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THE VAMAS FRACTURE TOUGHNESS TEST ROUND-ROBIN ON CERAMICS

by

Hideo Awaji, Jun-ichi Kon and Hiroshi Okuda

SUMMARY

This report is concerned with the results of VAMAS international round robin test for evaluating fracture toughness of ceramics. Thirteen laboratories in Japan, France, Germany, UK, Canada, Belgium and CEC (JRC Petten) took part in the test. The values of fracture toughness obtained by the SEPB, the IF and the IS methods are compared with one another for gas-pressure sintered silicon nitride (GPSSN) and zirconia-alumina composites (ZAC). Also examined are indentation load dependence of fracture toughness measured by the IF and the IS methods, and loading rate dependence of fracture toughness by the SEPB method.

The results showed that the toughness values evaluated by the SEPB method have relatively wide scatter among the participants, suggesting some difficulties of carrying out this technique. The IS method gave the smallest scatter in the values measured and the IF method the largest scatter. Fracture toughness measured by the SEPB method depends on loading rate, especially for ZAC. This may be caused by stress corrosion cracking. The values measured by the IF and the IS methods depend apparently on indentation load.

CONTENTS

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	National Research Laboratory of Metrology, Tsukuba
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	Bundesanstalt für Materialforschung (BAM), Berlin
UK	National Physical Laboratory (NPL), Teddington, Middlesex
	Harwell Laboratory, Didcot, Oxon
CANADA	Industrial Materials Research Institute, Boucherville, Quebec
BEIGIUM	Centre De Recherches de l'Industrie Belge de la Ceramicue, Mons
CEC	Joint Research Center, Petten, The Netherlands

1. PREFACE

Based on the mutual consent at the Versailles Summit, the Versailles Project on Advanced Materials and Standards (VAMAS) has been proceeding to provide the technical basis for drafting codes of practice and specifications for advanced materials[1]. Among several technical working areas in VAMAS Project, the ceramics division has carried out the international round robin test (RRT) of hardness, flexural strength and fracture toughness for several years, as a basic research for standardization of testing procedure for ceramics.

'89 Fracture Toughness RRT was conducted by Japan Fine Ceramics Center in the period between April, 1988 and June, 1990. Twenty-three laboratories including four Japanese participants took part in the RRT, and thirteen results have been returned. This suggests that one of these techniques used has testing difficulty for most participants. Before conducting the international RRT, Japanese participants finished the work in 1988, of which instruction was slightly different from the one for the international RRT. The instruction of the international RRT is shown in the Appendix.

2. INTRODUCTION

Strtuctural ceramics have excellent mechanical properties such as high strength even at elevated temperatures, high hardness, and corrosion resistance. Despite the several advantages, the strength of ceramics is very sensitive to microscopic defects because of their low toughness. Also, there are some obstacles in evaluating fracture toughness of ceramics such as the difficulty in making precrack, R curve behaviour and slow crack growth. Therefore, one of the primary subjects for the development of structural ceramics is how its fracture toughness is evaluated accurately.

The purpose of the '89 Fracture Toughness RRT is to assess the methods of measuring fracture toughness parameters of advanced ceramics. The ceramic materials used are gas-pressure sintered silicon nitride (GPSSN) and zirconia-alumina composites (ZAC).

The following three methods are adopted to evaluate fracture toughness of ceramics; Single Edge Pre-cracked Beam (SEPB) method[2][3], Indentation Microfracture or Indentation Fracture (IF) method[4], and Indentation Strength (IS) method[5]. In Japan, the SEPB method is regarded as one of the most reliable techniques[6]. This technique has several advantages such as theoretical simplicity and good reproduciblity for common structural ceramics. The SEPB method uses a "pop-in" pre-crack arising from a Vickers indent or a straight-through notch when the specimen is compressed with a bridge indentation fixture[2]. However, the disadvantages are that this technique is useless if the crack front is not visible with or without dye penetrant, and that it is difficult to induce a precrack in some ceramics.

The IF method is known as the most convenient technique. It only needs small area, and the procedure is quite simple; measurements of diagonals of a Vickers impression and crack lengths are only necessary for evaluating the fracture toughness. It is particularly useful technique for routine quality control in individual work. However, this technique has also several disadvantages such that the crack length measurement is difficult in some ceramics, and the measured fracture toughness apparently depends on the indentation load.

The IS method is another convenient technique. A specimen precracked by a Vickers indentation is subjected to a flexure load. The fracture toughness values can be calculated from Young's modulus, Vickers hardness, indentation load and flexural strength. The most important advantage of the technique is that the crack length measurement is not necessary. But it has been pointed out that the values of fracture toughness increase with increasing indentation load[11].

The values of fracture toughness for two materials measured by each participant using three methods are compiled and analyzed. Also examined are indentation load dependence of fracture toughness by the IF and the IS methods, and loading rate dependence of the fracture toughness by the SEPB method.

Twenty-three sets of the specimens and instructions for measurements were sent to six laboratories in USA, four in Japan and France, three in UK and Germany, one in Belgium, Canada and CEC. Thirteen laboratories have carried out the tests and returned the results.

3. RRT METHODS

3.1 MATERIALS

Following two kinds of ceramics were used.

GPSSN (EC-141, NTK)

ZAC (UTZ-20, NTK)

A material GPSSN contains only small defects and has homogeneous structure including alumina and yttria as additives. A material ZAC is a pressure-less sintered zirconia-alumina composites, consists of about 50 vol.% zirconia and alumina. Young's moduli of GPSSN is 310 GPa and that of ZAC is 280 GPa.

Twenty specimens per each material were sent to the participants. Dimensions of the specimen were 3mm X 4mm X 40mm. One of the 4mm width-side was mirror finished surface ground by #2000 diamond wheel to improve visibility of Vickers impression and cracks on the IS and the IF specimens. Of the three methods adopted here, the IS method was performed first, using twenty specimens per each material (each ten specimens for two indentation loads). Forty specimens with about a half length after measuring fracture toughness by the IS method were available for the SEPB method, which needed each ten specimens for two cross-head speed tests; faster (ex. 1 mm/min) and slower (ex. 0.005 mm/min). The specimens fractured for the IS method were used for the IF measurement.

3.2 IS METHOD

Mirror-finished surface was indented by Vickers hardness machine. To estimate indentation load dependence, the testing was performed under the loads of 49 and 294N for GPSSN, and 98 and 490N for ZAC. The indentations were made in the middle of the specimen. Then, three-point flexure strength of the specimen was measured with 30mm supporting span and 0.5mm/min cross-head speed. Fracture toughness was calculated by the following equation[5];

$$K_{Ic} = 0.59(E/HV)^{1/8}(\sigma_c P_v^{1/3})^{3/4} \qquad (1)$$

where, E is the Young's modulus, HV the Vickers hardness, σ_c the flexure strength and P_v the Indentation load.

Japanese participants did not carry out this test.

3.3 SEPB METHOD

The SEPB method uses a bridge-indentation fixure[2] to make a precrack which arises from a Vickers indent. The specimen with precrack is bent to obtain fracture toughness. Therefore, if a material shows stress corrosion cracking behaviour by moisture, the fracture toughness may depend on loading rate at the test. To estimate loading rate dependence, two cross-head speeds (1 and 0.005mm/min) were used.

As a precrack starter, one 98N-indent was made for each GPSSN specimen and three 196N-indents for ZAC. After making the precrack by a bridge indentation fixture, a dye penetrant mixed with acetone was used to improve the visibility of the precrack. Three-point flexure test with 16mm supporting span was used to fracture the specimens. The stress intensity factor is given by the following equations[7],

$$K_I = \frac{3SP}{2BW^2} a^{1/2} F(a/W) \qquad (2)$$

$$F(a/W) = \frac{1.99-(a/W)[1-(a/W)][2.15-3.93(a/W)+2.7(a/W)^2]}{[1+2(a/W)][1-(a/W)]^{3/2}}$$

where, S is the supporting span, a the precrack length, W the specimen height, B the specimen width, and P the load.

3.4 IF METHOD

The indentation loads used were 98 and 196N (196 and 294N in Japan) for GPSSN specimens, and 294 and 490N (only 294N in Japan) for ZAC specimens. Fracture toughness was calculated by the following two equations;

Miyoshi et al.[8],

$$K_c = 0.0264E^{0.5}P^{0.5}c^{-1.5}a \qquad (3)$$

Marshall and Evans[4]

$$K_c = 0.036E^{0.4}P^{0.6}a^{0.8}c^{-1.5} \qquad (4)$$

where, E is the Young's modulus, P the indentation load, a the half of the diagonal length of the impression, and c the half crack length.

4. RESULTS AND DISCUSSION

4.1 SEPB METHOD

There must have been some confusions in carrying out the SEPB method, because it was a new technique for the majority of participants and it needed special fixtures. Nevertheless, many participants tried to make the fixtures and evaluated the fracture toughness.

Table 1 summarizes all of the data for the means and the standard deviations of the fracture toughness measured by the SEPB method. Common cross-head speeds are 0.005 and 1.0 mm/min. Other speeds used are shown in parenthesis.

Figure 1 shows the loading rate dependence of the fracture toughness for GPSSN by the SEPB method, and Fig. 2 is for ZAC. Relatively wide spread of results among laboratories suggests that the SEPB measurement technique involved some difficulties, and that the workmanship of the fixture made by each participant considerably affects the results. The results obtained by the Japanese participants, however, show small scatter because they are used to this technique.

Almost every data increases with increasing cross-head speed. Subcritical crack growth by moisture is believed to be responsible for this behaviour, especially for ZAC. Therefore, the loading rate is considered to be one of the most important factors in evaluating fracture toughness of ceramics with some kinds of oxide, such as silica.

The precrack length ratio a/W should be long enough to avoid the influence of residual stresses at the crack tip resulting from the starting indentations. On the contrary, there is a possibility of oblique precrack extension and stable crack growth in the longer crack range. Therefore, the JIS[10] prescribes that the crack length ratio, a/W, should be within the range of $0.3 \leq a/W \leq 0.6$. However, some of the crack length ratios measured by the participants No. 12, 6 and 2 were larger than 0.6 for GPSSN and ZAC, and some of the values by the participants 9 and 10 were a little smaller than 0.3 for ZAC, which may cause wide scatter of the data.

Figures 3 to 6 show the each participants' standard deviations of the fracture toughness measurements by the SEPB method. In spite of the large scatter in Figs. 1 and 2, the standard deviations of each participants' measurements are relatively small. Among the participants, the standard deviations of No. 12 and 6 show high values. This suggests that the fixtures made by themselves have had enough precision. The scatter of data measured by the SEPB method seems to be mainly caused by the participants' technique and fixture.

4.2 IF METHOD

The indentation-load dependence of fracture toughness measured by the IF method is given in Table 2 and Fig. 7 for GPSSN, and Table 3 and Fig. 8 for ZAC. A slight decrease in fracture toughness with increasing load is seen.

The ratio of crack length to diagonal length c/a should be larger than 2.3 to guarantee a well developed median crack formation. In Fig. 7, some data obtained by 98N-indentation load do not satisfy this condition. The data of participant No.5 and 6 are not satisfied with the condition for all data obtained by 98N, and for almost every data by 196N. The data of No. 12 is not also satisfied with the condition for two specimens obtained by 98N. The participant No. 2 indented Vickers impression until they got ten data of which c/a was larger than 2.3, based on the proposal. As a result, about half of the data was useless. The 98N-indentation load for GPSSN may be too small to induce a median crack. This also depends on the condition of the diamond indenter used by each laboratory.

The material ZAC was difficult to make a mirror surface by a diamond wheel grinding compared with GPSSN, and detecting the crack tip on the surface was more difficult than that on GPSSN. Therefore, the measured values tend to depend on the personal bias and magnification of the optical equipment, as pointed out by the report on VAMAS hardness test round-robin on ceramic materials[9].

Figures 9 to 12 show the standard deviations of these results. The data of GPSSN has smaller scatter than the one of ZAC. The standard deviations of No. 5 and 6 generally show large values and their fracture toughness values are higher than others. This suggests the edge of their diamond indenter used was worn. Figure 13 shows the relationship between the mean fracture toughness and the coefficients of variation measured by each participant for ZAC with 294N-indentation, where the coefficients of variation is (the standard deviations)/(mean fracture toughness). It shows that the higher fracture toughness values have the higher coefficients of variation, which might be caused by using a worn indenter. Linear regression analysis based on least square approximation gives 0.62 as correlation coefficient.

4.3 IS METHOD

Table 4, Fig. 14 and 15 are the results of the indentation load dependence of fracture toughness measured by the IS method. The results show the quite small inter-laboratory scatter in fracture toughness values in comparison with that observed by other two techniques. Results on annealed specimens after indentation measured by participant No. 2 are also shown in Fig. 14 for reference. It means that the equation of Chantikul et al.[5] becomes invalid for the specimen after annealing treatment, because the analytical equation is based on the existence of the residual stress field induced by the Vickers indentation. It can also be considered that its applicability is doubtful at high temperature.

Figures 16 to 19 show the standard deviations of these results. The scatter is quite small, and it is independent of the participants and material difference, which means the materials used were very homogeneous, and were able to give very reproducible results.

