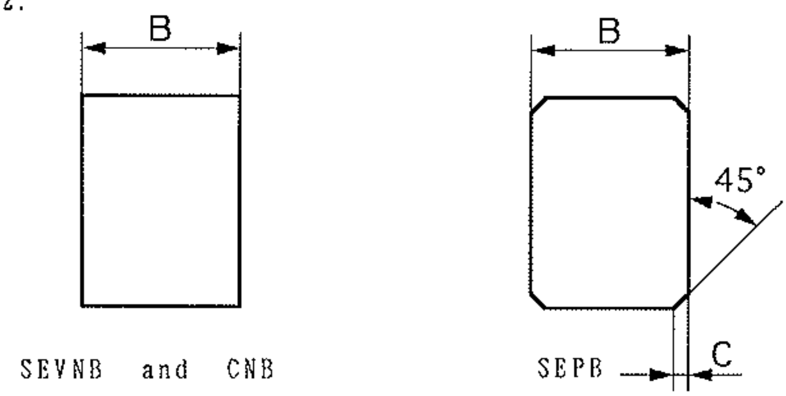


Fig.2. Cross-section of chamfered test piece.

c : 0.1 to 0.3 mm.

2.1.4 Roughness

The roughness of top, bottom, and side surfaces of a test piece is not more than 0.2 μm Ra specified in JIS B 0601, ANSI B 46.1 and BS 1134.

2.2 Apparatus and Support Devices

2.2.1 Testing Machine and Furnace

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

The high temperature furnace, capable of controlling the atmosphere around a test piece, should be attached to the testing machine.

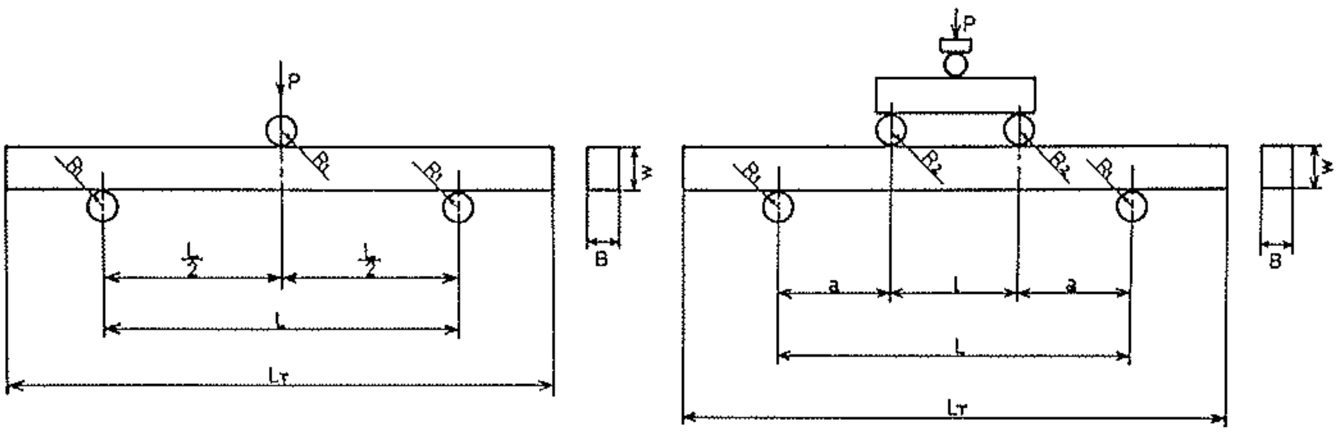
The testing in N_2 gas is recommended, and the testing in vacuum or in air is allowable for measuring fracture toughness at 1200°C.

Fracture toughness at room temperature is measured in air.

2.2.2 Flexure fixture

The material of flexure fixtures at supporting and loading points should have elastic modulus of 147 GPa(1.5×10^4 kgf/mm^2) or more, without plastic deformation and rupture during the test, and should not to adhere the test piece even at high temparature.

The radii of curvature of both supporting and loading rollers are shown in Fig.3.



(a) Three-point flexure (b) Four-point flexure

Fig.3. Dimensions and radius of curvature of flexure fixture.

Radius of curvature	R_1	: 2.0 to 3.0 mm
	R_2	: 0.5 to 3.0 mm
Support span	L	: 30 ± 0.5 mm, or 40 ± 0.5 mm
Load span	l	: 10 ± 0.5 mm, or 20 ± 0.5 mm
	a	: 10 ± 0.5 mm

The devices for 3-point flexure are used for SEVNB and SEPB methods. Those for 4-point flexure are used for CNB method and the spans 30 and 10 are recommemded. Spans 40 and 20 are allowable only if a participant has no fixture with spans 30 and 10.

Flexure fixtures made by SiC, graphite, mullite or alumina are recommended for measuring at 1200°C.

2.3 Distribution of Test Pieces

Test pieces, meeting the requirements mentioned above, are prepared by Japan Fine Ceramics Center (JFCC).

Table 1. Numbers of test pieces for each method.

		Crosshead Speed	
		0.5mm/min	0.005mm/min
SEVNB	R.T.	5 "	5 "
	1200°C	5 "	5 "
SEPB	1200°C	5 ""	5 ""
CNB	1200°C	5	5
spare pieces		5	

" V-notched , "" Chamfered

Test pieces for the SEVNB method have been already V-notched and those for the SEPB method chamfered by JFCC.

Forty-five test pieces for the three methods, as shown in Table 1, will be distributed to each participant from JFCC.

2.4 Deadline for Returning Data

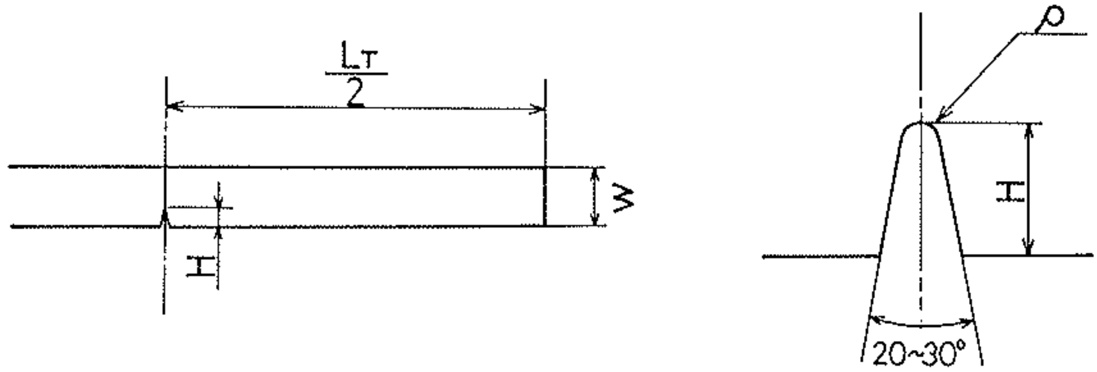
All the data sheets, test pieces after testing, and other data required should be sent back to JFCC by June , 1991.