Table 5 is the summary of the results. It shows range of the mean and the standard deviations of fracture toughness values measured by each participant, overall mean fracture toughness and its standard deviations, and the number of laboratories. This table also includes the data by the SEPB method with 1.0mm/min CHS, the IF method with 196N-indentation load for GPSSN and 294N for ZAC, and the IS method with 294N for GPSSN and 490N for ZAC. Material GPSSN shows small scatter than ZAC, especially that obtained by the IS method. The material GPSSN (EC-141,NTK) is known as having only small defects and homogeneous structure. As a result, the IS method has small scatter, because of its simple procedure. The difference among the overall mean fracture toughness obtained by these three techniques is rather small, compared with the scatter in results obtained by each laboratory.

The results of the SEPB method are slightly lower than others. Considering this fact, there were several comments from the participants as follows: (A) Span to depth ratio of the test specimen is too small to avoid shear effects. (B) There is a possible influence of residual stresses at the crack tip resulting from the starting indentations. The residual stresses may reduce the value of fracture toughness. (C) Dye penetration is not adequate to define clearly the length of the crack produced before the fracture test, especially in the dark materials.

For the comment (A), our thought is that the shear stresses do not affect the stress intensity factor of the specimen even if span to depth ratio is small. To avoid the influence of residual stresses, crack length ratio, a/W, should be larger than 0.3, and indentation load should be less than 98N[10]. In this RRT, we used a 98N-indentation for GPSSN and three 196N-indentation for ZAC. Therefore, the residual stresses might be possible to influence on ZAC specimen.

There were some bad reputations about SEPB method because it was unfamiliar technique for the most participants, and it needed special fixtures. On the contrary, there were affirmative comments that the SEPB bridge-indentation fixture worked well.

5. CONCLUSIONS

The results of the VAMAS Fracture Toughness Round Robin Test have been analyzed to assess the methods of measuring fracture toughness for advanced ceramics. It is concluded that:

(1) The material GPSSN used was very homogeneous and was capable of giving reproducible results. The scatter seems to be caused by the participants' technique, fixtures or equipments rather than by the materials.

(2) Relatively wide scatter in the results obtained by the SEPB method among laboratories would be caused by some difficulties in measurement technique. The fracture toughness values measured by

the SEPB method were the smallest of these three techniques.

(3) The loading rate dependence of the fracture toughness measured by the SEPB method was significant for ZAC.

(4) The IF method gave the largest spread of results because of the difficulty in detecting the crack tips, specially in white (tanslucent) materials.

(5) The fracture toughness measured by the IF method is sensitive to the condition of the diamond indenter used. A worn indenter may give higher fracture toughness and higher standard deviations.

(6) The IS method shows small inter-laboratory scatter in comparison with the other two techniques. The analytical expression to calculate fracture toughness is only valid when the residual stress field is still present.

(7) The fracture toughness measured by the IF and the IS method depends on the indentation load.

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Table 1 The means and the standard deviations data of the fracture toughness measured by SEPB method for GPSSB and ZAC. Common CHSs are 0.005 and 1.0 mm/min. Other speeds are shown in parentheses.

$MPam^{1/2}$

Materials		Si_3N_4			ZAC		
CHS mm/min		0.005	1.0	others	0.005	1.0	others
PARTICIPANTS NO.	1	5.50±.276	5.64±.159		5.31±.106	6.37±.312	
	2	5.74±.24	5.44±.39		5.71±.21	5.81±.14	
	3	4.94±.41	5.16±.27		5.29±.57	5.65±.39	
	4		5.62±.13	(0.05) 5.42±.09		6.05±.34	(0.05) 6.20±.49
	5		5.62±.235	(0.01) 5.91±.313		6.10±.07	
	6		[1] 6.84±.31			[1] 7.30±.64	
	7	5.61±.159		(0.1) 5.95±.207	5.52±.130		(0.1) 6.40±.158
	8	5.61±.41	5.66±.24		5.31±.15	6.17±.22	
	9		6.42±.19	(0.08) 6.30±.08		6.56±.17	(0.08) 6.19±.12
	10	5.54±.12	5.88±.12		4.99±.27	6.27±.18	
	11	5.54±.19	5.47±.15		5.49±.18	6.29±.10	
	12	5.51±.75		(0.5) 7.11±1.88	5.22±.92		(0.5) 6.60±1.34
	13		[2] 5.72±.25	(0.05) [3] 5.70±.16		[2] 6.12±.12	(0.05) [4] 5.81±.17

[1] Span is 15mm

[2] 7 specimens

[3] 8 specimens

[4] 6 specimens

Table 2 The means and the standard deviations data of the fracture toughness measured by IF method for GPSSN.
(1): Calculation by formula of Miyoshi et al.
(2): Calculation by formula of Marshall et al.

$MPam^{1/2}$

Indented Load (N)		98		196		294	
Used equations		(1)	(2)	(1)	(2)	(1)	(2)
PARTICIPANTS NO.	1	5.42±.127	5.92±.37	5.37±.175	5.86±.182		
	2	5.42±.28	5.90±.31	5.48±.20	5.96±.21		
	3	5.28±.15	5.73±.15	5.51±.12	5.98±.13		
	4	5.40±.124	5.86±.132	5.41±.090	5.87±.098		
	5	7.70±.276	8.29±.313	6.63±.327	7.17±.368		
	6	7.49±.749	8.10±.811	6.36±.417	6.88±.441		
	7	5.47±.167	5.93±.177	5.53±.303	5.94±.283		
	8			6.19±.24	6.70±.25	5.76±.16	6.23±.17
	9			5.93±.163	6.43±.177	5.70±.16	6.18±.17
	10			5.73±.19	6.21±.20	5.40±.07	5.85±.07
	11			5.70±.14	6.18±.15	5.65±.11	6.12±.12
	12	5.41±.17	5.89±.21	5.14±.11	5.60±.12		
	13	5.39±.08	5.86±.08				

Table 3 The means and the standard deviations data of the fracture toughness measured by IF method for ZAC.

(1): Calculation by formula of Miyoshi et al.

(2): Calculation by formula of Marshall et al.

MPam$^{1/2}$

Indented Load (N)		294		490	
Used equations		(1)	(2)	(1)	(2)
PARTICIPANTS NO.	1	6.51±.253	7.04±.281	6.10±.203	6.61±.223
	2	6.02±.18	6.52±.18	5.83±.11	6.32±.11
	3	7.18±.27	7.74±.29	7.20±.33	7.76±.36
	4	6.53±.166	7.09±.223	7.47±.355	8.39±.459
	5	9.15±.515	9.89±.566	8.19±.716	8.84±.773
	6	8.64±.887	9.28±.930	7.68±.136	8.23±.155
	7	7.41±.507	8.02±.549	7.91±.435	8.55±.445
	8	7.89±.48	8.51±.51		
	9	6.24±.06	6.73±.06		
	10	8.61±.17	9.26±.20		
	11	7.18±.35	7.76±.38		
	12	8.19±.40	8.87±.43	7.38±.41	7.95±.46
	13	6.08±.05	6.54±.05	5.86±.05	6.32±.06

Table 4 The means and the standard deviations data of the fracture toughness measured by IS method for GPSSN and ZAC.

$MPam^{1/2}$

Materials		Si_3N_4		ZAC	
Indented Load (N)		49	294	98	490
PARTICIPANTS NO.	1	5.78±.081	6.29±.149	7.00±.094	7.50±.135
	2	5.72±.12	6.31±.11	6.95±.26	7.46±.08
	3	5.61±.11	6.26±.17	6.54±.12	7.10±.08
	4	5.75±.171	6.29±.181	6.56±.101	7.36±.069
	5	5.99±.151		6.54±.180	
	6		6.54±.172		6.69±.113
	7	5.85±.175	6.40±.114	7.28±.165	7.58±.106
	8				
	9				
	10				
	11				
	12	5.99±.14	6.53±.19	7.14±.24	7.96±.29
	13	5.64±.17	6.33±.11	6.62±.18	7.59±.36

Table 5 Summary of the fracture toughness measured by SEPB, IF and IS methods.

	Material GPSSN			Material ZAC		
	SEPB 1.0	IF 196N	IS 294N	SEPB 1.0	IF 294N	IS 490N
Range of mean fracture toughness, $MPam^{1/2}$	5.16 -6.42	5.14 -6.63	6.26 -6.54	5.65 -6.56	6.02 -9.15	6.69 -7.96
Range of std. deviations, ±	0.12 -0.39	0.09 -0.42	0.11 -0.18	0.07 -0.39	0.05 -0.89	0.07 -0.29
Overall mean fracture toughness, $MPam^{1/2}$	5.66	5.75	6.37	6.14	7.36	7.41
Std. dev. of mean, ±	0.35	0.45	0.11	0.28	1.06	0.38
Number of labs.	9	12	8	9	13	8

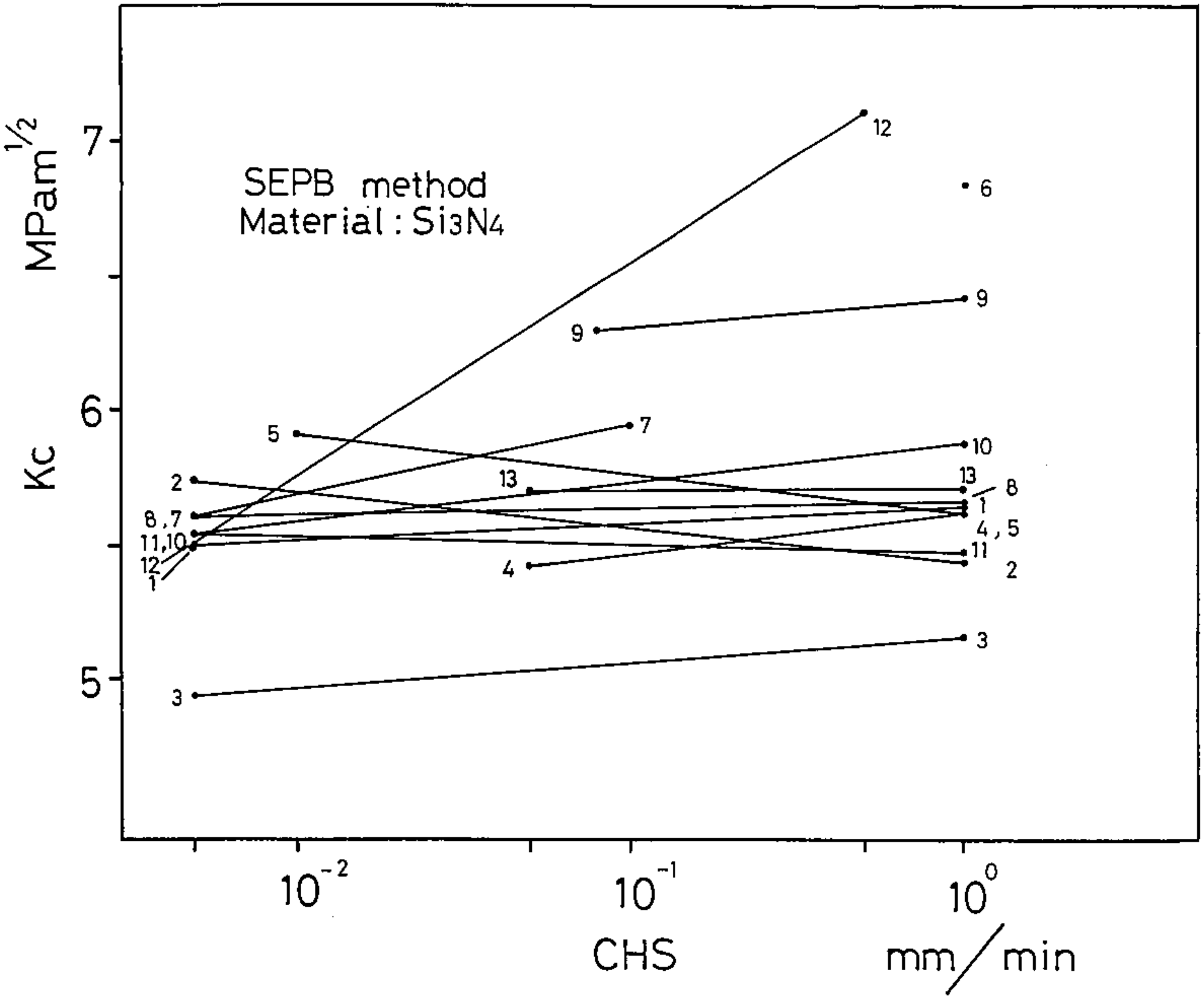

Figure 1 Loading rate dependence of the fracture toughness measured by SEPB method for GPSSN.