Ⅲ. Testing Procedure

3.1 SEVNB Method

3.1.1 Test Piece

Test pieces distributed have already been V-notched using a diamond wheel cutter as shown in Fig.4.



Depth of V-notch H : 1.0～2.4 mm
Tip radius ρ : 10～30 μm

Fig.4 Shape and dimensions of V-notch

3.1.2 Cleaning of Test Pieces

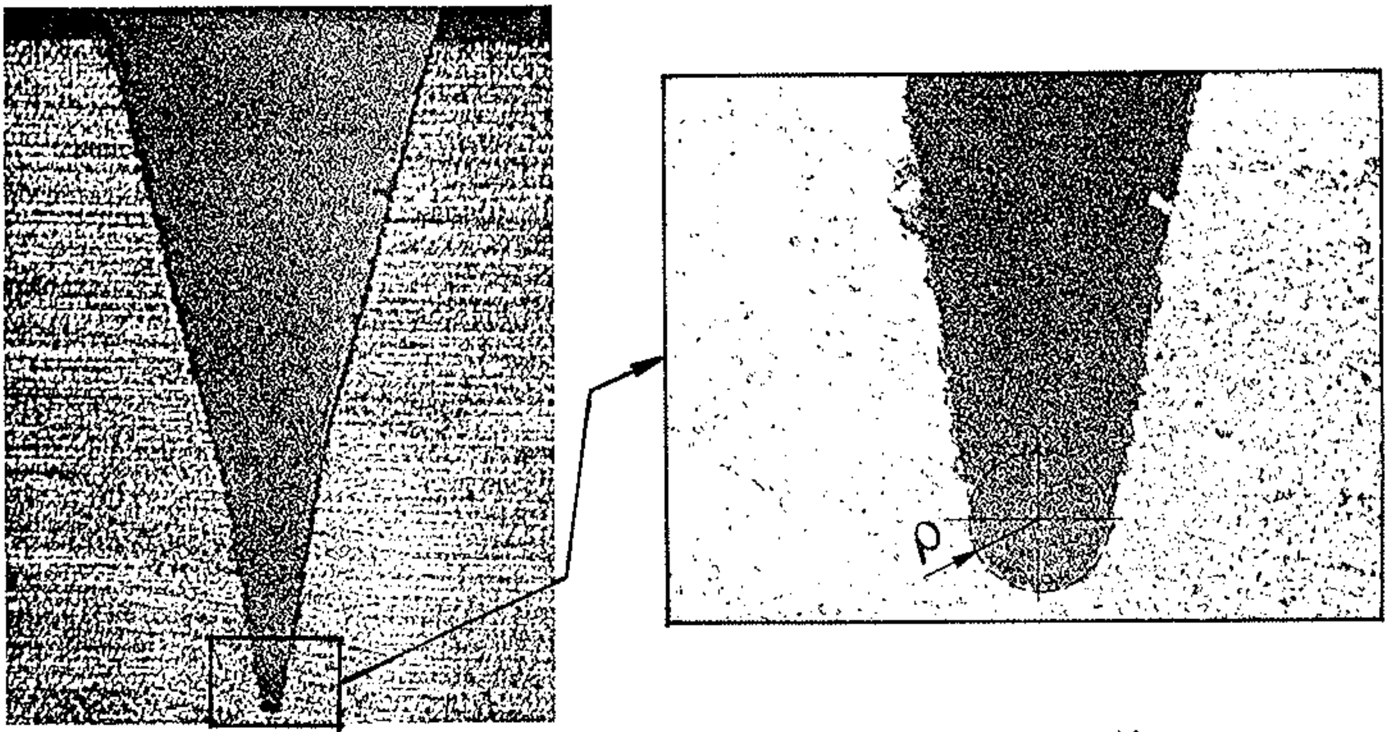
A Test piece should be cleaned of adhesive materials in acetone.

3.1.3 Measuring Dimensions of Test Piece

Give a specific number to a test piece.

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

Measure the radius of curvature (ρ) at the tip of a V-notch on a microscope of about 400 × magnification. Calibrate the microscope magnification using a graticule. The photograph of the notch is convenient for reading the value (Fig.5).



$\rho \approx 16\,\mu m$

Fig. 5. Photograph of V-notch and measuring the tip radius.

3.1.4 Placing Test Piece in Fixture

Place the 3mm-width face with a V-notch down.
Use 3-point flexure fixtures.

3.1.5 Measuring Fracture Load

The fracture load is defined as the maximum load when a test piece is loaded. Record load to 3 significant figures.
Measuring conditions are as follows.

Testing at 1200°C

Atmosphere	:	in N_2, in vacuum, or in air
Heating temperature	:	1,200℃ ± 12℃
Heating rate	:	5 to 15℃／min
		(from room temperature to 1200℃)
Duration at temperature	:	about 10 min or more
Cross-head speed	:	0.5 mm／min and 0.005 mm／mim. In other cases, it should be described in the report.

Testing at room temperature

Atmosphere : in air

Cross-head speed : the same as for at 1200°C

3.1.6 Measuring Average Depth of V-notch

The depth of a V-notch is measured by observing fracture surface, using a microscope of more than 20 × magnification. Photographs of the surface is convenient for reading the values but should be of calibrated magnification using a graticule.

An average depth of a V-notch (a) is obtained by eqn.(1).

$$a = \frac{a_1 + a_2 + a_3}{3} \quad \cdots\cdots\cdots\cdots (1)$$

where a_1, a_2 and a_3 are shown in Fig.6. Read them in mm down to 2 decimal places (i.e. ±0.01 mm). However, these values should be in the range from 1.2 to 2.4 mm and satisfy eqn(2).

$$\frac{a_{max} - a_{min}}{a} \leqq 0.1 \quad \cdots\cdots\cdots\cdots (2)$$

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

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

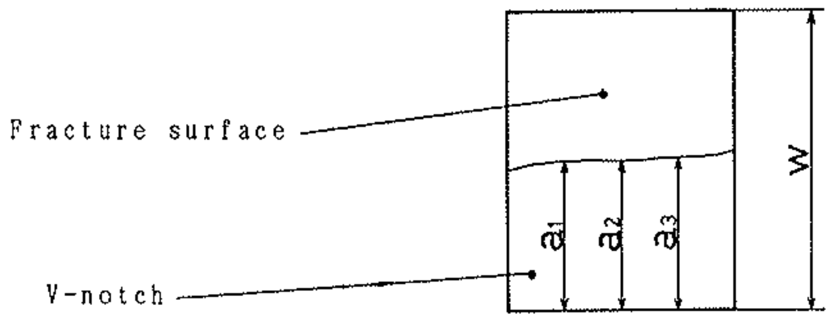


Fig.6. Measurement of V-notch depth.

3.1.7 Calculation of Fracture Toughness

The fracture toughness for a V-notched specimen, Kc,n, is calculated according to eqns.(3) and (4)*4 from the measured values of individual test pieces.

$$K_{c,n} = \frac{3}{2} \cdot \frac{P\,S}{B\,W^{1.5}} \cdot \alpha^{0.5} F(\alpha) \cdots\cdots\cdots (3)$$

$$\alpha = \frac{a}{W}$$

$K_{c,n}$	:	Fracture toughness	($MPam^{0.5}$)
P	:	Fracture load	(MN)
S	:	Support span	(m)
B	:	Thickness of test piece	(m)
W	:	Width of test piece	(m)
a	:	Average V-notch depth	(m)

$$F(\alpha) = 1.964 - 2.837\alpha + 13.711\alpha^2 - 23.250\alpha^3 + 24.129\alpha^4 \cdots\cdots\cdots (4)$$

Where, ratio S/W should be 7.5.

The fracture toughness values obtained by SEVNB method, Kc,n, depend on the values of the root radius of a V-notch tip. Based on the local fracture criterion, the ratio of Kc,n to K_{IC} (fracture toughness for the ideally cracked specimen) is calculated as follows[1];

$$\frac{K_{c,n}}{K_{IC}} = \frac{(1+\rho/2r_o)^{1.5}}{1+\rho/r_o} \cdots\cdots\cdots\cdots\cdots\cdots\cdots (5)$$

Where, ρ is the root radius of a V-notch tip, ro is a critical distance from the notch tip. The distance ro is determined experimentally using the measured Kc,n/K_{IC} ratio for the material. Our experiments show that the ro value was about 6 times of an average grain size for silicon niride. The average grain size (d) of SSN-H is 1.1μm .

Therefore, if the root radius of the specimen is measured, the ratio $K_{c,n}/K_{IC}$ can be calculated according to eqn.(5). Then, the fracture toughness values by SEVNB method are corrected using the root radius value of the V-notch tip, as follows.

$$K_{c,n}(\text{corrected}) = K_{c,n} \times \frac{1 + \rho / r_o}{(1 + \rho / 2r_o)^{1.5}} \cdots\cdots\cdots\cdots (6)$$

Where, $K_{c,n}$(corrected) is the corrected value for the root radius. The significant figure of the value is three.

Calculate the average $K_{c,n}$(corrected) value from five test pieces, and round it off the fractions to a decimal place.

3.1.8 Data Sheet on $K_{c,n}$ by SEVNB Method

Data sheets (A) and (B), for high temperature and room temperature data ,respectively, must be filled with the experimental and calculated data. They should be returned to JFCC with all the test pieces after SEVNB testing by June, 1991.

3.2 SEPB Method

3.2.1 Cleaning of Test Piece

See 3.1.2.

3.2.2 Measuring the Dimensions of Test Piece

Give a specific number to a test piece.

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

3.2.3 Vickers Impression

Introduce a Vickers impression , as shown in Fig.7, into the center region of the 3mm-width surface with chamfering edges of a test piece.

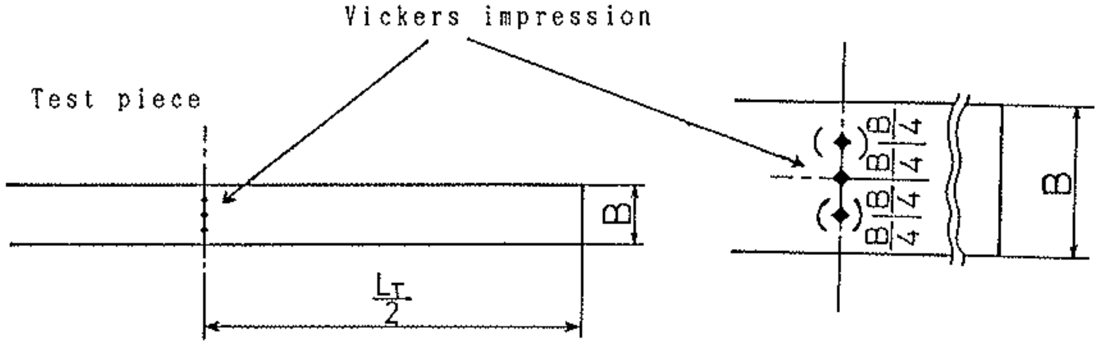


Fig.7 Precrack starter (Vickers Impression(s)) and its site.

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

3.2.4 Introducing a Precrack

The precracking fixture consists of a pusher and an anvil with a deflection groove as shown in Fig.8.