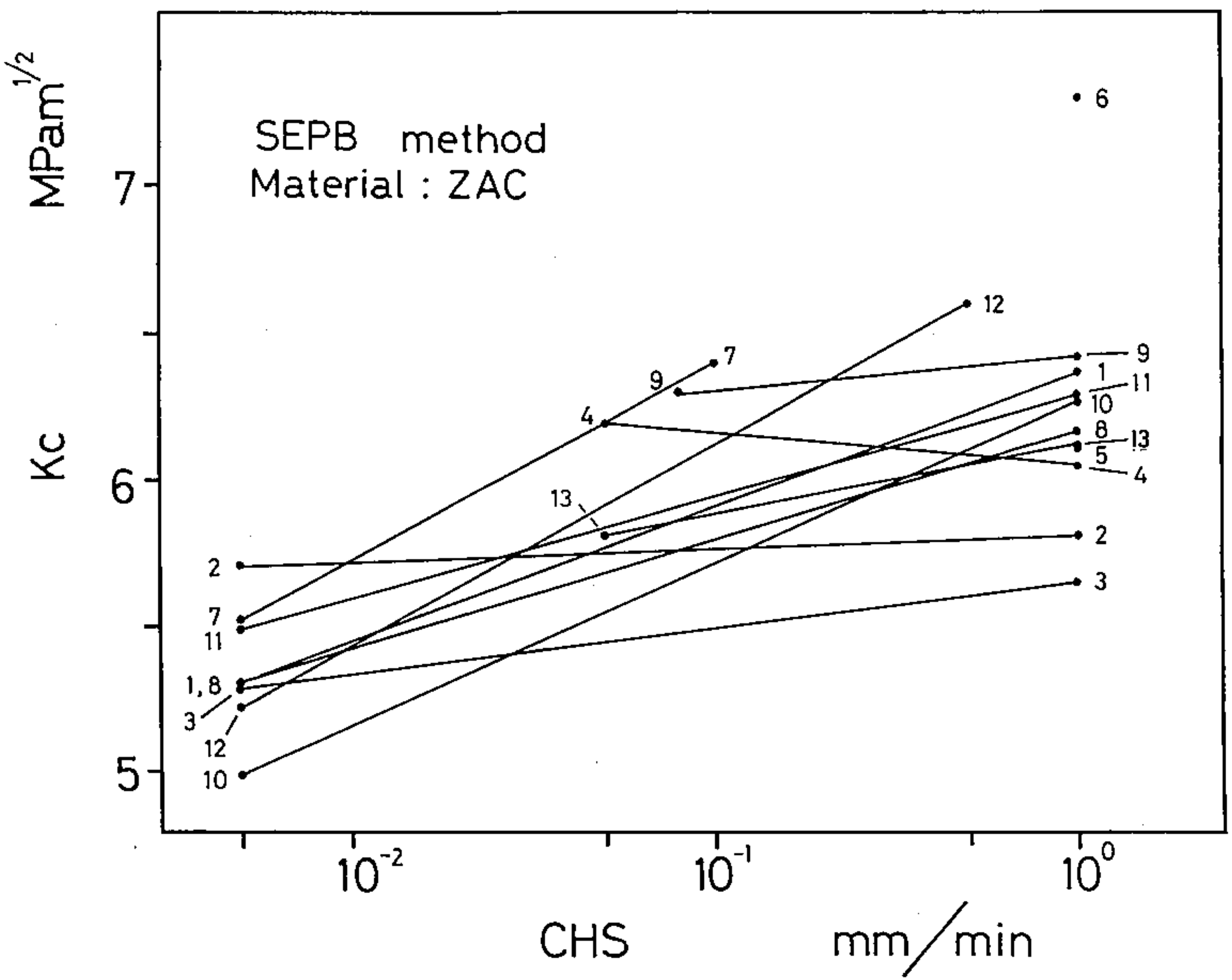

Figure 2 Loading rate dependence of the fracture toughness measured by SEPB method for ZAC.

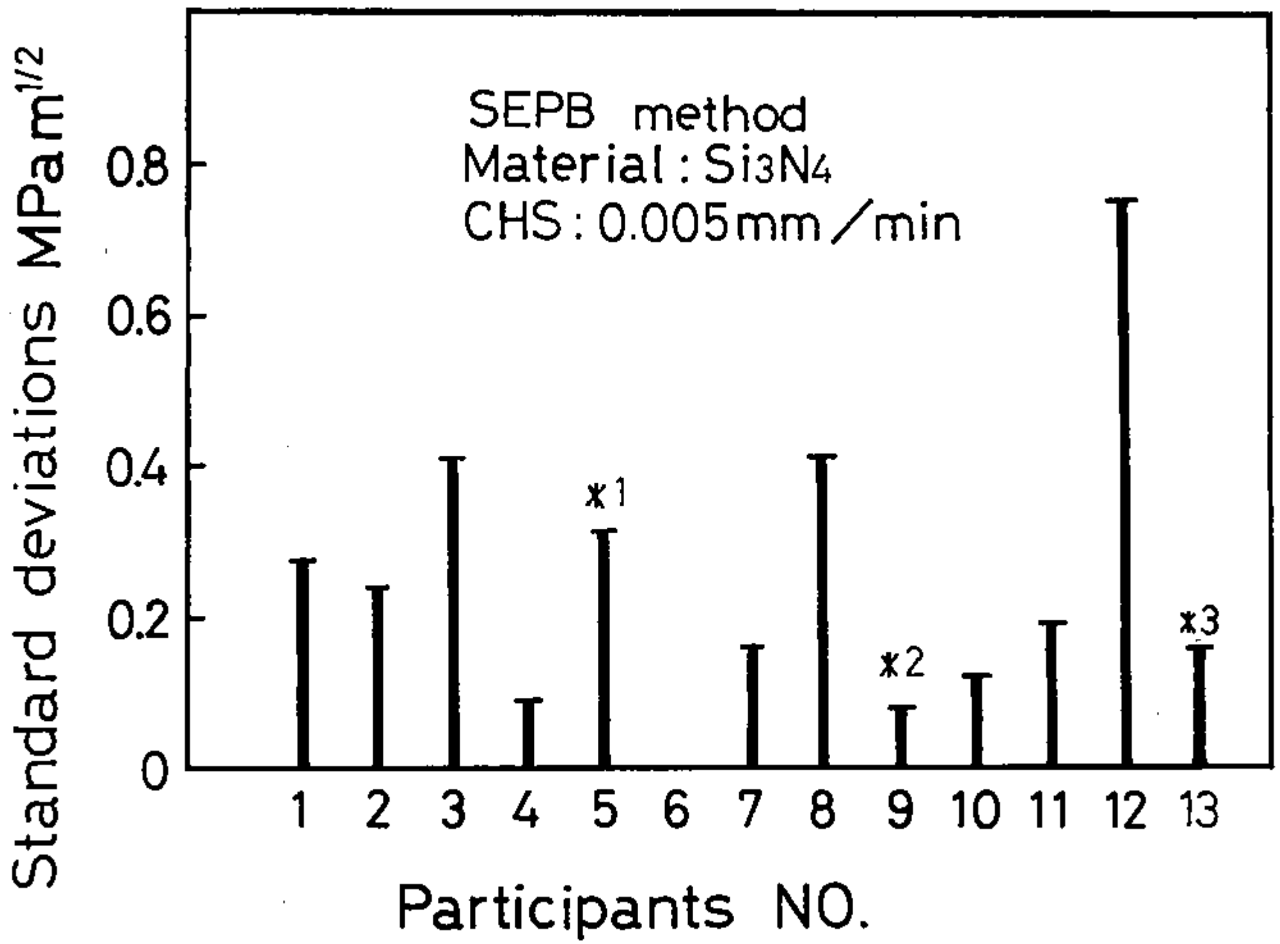

Figure 3 Standard deviations of the fracture toughness measured by SEPB method for GPSSN.

*1: CHS = 0.01 mm/min, *2: CHS = 0.08 mm/min

*3: CHS = 0.05 mm/min and 8 specimens.

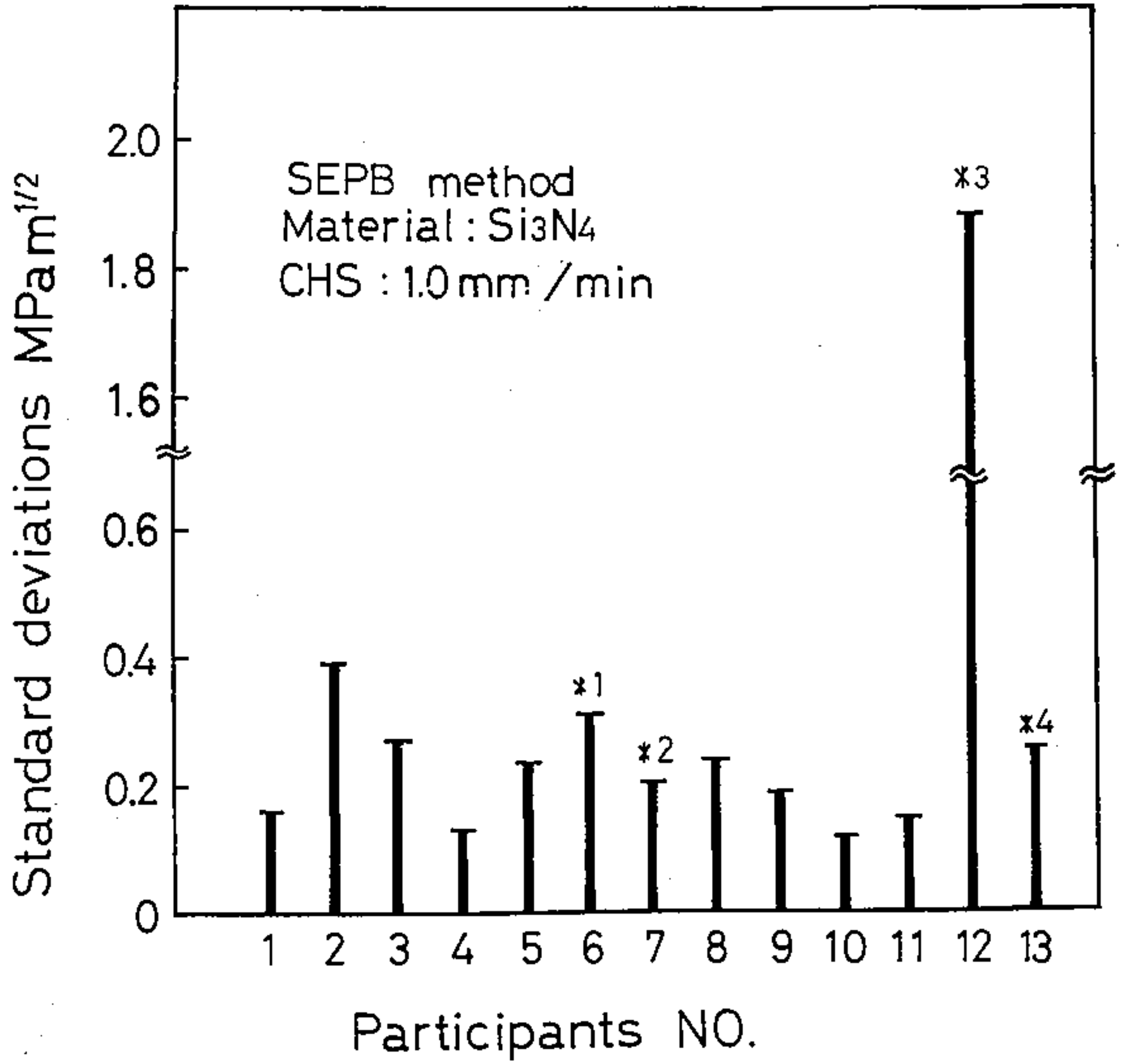

Figure 4 Standard deviations of the fracture toughness measured by SEPB method GPSSN.

*1: Span = 15 mm, *2: CHS = 0.1 mm/min, *3: CHS = 0.5 mm/min

*4: 7 specimens.

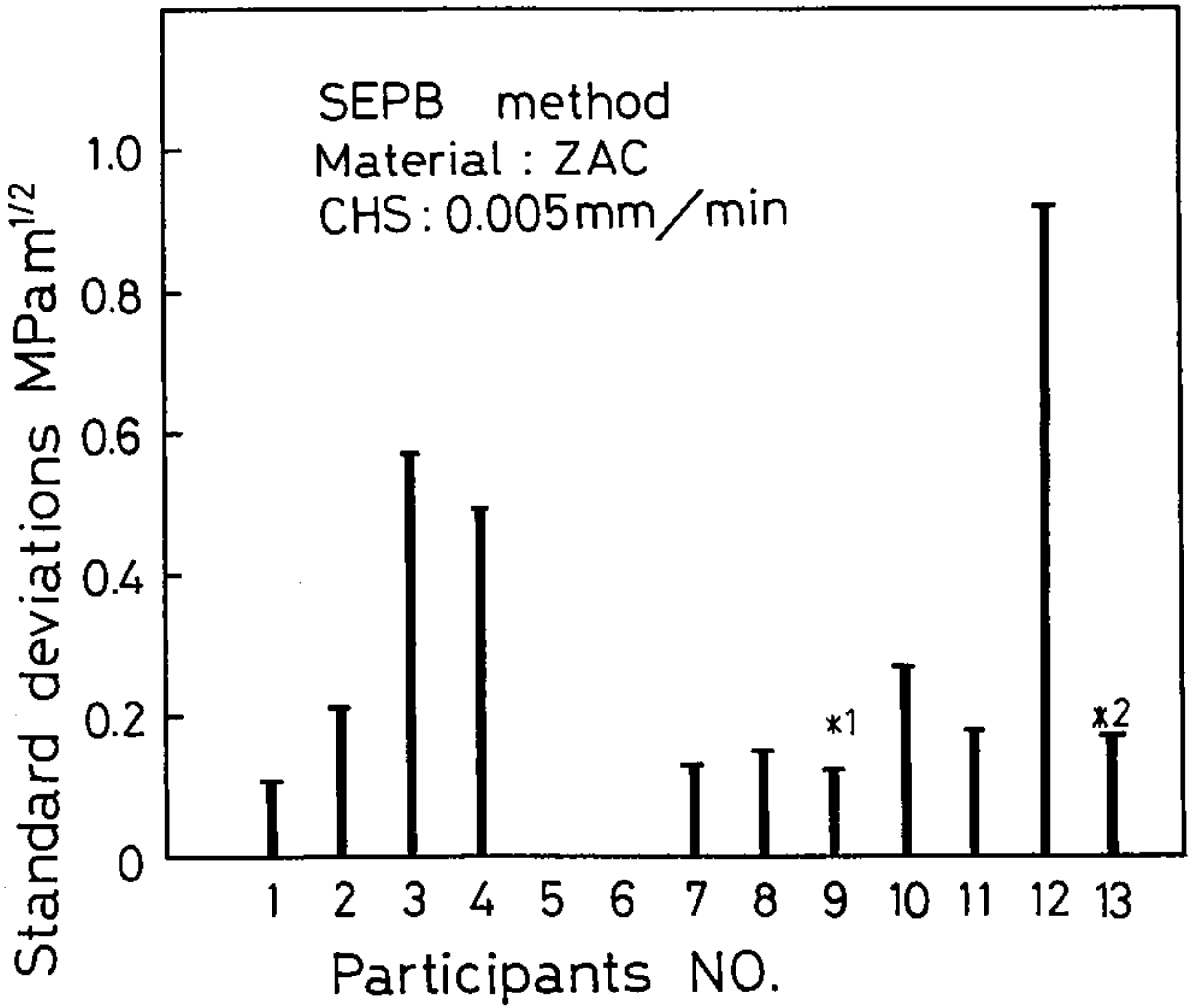

Figure 5 Standard deviations of the fracture toughness measured by SEPB method for ZAC.

*1: CHS = 0.08 mm/min, *2: CHS = 0.05 mm/min and 7 specimens.

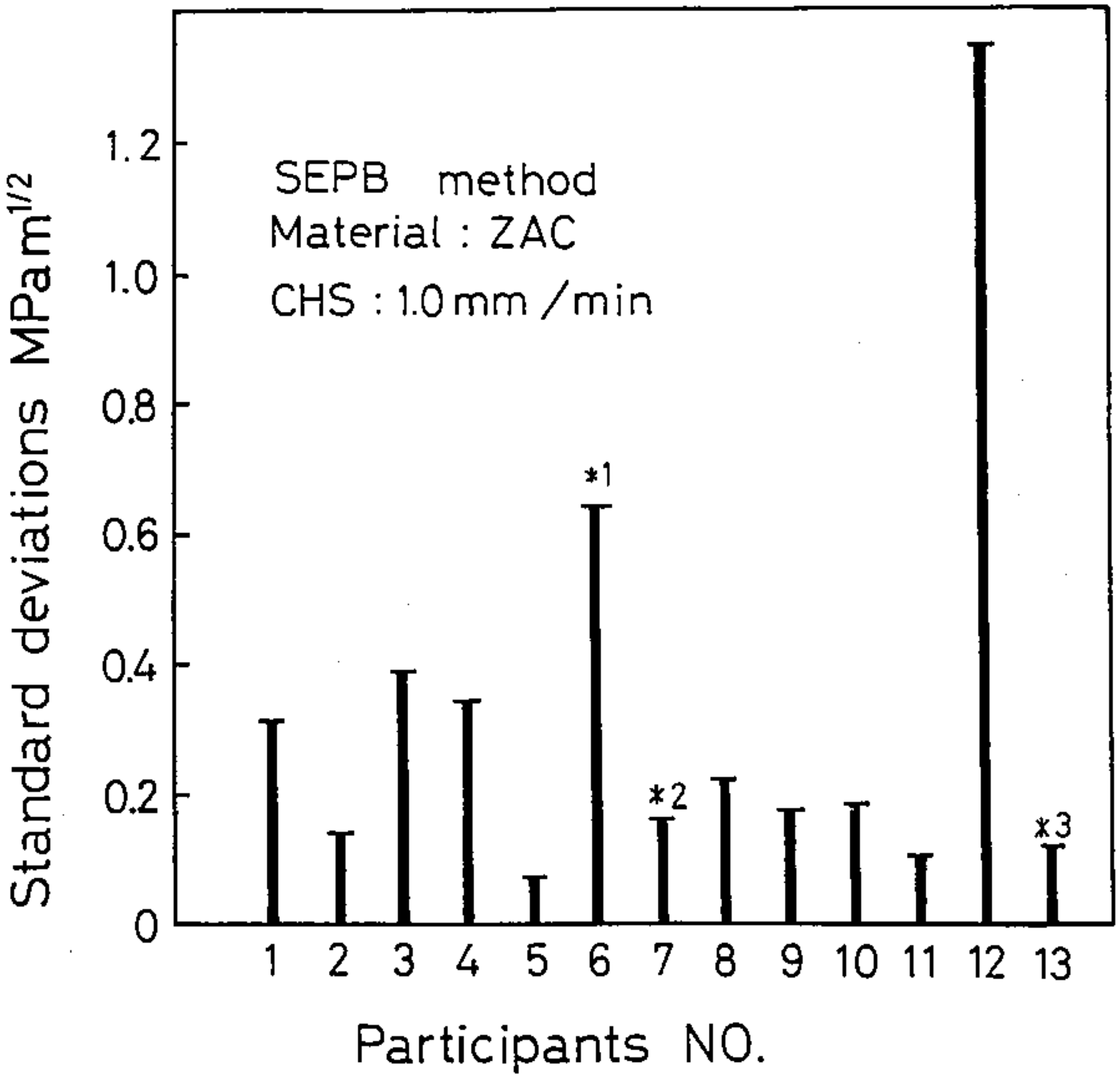

Figure 6 Standard deviations of the fracture toughness measured by SEPB method for ZAC.
*1: Span = 15 mm, *2: CHS = 0.1 mm/min, *3: 7 specimens.

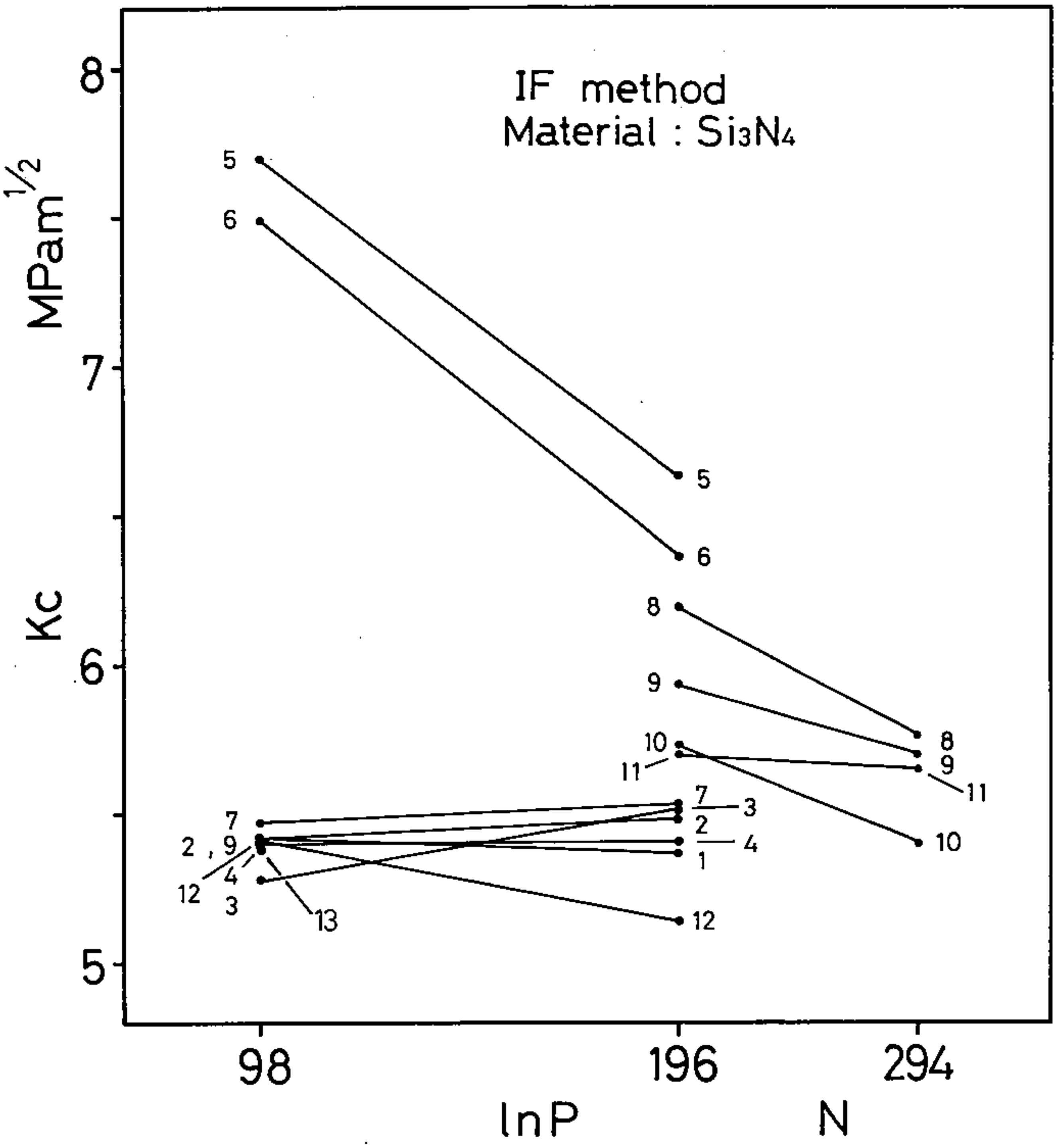

Figure 7 Indentation load dependence of the fracture toughness by IF method for GPSSN.

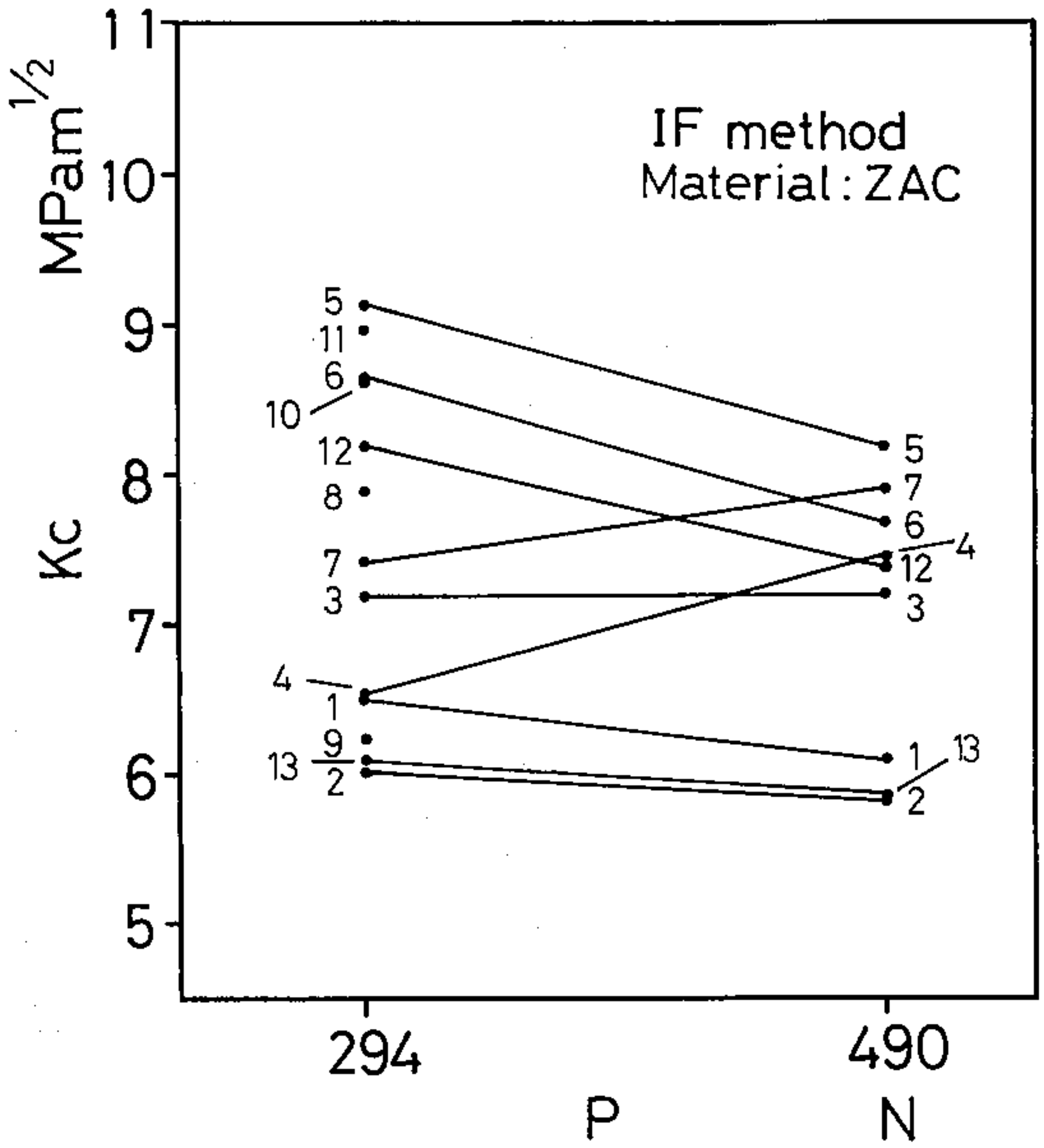

Figure 8 Indentation load dependence of the fracture toughness by IF method for ZAC.