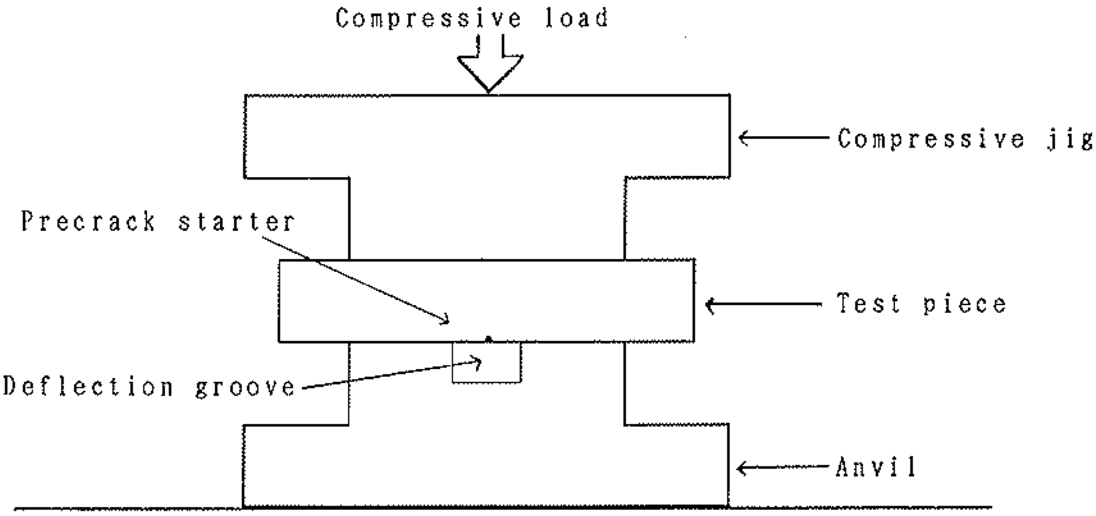


Fig 8 Precracking fixture.[2)]

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

Clean the fixture and the test piece surfaces with acetone.

Place the test piece 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.

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.

Valid range of precrack length : a = 1.2～2.4 mm.

3.2.5 Dye Penetration of Precrack

Dye penetrant, e.g. oil paint, mixed with acetone may be used to improve the visibility of the precrack.

3.2.6 Measuring Fracture Load

See 3.1.5

3.2.7 Measuring Precrack Length

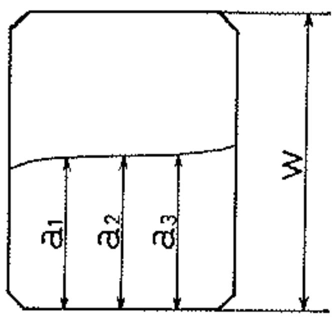
The precrack length is measured by observing the fracture surface of a test piece, using a microscope of more than 20 × magnification.

An average length of a precrack(a) is calculated in the same way as an average depth of a V-notch, using eqn.(7) (See 3.1.6).

Fracture surface

Precrack front

Vickers impression



$$a = \frac{a_1 + a_2 + a_3}{3} \quad \cdots (7)$$

Fig.9 Measurement of precrack length.

Lengths a_1, a_2, and a_3 are shown in Fig.9. Read them dowm to a decimal place. These values shall be in the range from 1.2 to 2.4 mm and satisfy eqn.(2) (See page 9).

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

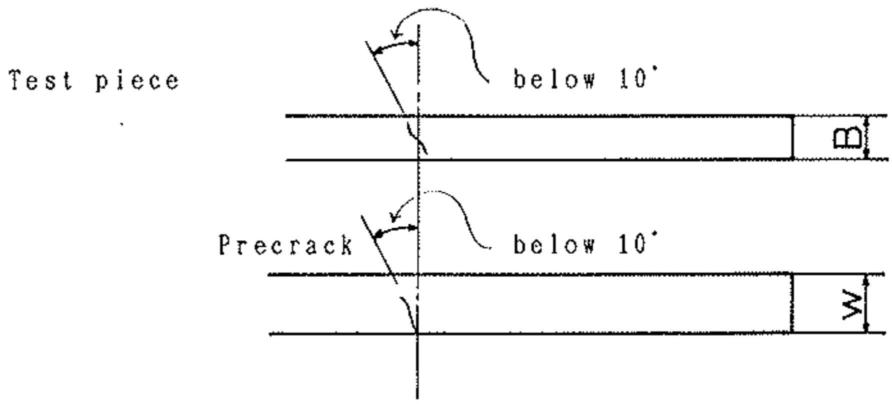


Fig.10 Angle allowance of precrack extension.

3.2.8 Calculation of K_{IC}

Calculate K_{IC} according to eqns.(8) and (9).

$$K_{IC} = \left(\frac{PS}{BW^{1.5}}\right)\left\{\frac{3}{2}\left(\frac{a}{W}\right)^{0.5}\cdot Y\left(\frac{a}{W}\right)\right\} \cdots\cdots\cdots (8)$$

$$Y\left(\frac{a}{W}\right) = 1.964 - 2.837\frac{a}{W} + 13.711\left(\frac{a}{W}\right)^2 - 23.250\left(\frac{a}{W}\right)^3 + 24.129\left(\frac{a}{W}\right)^4 \cdots\cdots\cdots (9)$$

Where

K_{IC}	:	Fracture toughness	($MPam^{0.5}$)
P	:	Fracture load	(MN)
S	:	Support Span	(m)
B	:	Thickness of test piece	(m)
W	:	Width of test piece	(m)
a	:	Precrack length	(m)

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

3.2.9 Data on K_{IC} by SEPB Method

Data sheet(C) should be filled with the experimental and calculated data, and should be returned to JFCC with all the test pieces after SEPB testing.

See 3.1.9.

3.3 CNB Method

3.3.1 Test Piece

Test pieces are required to be chevron-notched by participants using a diamond wheel cutter, wire saw, or even the V-notch cutter as shown in Fig. 11.

The groove is at the center of the test piece and is cut on the 3 mm-width side with chamfered edges.

The tip of a diamond wheel should be rounded, if possible.

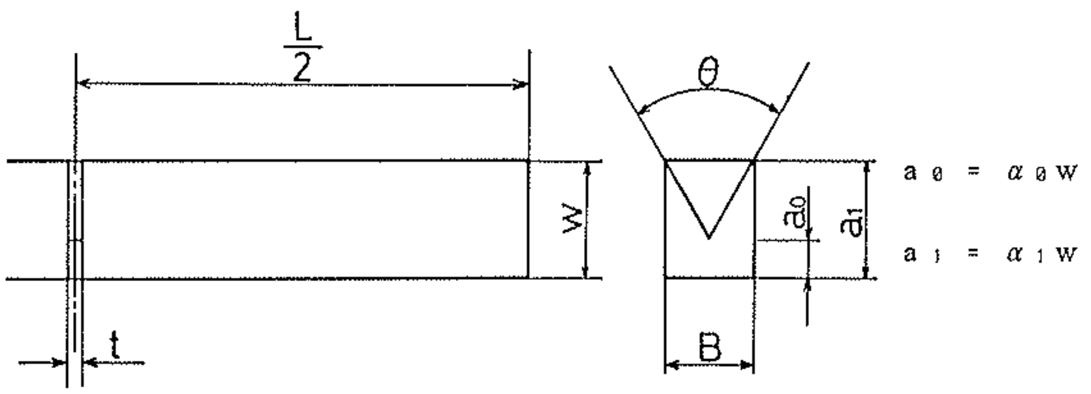


Fig.11 Chevron-notched Test Piece

w	:	4.0 mm
α_0	:	0.35 ±
α_1	:	0.99 to 1.00
θ	:	60 deg.
t	:	not more than 0.2 mm.

3.3.2 Cleaning of Test Piece

See 3.1.2.

3.3.3 Measuring Dimensions of Test Piece

Give a specific number to a test piece.

Measure the thickness and width of the test piece by micrometer calipers. Read the values in mm down to three decimal places.

Measure the width(t) of a chevron notch by its photograph (photo.1) on a microscope of about 100 × magnification. The photograph , showing the shape of a notch tip, from the direction as shown in Fig.11 is recommended.

3.3.4 Placing Test Piece in Fixture

Place the 3mm-width face notched down (Fig.12).

Use 4-point flexure fixtures.

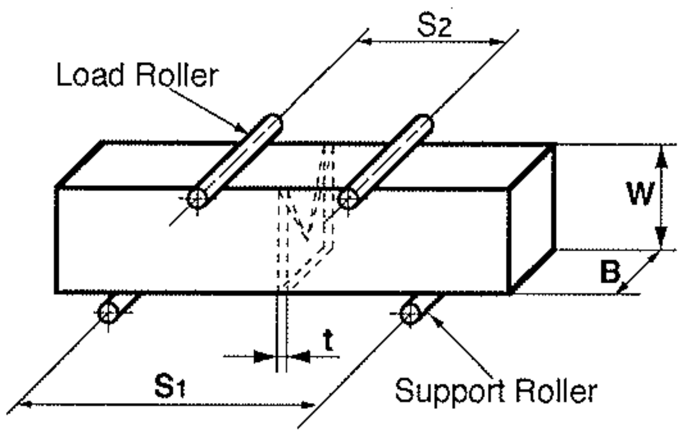


Fig.12 Placing test piece in fixture.

3.3.5 Measuring Fracture Load

See 3.1.5.

A load vs. time or a load vs. displacement curve, as shown in Fig. 13(a) , should be recorded in process of measuring fracture load.

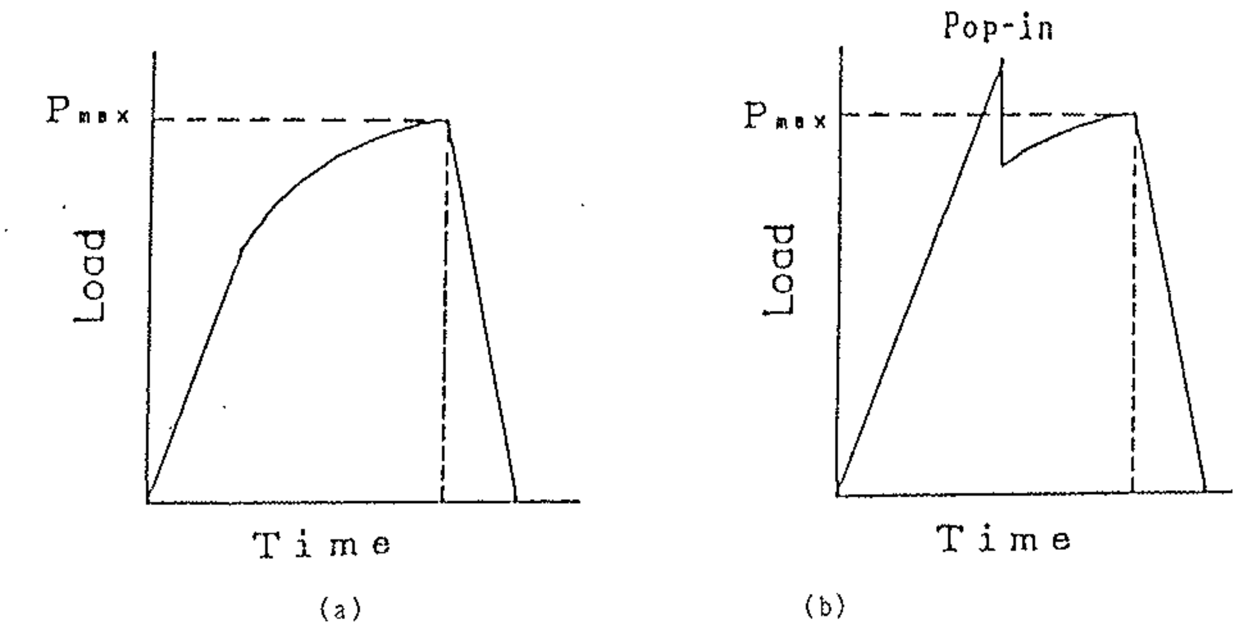
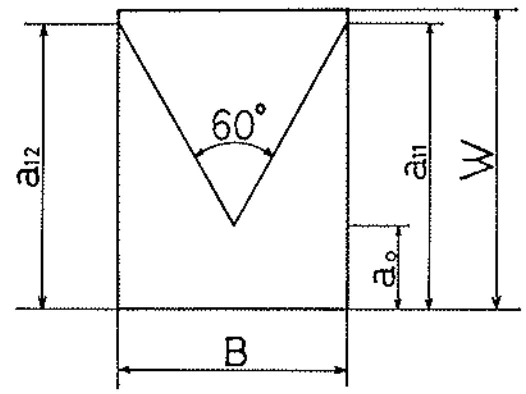


Fig.13 A load vs. time curve.

If a pop-in precrack is introduced to a test piece as shown in Fig. 13(b), the fracture load is defined as the maximum load after introducing a pop-in crack.

3.3.6 Measuring Initial Crack Length

Observe the fracture surface of a test piece on scanning electron microscope(SEM) or optical microscope(including a travelling microscope) of about 50 magnifications to measure the initial crack lengths(a_0 and a_1) of a chevron notch, as shown in Fig.14. The number of three figures is significant. Calibration for accurate measurements is necessary using a graticule.



$$a_1 = \frac{a_{11} + a_{12}}{2}$$

Fig.14 Measuring initial crack length.