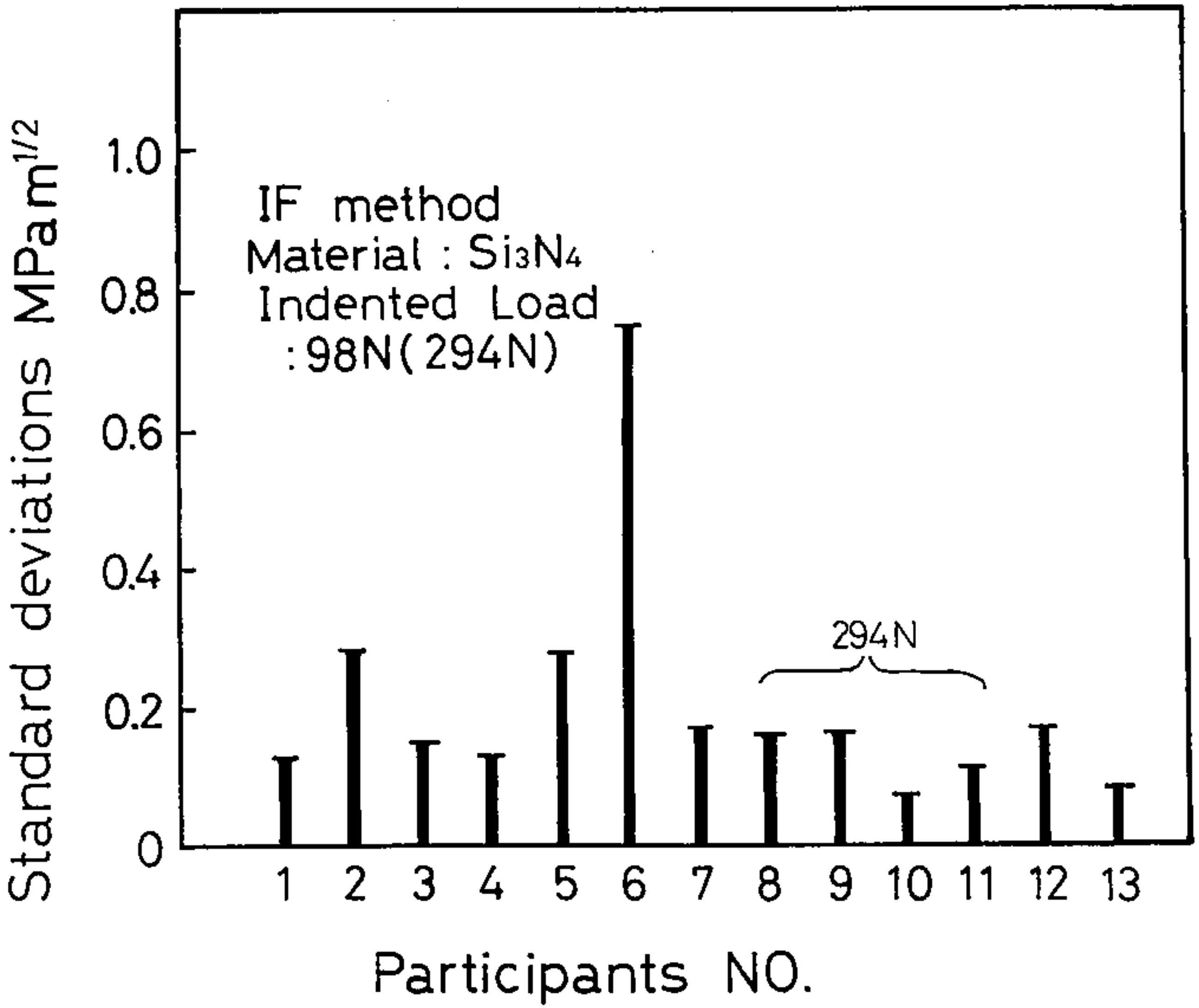

Figure 9 Standard deviations of the fracture toughness measured by IF method for GPSSN.

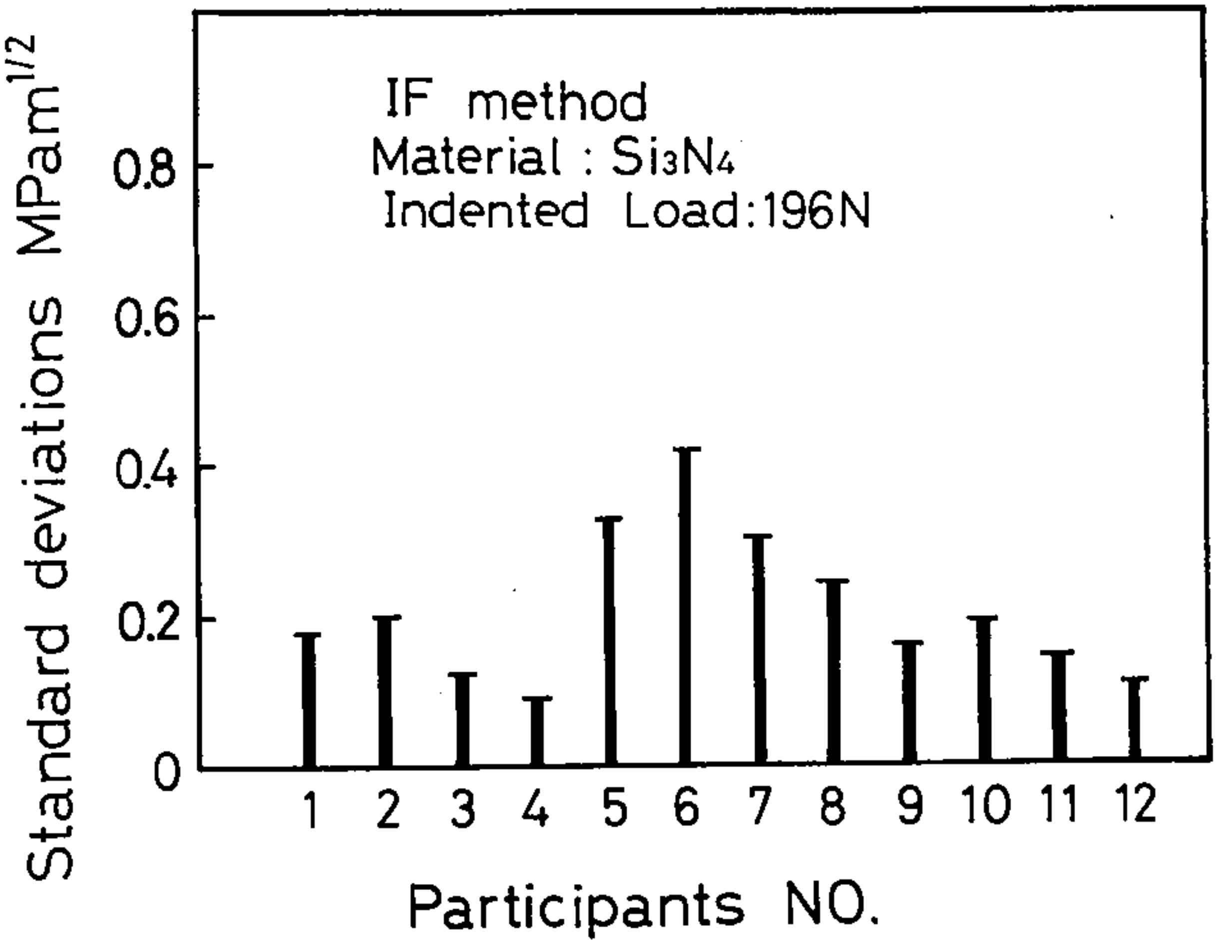

Figure 10 Standard deviations of the fracture toughness measured by IF method for GPSSN.

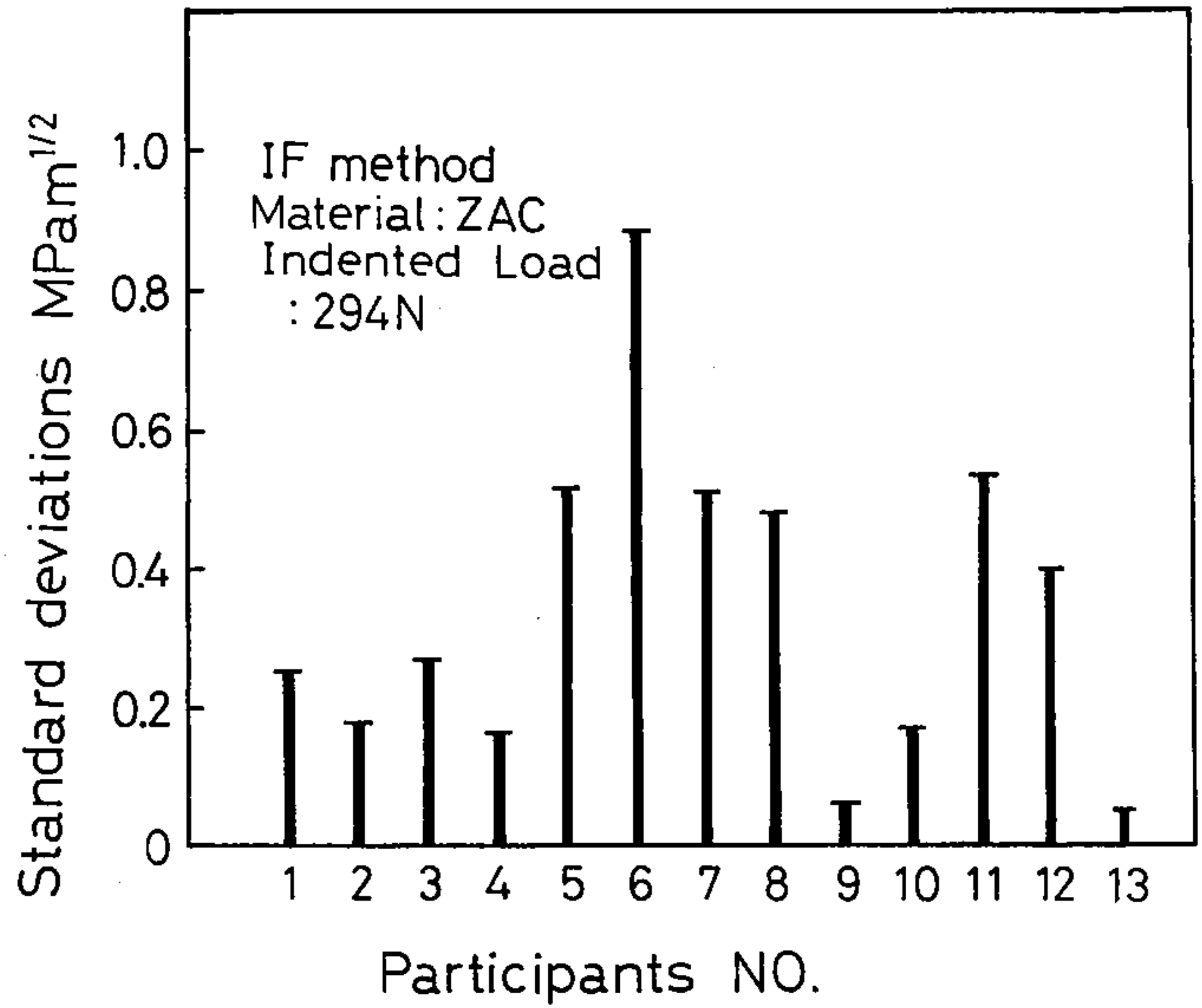

Figure 11 Standard deviations of the fracture toughness measured by IF method for ZAC.