3.3.7 Caluclation of K_{IC}

Caluculate K_{IC} according to eqn(10) for spans 30 and 10, and eqn(11) for spans 40 and 20. The number of three figures is significant.

Calculate the average K_{IC} value from five test pieces, and round it off the fractions to a decimal place.

In case of spans 30 and 10,

$$K_{IC} = \frac{P_{max}}{B \cdot W^{0.5}} (2.848 + 6.88\alpha_0 - 2.78\alpha_0^2 + 27.3\alpha_0^3) \times \frac{S_1 - S_2}{W} \cdot \frac{\alpha_1 - \alpha_0}{1 - \alpha_0} \left[1 + 0.007 \left(\frac{S_1 S_2}{W^2}\right)^{0.5}\right] \cdots\cdots (10)$$

In case of spans 40 and 20,

$$K_{IC} = \frac{P_{max}}{B \cdot W^{0.5}} (2.982 + 6.29\alpha_0 - 1.78\alpha_0^2 + 28.0\alpha_0^3) \times \frac{S_1 - S_2}{W} \cdot \frac{\alpha_1 - \alpha_0}{1 - \alpha_0} \left[1 + 0.007 \left(\frac{S_1 S_2}{W^2}\right)^{0.5}\right] \cdots\cdots (11)$$

Where

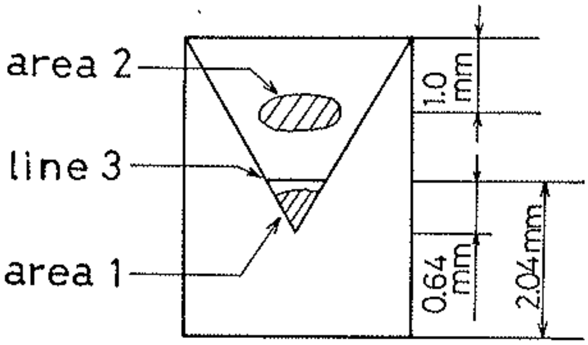
K_{IC}	: Fracture toughness	($MPam^{0.5}$)
P_{max}	: Fracture Load	(MN)
B	: Thickness of test piece	(m)
W	: Width of test piece	(m)
α_0	: Initial crack relative length	(a_0 / W)
α_1	: Initial crack relative length	(a_1 / W)
S_1	: Support span	(m)
S_2	: Load span	(m)

3.3.8 Observation of Fracture Surface

Observe the fracture surface of a chevron notch ligament on SEM.

Examine the differences of fracture behavior, regarding especially stable or unstable crack propagation, intergranular or transgranular fracture, and the boundary between intergranular and transgranular fracture (line 3 in Fig.15 is the boundary calculated theoretically).

Take at least two photographs (Photos. 2 and 3)of about 4000 magnifications in areas 1 and 2, as shown in Fig.15.



area 1 : stable crack propagation

area 2 : unstable crack propagation

line 3 : Theoretically calculated crack length at the maximum load at the minimum of Y* is 2.04 mm, as shown in Fig.15.

Y* : Dimensionless stress intensity factor coefficient.

Fig.15 Areas for photos. on fracture surface.

3.3.9 Data on K_{IC} by CNB Method

Data sheet (D) should be filled with the experimental and calculated data.

The sheet and photos (1)~(3) for each test piece should be returned to JFCC with all the test pieces after CNB testing.

R E F E R E N C E S

[1] H.Awaji et al., "Evaluation of Fracture Toughness by a Single-Edge V-notched Beam Method", Trans. Japan Soc. Mech.Eng.,56[525],1148-53 (1990) (in Japanese).

[2] T.Nose et al.,"Evaluation of Fracture Toughness for Ceramic Materials by a Single-Edge-Precracked-Beam Method", J. Am. Ceram. Soc.,71[5], 328-33 (1988).

[3] D.Munz, et al.,"Fracture Toughness Determination of Al_2O_3 Using Four-Point-Bend Specimens with Straight-Through and Chevron Notches", J. Am. Ceram.Soc.63[5-6],300-05(1980).

[4] JIS R 1607.

DATA SHEET(A) for SEVNB METHOD at HIGH TEMPERATURE

Laboratory : Date :

Cross-head speed: mm/min Support span: mm Atmosphere: N_2, Vac., Air Test temperature: °C Heating rate: °C/hr Duration time at high temp. min Furnace:	Testing machine: Max.capacity of machine: Load cell capacity: Load cell full scale: Chart speed: mm/min Material of fixture: Investigator:

	Test Piece No.					
	1	2	3	4	5	
B (10^{-3} m)						
W (10^{-3} m)						
a_1 (10^{-3} m)						
a_2 (10^{-3} m)						
a_3 (10^{-3} m)						
a (10^{-3} m)						
P (MN)						
F (α)						
ρ (10^{-3} m)						
$K_{c,n}$ / $K_{c,n}$(cor)						
$K_{c,n}$ ($MPam^{0.5}$)						
$K_{c,n}$(cor) ($MPam^{0.5}$)						
$K_{c,n}$(cor) (mean)	($MPam^{0.5}$)					

$r_0 = 6d = 6.6 \times 10^{-3}$ m

DATA SHEET(B) for SEVNB METHOD at ROOM TEMPERATURE

Laboratory : Date :

Cross-head speed: mm/min Support span: mm Atmosphere: Air Temperature: °C	Testing machine: Max.capacity of machine: Load cell capacity: Load cell full scale: Chart speed: mm/min Material of fixture: Investigator:

	Test Piece No.					
	1	2	3	4	5	
B (10^{-3} m)						
W (10^{-3} m)						
a_1 (10^{-3} m)						
a_2 (10^{-3} m)						
a_3 (10^{-3} m)						
a (10^{-3} m)						
P (MN)						
F (α)						
ρ (10^{-3} m)						
Kc,n／Kc,n(cor)						
Kc,n ($MPam^{0.5}$)						
Kc,n(cor) ($MPam^{0.5}$)						
Kc,n(cor) (mean)	($MPam^{0.5}$)					

DATA SHEET(C) for SEPB METHOD at HIGH TEMPERATURE

Laboratory : Date :

Cross-head speed: mm/min Support span: mm Atmosphere: N_2, Vac., Air Test temperature: °C Heating rate: °C/hr Duration time at high temp. min Furnace:	Testing machine: Max.capacity of machine: Load cell capacity: Load cell full scale: Chart speed: mm/min Material of fixture: Investigator:

	Test Piece No.					
	1	2	3	4	5	
B (10^{-3} m)						
W (10^{-3} m)						
a_1 (10^{-3} m)						
a_2 (10^{-3} m)						
a_3 (10^{-3} m)						
a (10^{-3} m)						
P (MN)						
K_{IC} ($MPam^{0.5}$)						
K_{IC} (mean)	($MPam^{0.5}$)					

DATA SHEET(D) for CNB METHOD at HIGH TEMPERATURE

Laboratory : Date :

Cross-head speed: mm/min	Testing machine:
Support span: mm	Max.capacity of machine:
Loading span: mm	Load cell capacity:
Atmosphere: N_2, Vac., Air	Load cell full scale:
Test temperature: °C	Chart speed: mm/min
Heating rate: °C/hr	
Duration time at high temp. min	Material of fixture:
Furnace:	Investigator:

	Test Piece No.					
	1	2	3	4	5	
B (10^{-3} m)						
W (10^{-3} m)						
a_0 (10^{-3} m)						
α_0						
a_{i1} (10^{-3} m)						
a_{i2} (10^{-3} m)						
a_i (10^{-3} m)						
α_i						
t (10^{-3} m)						
P_{max} (MN)						
K_{IC} ($MPam^{0.5}$)						
K_{IC} (mean)	($MPam^{0.5}$)					

Appendix A-3. Fracture Toughness Data Sheet from Laboratories.
(1) SEVNB Method.

SEVNB $K_{c,n}$ (MPa·$m^{0.5}$) − ρ (μm)
3 − Point Flexure : Span 30 mm

Temperature		Room Temperature : in Air			
CHS (mm/min)		0.005	0.02	0.05	0.5
1	Avr.	5. 2		5. 6	5. 1
		4.83-22 5.49-22 5.86-22 5.16-22 4.59- 7		5.36-22 5.87-22 5.41-20 5.88-22 5.58-22	5.81-24 4.67- 7 5.49-22 5.01-26 4.68- 7
	σ_{n-1}	0. 5		0. 5	0. 5
2	Avr.	4. 6			4. 5
		5.02-14 4.35-14 4.55-11 4.47-15 4.77- 8			4.85-13 4.57-14 4.70-15 4.56-14 3.96-10
	σ_{n-1}	0. 3			0. 3
3	Avr.	4. 9			4. 9
		4.47-17 4.77-16 4.94-17 5.04-17 5.07-17			4.71-15 5.05-12 4.72-15 5.02-16 4.83-16
	σ_{n-1}	0. 2			0. 2
4	Avr.	4. 8			4. 7
		4.19-14 5.24-14 5.03-13 4.88-13 4.79-13			4.98-13 4.67-13 4.46-14 4.51-14 4.78-10
	σ_{n-1}	0. 4			0. 2

SEVNB $K_{c,n}$ (MPa·$m^{0.5}$) − ρ (μm)
3 − Point Flexure : Span 30 mm, Span 40 mm

Temperature		Room Temperature : in Air			
CHS (mm/min)		0.005	0.02	0.05	0.5
5	Avr.	5. 6			5. 7
		5.54-28 5.74-33 5.51-33 5.19-28 5.85-29			5.63-33 6.18-33 5.63-32 5.21-28 5.87-33
	σ_{n-1}	0. 3			0. 4
6	Avr.		4. 6		4. 6
			4.78-12 4.30-11 4.71- 9 4.39-11 5.02-11		4.64- 9 4.54-14 4.54-14 4.56- 9 4.67- 9
	σ_{n-1}		0. 3		0. 1
7	Avr.	5. 1			5. 2
		5.06-30 4.90-29 5.24-28 5.01-27			4.84-30 5.60-29 5.16-29 4.80-30 5.63-30
	σ_{n-1}	0. 1			0. 4
8	Avr.	5. 2			5. 7
		5.28-32 4.79-32 4.96-23 5.59-32			5.57-31 5.53-31 5.72-32 5.56-26 5.87-33
	σ_{n-1}	0. 4			0. 1

S E V N B 3 − Point Flexure: Span 30mm in Air or in N_2, Span 40mm in Air
Kc.n($MPa \cdot m^{0.5}$) − ρ (μm) 4 − Point Flexure: Spans 40 & 20mm in Air

Temperature		1,200° C						
CHS (mm/min)		0.005		0.02	0.05		0.5	
Atmosphere		N_2	Air	Air	N_2	Air	N_2	Air
1	Avr.	4.9	6.2		4.9	5.7	5.0	6.3
		4.81-7 4.59-15 4.91-8 5.28-30	5.83-25 6.36-25 7.07-25 6.08-25 5.89-25		5.15-20 4.86-8 4.60-8 4.92-8 4.78-8	4.67-15 5.31-15 5.93-28 6.12-28 6.16-28	5.47-30 4.90-7 4.70-8 4.65-8 5.36-30	7.23-28 6.57-28 5.43-28 5.83-28 6.34-28
	σ n-1	0.3	0.5		0.2	0.6	0.4	0.7
2	Avr.	5.9					5.3	
		6.14-12 5.39-8 7.10-8 5.50-11 5.15-14					4.65-13 4.92-14 4.89-14 4.94-11 7.05-11	
	σ n-1	0.8					1.0	
3	Avr.	4.3					4.4	
		4.21-16 4.34-19 4.50-15 4.10-12 4.19-14					4.44-17 4.41-16 4.26-13 4.39-12 4.38-13	
	σ n-1	0.2					0.1	
4	Avr.		6.7					5.7
			6.27-13 7.14-13 6.39-13 6.72-14 6.30-9					5.36-14 5.66-13 6.39-13 5.96-14 5.31-10
	σ n-1		0.3					0.4

S E V N B 3 − Point Flexure: Span 30mm in Air or in N_2, Span 40mm in Air
Kc.n($MPa \cdot m^{0.5}$) − ρ (μm) 4 − Point Flexure: Spans 40 & 20mm in Air