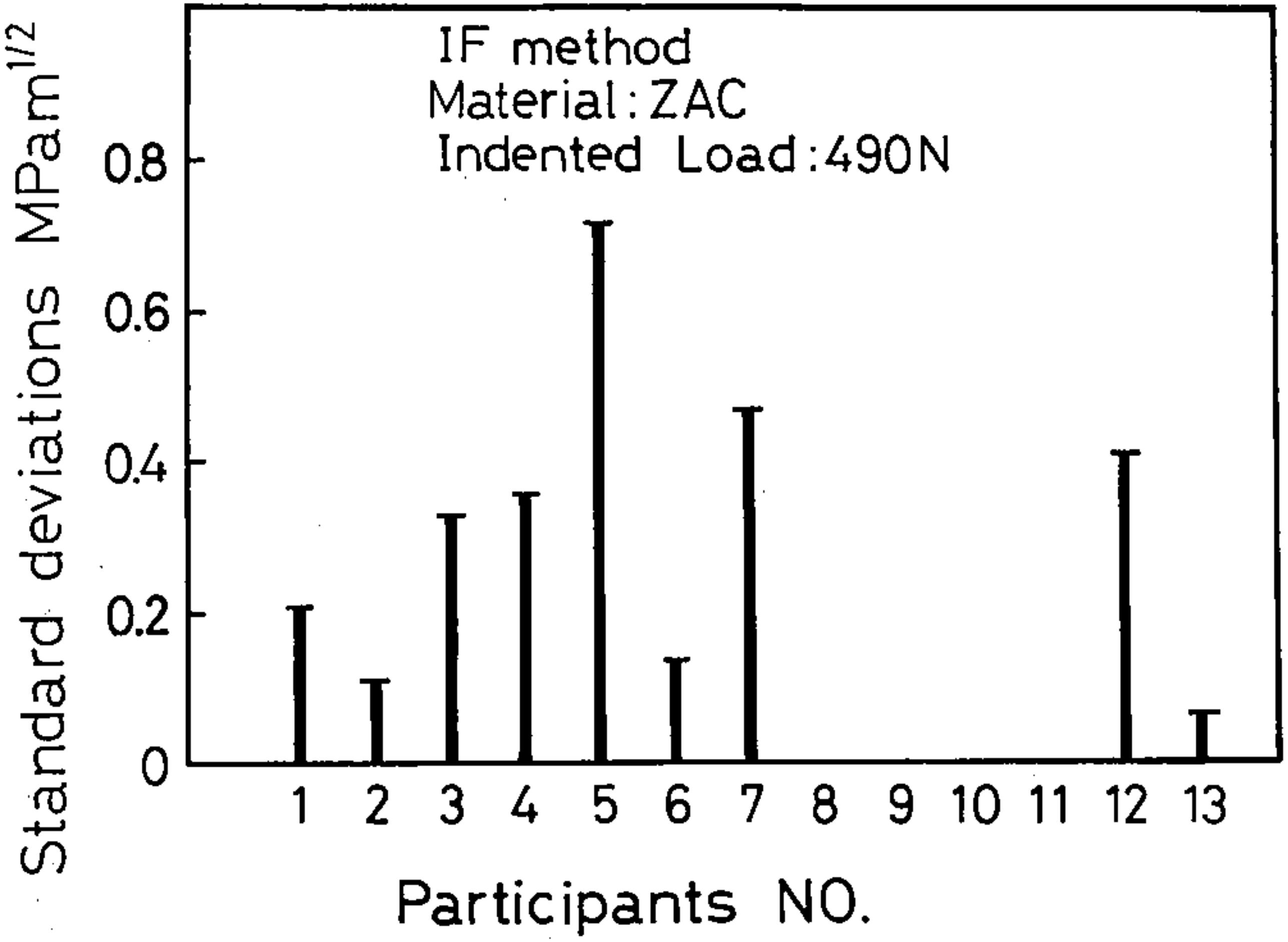

Figure 12 Standard deviations of the fracture toughness measured by IF method for ZAC.

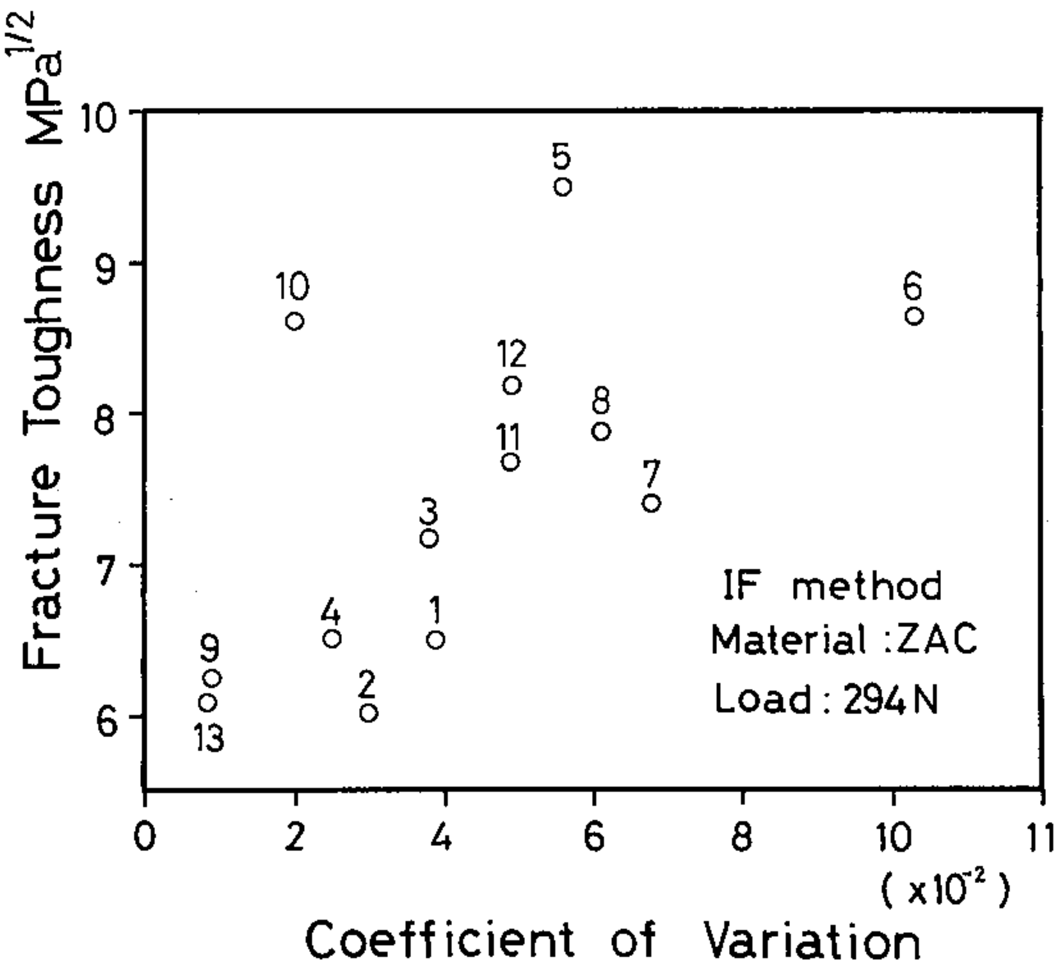

Figure 13 Relation between the mean and the coefficient of variation of the fracture toughness measured by each participant.

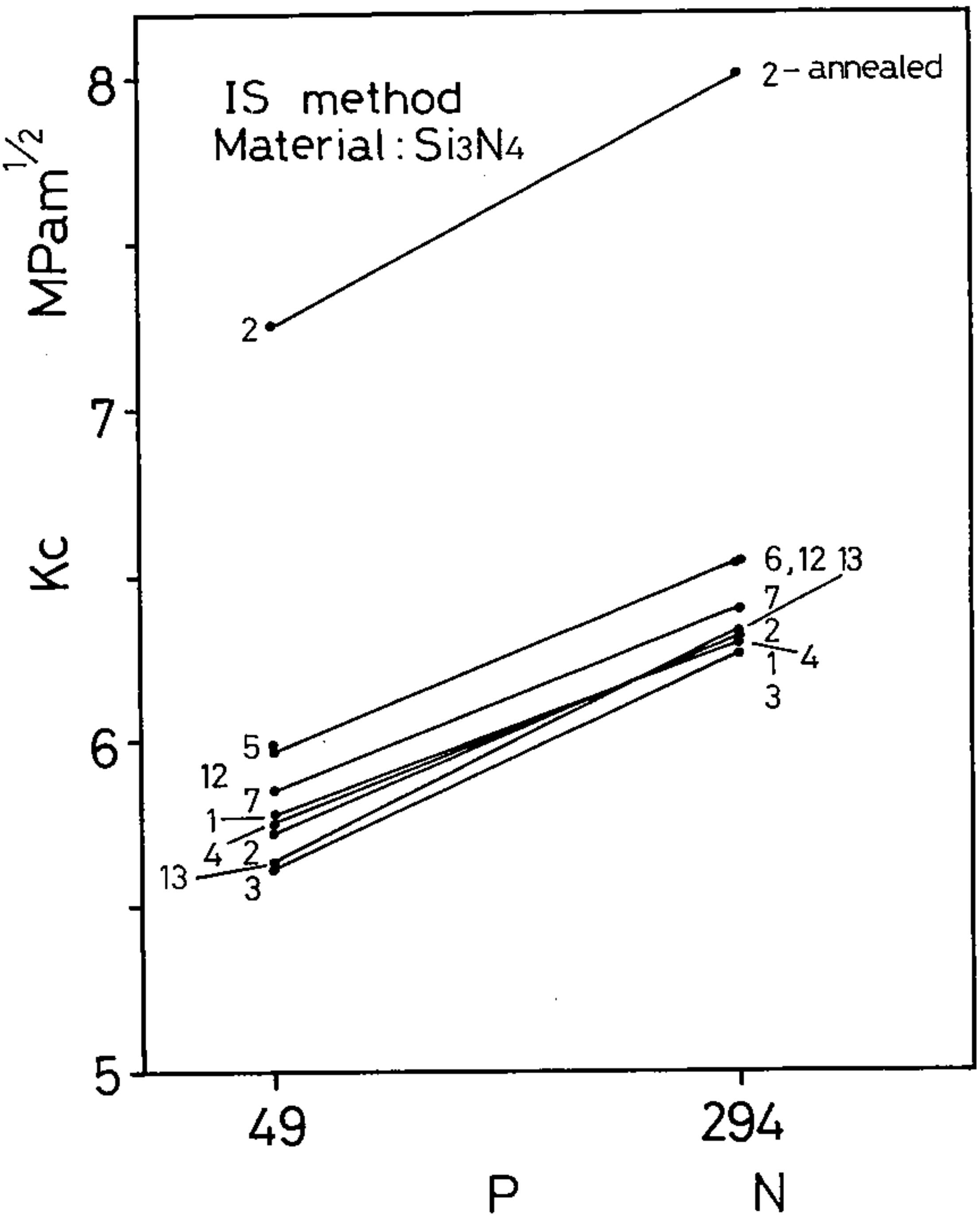

Figure 14 Indentation load dependence of the fracture toughness measured by IS method for GPSSN.

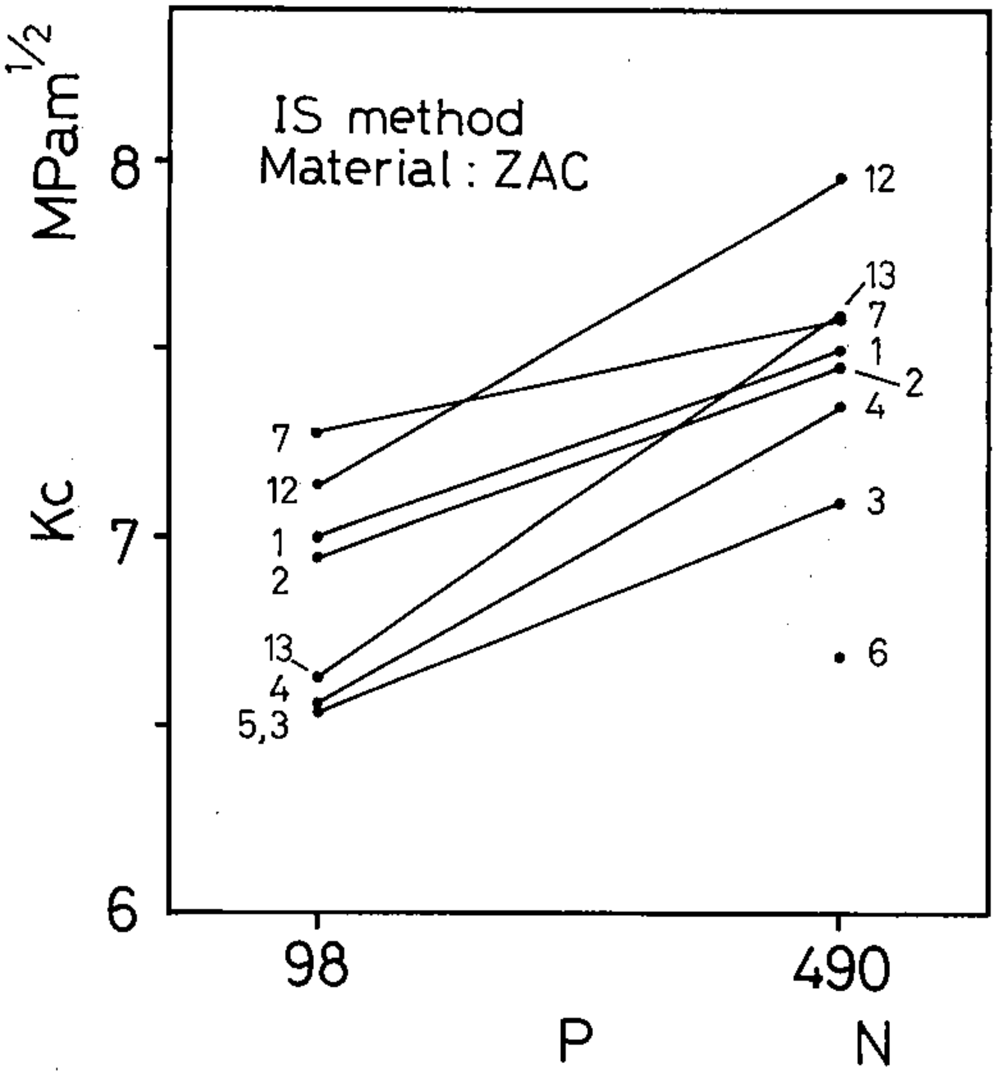

Figure 15 Indentation load dependence of the fracture toughness measured by IS method for ZAC.