Temperature		1,200° C						
CHS (mm/min)		0.005		0.02	0.05		0.5	
Atmosphere		N_2	Air	Air	N_2	Air	N_2	Air
5	Avr.		6.9					6.8
			7.52-28 6.78-28 6.92-30 6.42-32 6.66-32					6.31-32 7.14-32 6.41-33 6.46-32 5.99-33
	σ n-1		0.4					0.3
6	Avr.			6.2				5.3
				6.16-11 6.16-12 6.16-11 6.40-11				4.96-9 5.46-9 4.99-10 6.42-8 4.60-9
	σ n-1			0.1				0.7
7	Avr.		6.1					5.5
			6.57-30 6.13-22 6.05-30 5.54-30					5.60-29 7.21-28 4.61-14 4.83-15 5.17-30
	σ n-1		0.4					1.0
8	Avr.		6.4					6.2
			6.23-33 6.41-32 6.92-31 5.87-15 5.79-32					5.38-31 6.34-32 6.16-32 6.39-25 5.58-33
	σ n-1		0.4					0.5

Appendix A-3. Fracture Toughness Data Sheet from Laboratories.
(2) SEPB Method.

SEPB, K_{IC} ($MPa \cdot m^{0.5}$)
3 - Point Flexure : Span 30mm in Air or in N_2, Span 40mm in Air
4 - Point Flexure : Spans 40 & 20mm in Air

Temperature		Room Temperature		1200°C				
CHS (mm/min)		0.005	0.5	0.005		0.02	0.5	
Atmosphere		Air	Air	N_2	Air	Air	N_2	Air
1	Avr.	5. 9	5. 9	5. 2	8. 6		5. 4	8. 6
		6.02 5.84 5.78	6.02 5.97 5.57	5.23 5.24 5.24	9.53 8.22 8.08 9.94 7.24		5.34 5.29 5.42	10.2 8.96 7.75 8.74 7.31
	σ n-1	0. 1	0. 2	0. 1	1. 1		0. 1	1. 1
2	Avr.			5. 3			5. 2	
				5.35 5.69 4.70 5.51 5.27			5.14 4.23 5.36 5.66 5.44	
	σ n-1			0. 4			0. 6	
3	Avr.			4. 7			4. 8	
				4.48 4.92 4.43 4.99 4.62			5.04 4.66 4.77 4.61	
	σ n-1			0. 3			0. 2	

SEPB, K_{IC} ($MPa \cdot m^{0.5}$)
3 - Point Flexure : Span 30mm in Air or in N_2, Span 40mm in Air
4 - Point Flexure : Spans 40 & 20mm in Air

Temperature		Room Temperature		1200°C				
CHS (mm/min)		0.005	0.5	0.005		0.02	0.5	
Atmosphere		Air	Air	N_2	Air	Air	N_2	Air
5	Avr.				8. 1			8. 0
					9.04 7.65 8.33 7.45 7.54			9.26 7.27 7.27 8.49 7.76
	σ n-1				0. 8			0. 9
6	Avr.					8. 6		8. 6
						9.31 8.42 8.94 7.34		8.11 8.38 8.94
	σ n-1					1. 0		0. 5
7	Avr.				8. 6			8. 5
					10.7 7.74 8.39 8.00 7.70			8.33 7.91 9.44 8.39
	σ n-1				1. 1			0. 5
8	Avr.				8. 3			9. 0
					8.91 7.41 9.72 8.00 7.54			8.41 9.87 7.12 10.2 9.18
	σ n-1				1. 0			1. 2

Appendix A-3. Fracture Toughness Data Sheet from Laboratories.
(3) CNB Method.

CNB, K_{IC} (MPa·m$^{0.5}$) — 4-Point Flexure : Spans 30 & 10mm in Air or in N_2 / Spans 40 & 20 mm in Air or in N_2

Temperature		Room Temperature				1,200° C						
CHS (mm/min)		0.005	0.05	0.5		0.005		0.05		0.5		
Atmosphere		Air	Air	Air		N_2	Air	N_2	Air	N_2		Air
1 (#1)	Avr.	5.3	5.6	5.4	5.6	5.2	5.4	5.4	5.4	5.3	5.5	5.7
		4.90 5.22 5.56 5.50 5.50	6.00 5.83 4.65 6.05	5.73 4.95 4.66 5.62 5.85	5.53 5.54 6.02 5.12 5.68	5.22 5.29 5.10 5.42 5.15	5.20 5.33 5.64 5.31 5.45	5.47 5.44 5.67 5.22 5.42	5.42 5.45 5.33 5.38	4.86 5.40 5.07 5.49 5.65	5.44 5.30 5.45 5.86 5.52	5.64 5.45 5.89 5.75 5.81
	σ_{n-1}	0.3	0.7	0.5	0.3	0.1	0.2	0.2	0.1	0.3	0.2	0.2
2 (#2)	Avr.					6.7				6.6		
						7.39 6.35 7.05 6.97 5.74				5.49 5.49 5.66 9.72		
	σ_{n-1}					0.6				2.1		
4 (#2)	Avr.						5.6					6.7
							5.61 5.55 5.49 5.51 5.88					6.42 6.46 7.24 6.42 6.85
	σ_{n-1}						0.2					0.4

#1: V-notched #2: Straight-notched

CNB, K_{IC} (MPa·m$^{0.5}$) — 4-Point Flexure : Spans 30 & 10mm in Air or in N_2 / Spans 40 & 20 mm in Air or in N_2

Temperature		Room Temperature				1,200° C						
CHS (mm/min)		0.005	0.05	0.5		0.005		0.05		0.5		
Atmosphere		Air	Air	Air		N_2	Air	N_2	Air	N_2		Air
5 (#2)	Avr.						5.6					5.9
							5.50#3 6.56 5.22#3 5.27#3					6.03#3 6.08#3 5.89 5.49
	σ_{n-1}						0.6					0.3
6 (#2)	Avr.						6.4					6.8
							6.69 6.90 5.67					8.57, 6.19 6.18, 6.26 5.74, 7.52 7.98, 6.33 5.97, 7.14
	σ_{n-1}						0.7					1.0
7 (#2)	Avr.						4.5					4.7
							4.21 4.98 4.17					5.06 4.36 4.79
	σ_{n-1}						0.4					0.3
8 (#2)	Avr.						5.5					5.8
							5.40 5.91 5.72 5.07					5.75 5.64 6.15 5.46 5.74
	σ_{n-1}						0.4					0.3

#1: V-notched #2: Straight-notched #3: Chevron was slightly overcut.