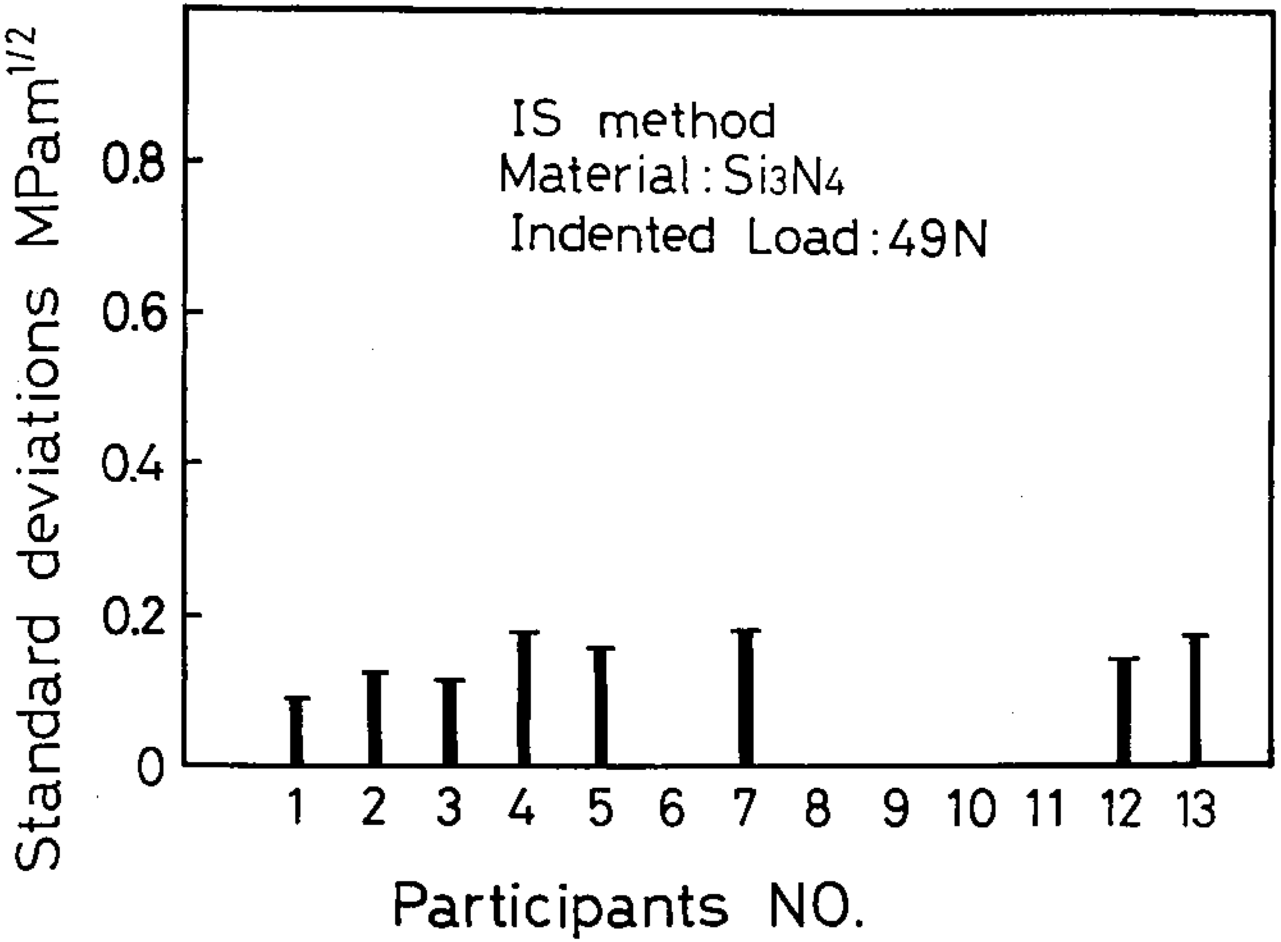

Figure 16 Standard deviations of the fracture toughness measured by IS method for GPSSN.

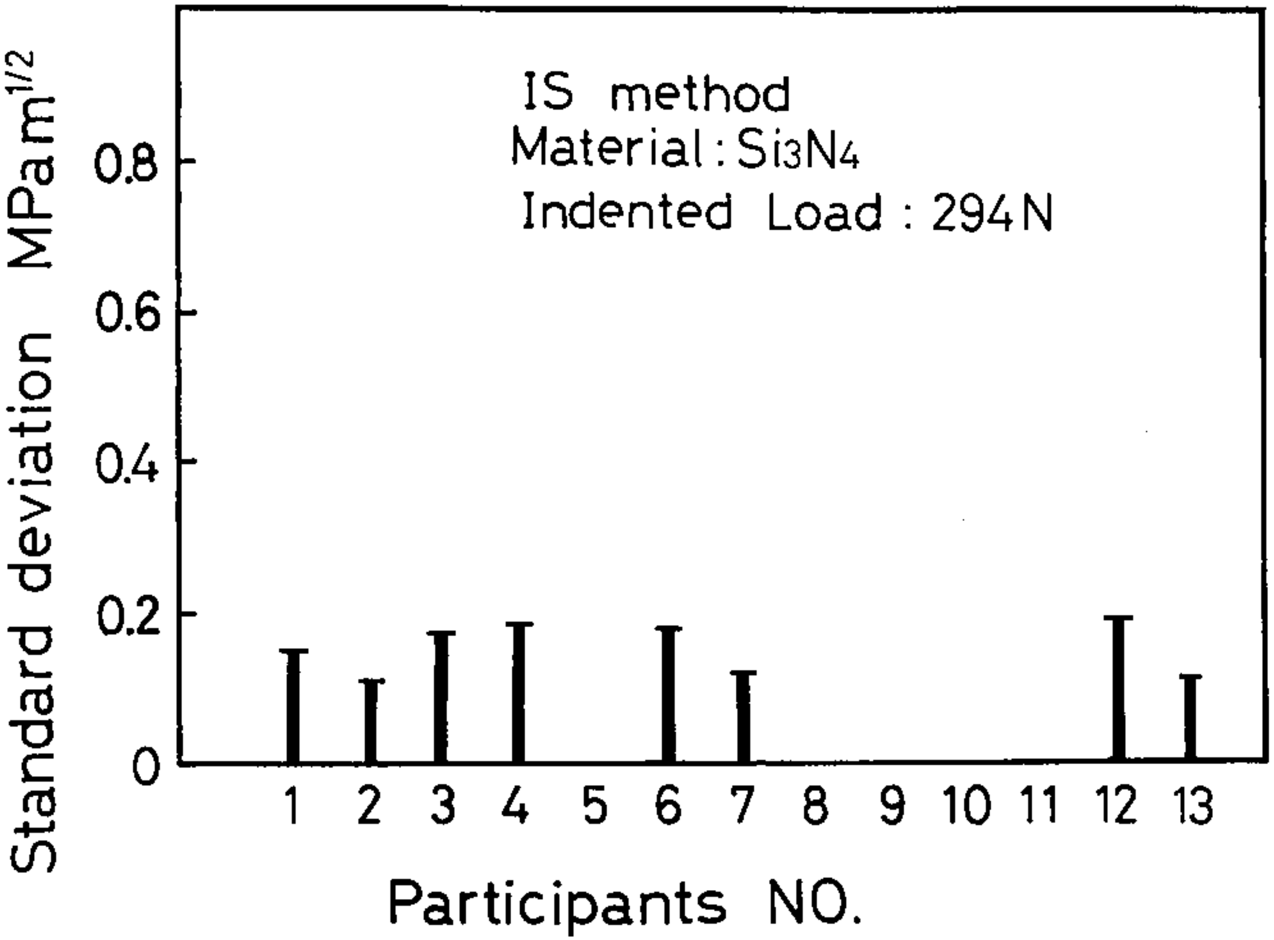

Figure 17 Standard deviations of the fracture toughness measured by IS method for GPSSN.

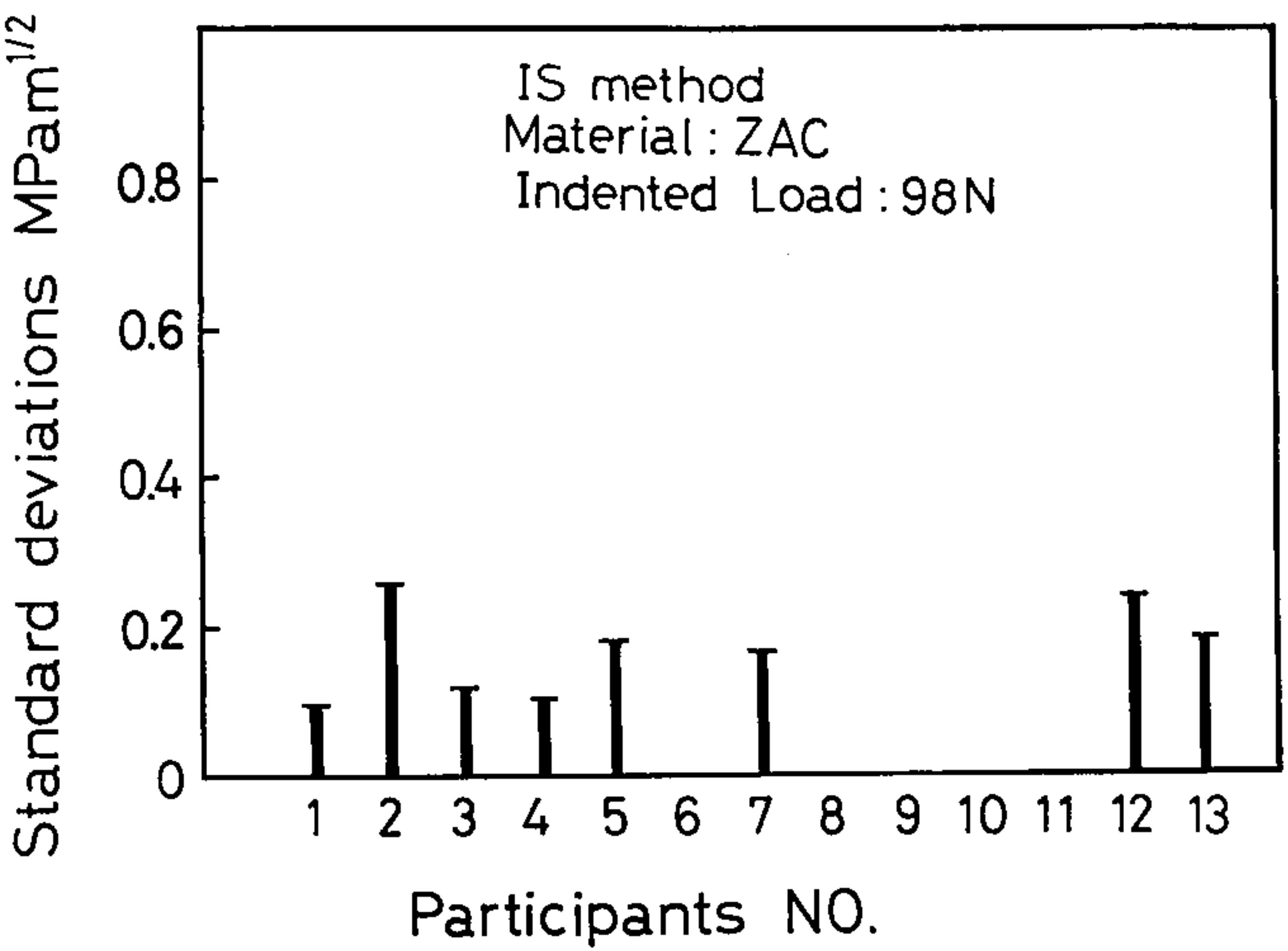

Figure 18 Standard deviations of the fracture toughness measured by IS method for ZAC.

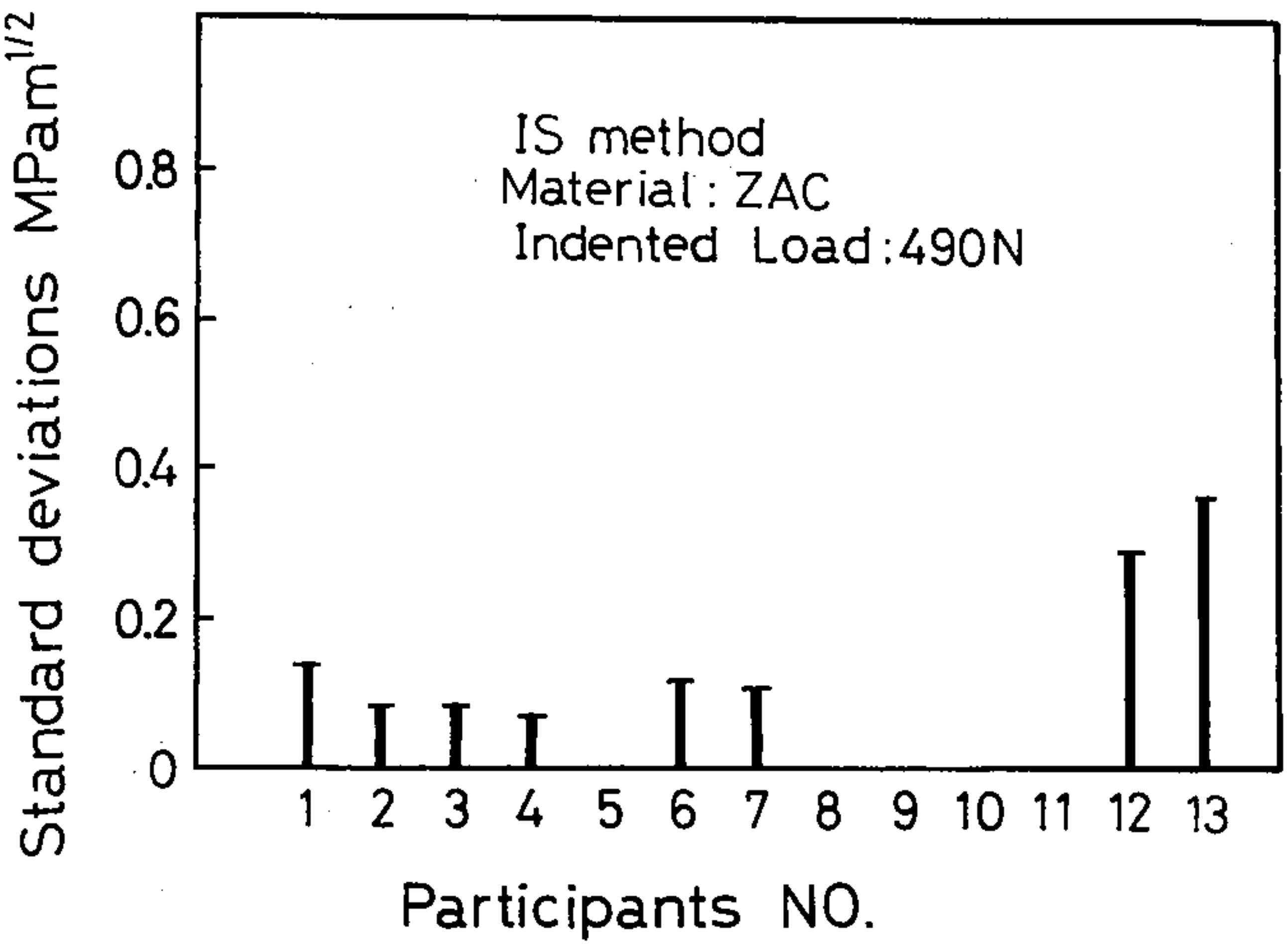

Figure 19 Standard deviations of the fracture toughness measured by IS method for ZAC.

APPENDIX

PROPOSAL OF '89 FRACTURE TOUGHNESS TESTING

Japan Fine Ceramics Center

A. Introduction

Under the auspices of the Ministry of International Trade and Industry in Japan, a committee of investigation and research on standardization of fine ceramics organized by JFCA (Japan Fine Ceramics Association) has been working several years to prepare a standard of fracture toughness measurement. Based on the research, SEPB and IF method are on the recommendation of the standard in Japan. SEPB (Single Edge Pre-cracked Beam) method [1], [2] (or Bridge Indentation method) has several advantages for measuring the parameters of fracture toughness in ceramics. Theoretical simplicity, reproducibility for several materials and long pre-crack are the most important advantages. However, if the pre-crack front is not visible with or without dye penetrant, this method is no use.

IF (Indentation Fracture) method (or Indentation Microfracture method) [3], [4] is a most convenient way to measure a fracture toughness. It needs only small area and simple procedure. This method is particularly useful for routine quality control in individual works. However, there are also several disadvantages. For instance, measuring crack length is difficult in several ceramics. There is apparent load dependency on the measured fracture toughness, and so on.

Another widely spread way in the world is IS (Indentation Strength) method [5]. The most important advantage of the method is that it is not necessary to measure crack length. But its results also show indented load dependency.

The purpose of the '89 Fracture Toughness Round Robin Testing is to assess methods of determining fracture toughness parameters of fine ceramics. The adopted methods for the Round Robin Test are IS method, IF method and SEPB method. Selected materials are silicon nitride and zirconia-alumina composite. In the Round Robin Test, the values of fracture toughness by each method are compared in each materials and in each participant. Also, indented load dependency on the fracture toughness in IS and IF method, and loading rate dependency on the fracture toughness in SEPB method are examined.

B. Materials

Following two kinds of ceramics are used.

Si_3N_4 (NTK, EC-141)

ZAC (NTK, UTZ-20)

Si_3N_4 is a gas pressure sintered silicon nitride. ZAC (Zirconia-Alumina Composite) is a pressure-less sintered zirconia-alumina ceramics. Young's moduli of these ceramics are as follows,

Si_3N_4 : 310 GPa

ZAC : 280 GPa

C. Specimen Distribution

20 specimens per each materials will be sent to the participants. In the three methods , IS method should be done first. IS method will use 20 specimens per each materials. After measuring fracture toughness by IS method, 40 specimens with half length will be available for SEPB method, which test will need each 10 specimens for faster and slower cross-head speed. Also, several spare specimens shall be used for a trial test of SEPB method.

IF method will need only one specimen per each materials. Then one of the used specimens for IS method is appropriated.

D. IS Method

1. Specimens

size : 3x4x40

20 specimens per each materials.

One of the 4mm width-side is mirror finishing surface, which side should be indent of Vickers indentation.

2. Testing Conditions

a. Indent a Vickers indentation on a mirror surface. Loads of the Vickers indentation P are as follows,

Si_3N_4 : 49N or 294N

ZAC : 98N or 490N

Use 10 specimens for each load.

b. Measure Vickers hardness value H.

c. Use three points bending procedure with 30mm span length and 0.5mm/min cross-head speed to get bending strength σ_c. Bending strength are,

$$\sigma_c = \frac{3P_cL}{2WH^2}$$

where

P_c : Maximam load

L : Span length (=30)

W : Specimen width

H : Specimen height

d. Calculate fracture toughness by following equation [5].

$$K_{IC} = 0.59\ (E/H)^{1/8}\ (\sigma_c\ P^{1/3})^{3/4}$$

Where

E : Young's modulus

H : Vickers hardness

σ_c : Bending strength

P : Indented load

e. Full up the results sheet.

E. SEPB Method

1. Specimen

The used specimen for IS method are appropriated for SEPB method. The pre-crack indentation should be indent in the 3mm width surface which surface has finished less than

0.8S grinding.

2. Pre-crack Starter

The SEPB equipment is needful to make a pre-crack. The detailed drawings are in the appendix. There are four kinds of anvil width, namely 3, 4, 5 and 6mm width. The anvil width should be selected so that the pre-crack length is in 1.2 to 2.4mm.

Vickers indentation load is as follows,

For Si_3N_4 : Use one 98N Vickers indentation.

For ZAC : Use three 196N Vickers indentation. The arrangement of the indents are shown in Fig.1.

3. Bearable load on the SEPB equipment

Bearable load on the SEPB equipment is 50kN.

4. Procedure of SEPB Method

(1) A pre-crack starter should be made on the surface of the 3mm width-side of the specimen.

(2) Set a specimen on the loading fixture (SEPB equipment, shown in Fig.2). Increase the load gradually until a pop-in sound is detected by ear or a sonic sensor*.
Then decrease the load immediately.

* We use microsensor AE-900M (NF Electric Instrument, 800KHz, 57dB(700V/m/s)) as a sonic sensor. The sonic sensor is attached on one of the surface of the SEPB equipment by cement (Phenyl Salicylate). The other terminal of the sensor is connected with a oscilloscope terminal directly, which is shown in Fig.3.

(3) A dye penetrant mixed with acetone may be used to improve the visibility of the pre-crack. After penetrant, dry the

specimen at 323K during one hour.

(4) Measure a fracture load, P_c, by three points bending test. Faster cross-head speed (ex. 1mm/min) is used for 10 specimens, and slower cross-head speed (ex. 0.005mm/min) is used for 10 specimens. 16mm span length should be used.

(5) Measure a crack length at three positions as shown in Fig.4. Use the average of these three measurements as the crack length to calculate fracture toughness. The following requirements shall apply to the pre-crack front: (a) The difference between any two of the three crack length measurements shall not exceed 10% of the average [6]. (b) The plane of the crack shall be parallel to both the specimen width and thickness direction within 10%.

(6) Calculate fracture toughness using Srawley's equation [7], as follows,

$$K_I = \frac{3SP}{2BW^2} a^{1/2} F(\alpha)$$

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

$$F(\alpha) = \frac{1.99 - \alpha(1-\alpha)(2.15 - 3.93\alpha + 2.7\alpha^2)}{(1+2\alpha)(1-\alpha)^{3/2}}$$

where

S : Fulcrum distance (=16)

a : Pre-crack length

W : Specimen height

(7) Full up the results sheet.

E. IF Method

1. Specimen

One of the used specimens for IS method is appropriated.

2. Indentation

Indent a Vickers indentation on a mirror surface. Indented loads are 98N and 196N for Si_3N_4, and 294N and 490N for ZAC. Ten indentations should be measured for each load.

3. Calculation

Calculate fracture toughness using the following two equations,

Miyoshi et al [4]

$$K_c = 0.018\ (E/H)^{0.5}\ (P/c^{1.5})$$
$$= 0.0264\ E^{0.5}\ P^{0.5}\ c^{-1.5}\ a \qquad -(1)$$

Marshall and Evans [3]

$$K_c = 0.036\ E^{0.4}\ P^{0.6}\ a^{-0.7}\ (c/a)^{-1.5}$$
$$= 0.036\ E^{0.4}\ P^{0.6}\ a^{0.8}\ c^{-1.5} \qquad -(2)$$

where

E : Young's modulus

P : Indented load

H : Vickers hardness

a : Indentation length (half)

c : Crack length (half)

IF the ratio of the crack length and the indentation length, c/a, is less than 2.3, or if there are some crack blanching, the data should be rejected.

4. Results sheet

full up result sheet.

F. Return Address

After the test, all results should be sent back to the following address until the end of September, 1989.

Hiroshi OKUDA, Dr.
Director of Japan Fine Ceramics Center
2-4-1 Mutsuno, Atsuta-ku
NAGOYA, 456 JAPAN

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[1] T.Nose and T.Fujii; J. Am. Ceram. Soc. 71-5 (1988), P328.

[2] T.Sadahiro; J. Japan Inst. Metals 45-3 (1981), P291 (in Japanese).

[3] D.B.Marshall and R.G.Evans; J. Am. Ceram. Soc., 64-12(1981), c-182.

[4] T.Miyoshi, N.Sagawa and T.Sassa ; Proc. JSME, A1-471(1985), P2489 (in Japanese).

[5] P.Chantikul, G.R.Anstis, B.R.Lawn and D.B.Marshall ; J. Am. Ceram. Soc. 64-9 (1981), P539.

[6] ASTM E399-83.

[7] J.E.Srawley ; ASTM E399 , Int. J. Fracture Mech., 12(1976), P475.

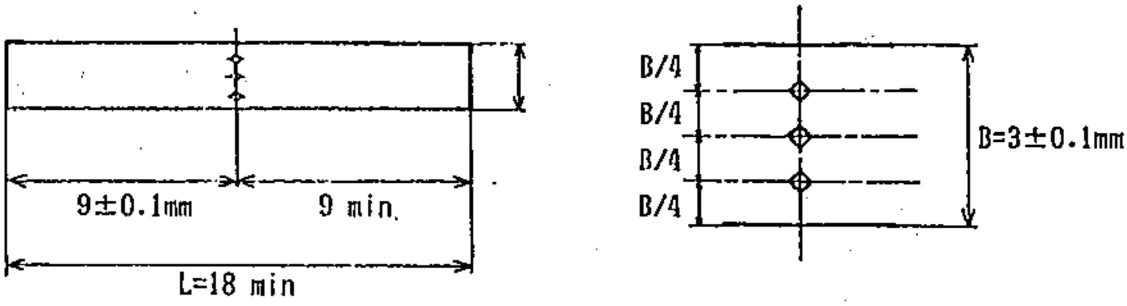

Figure 1 Position of the indentations

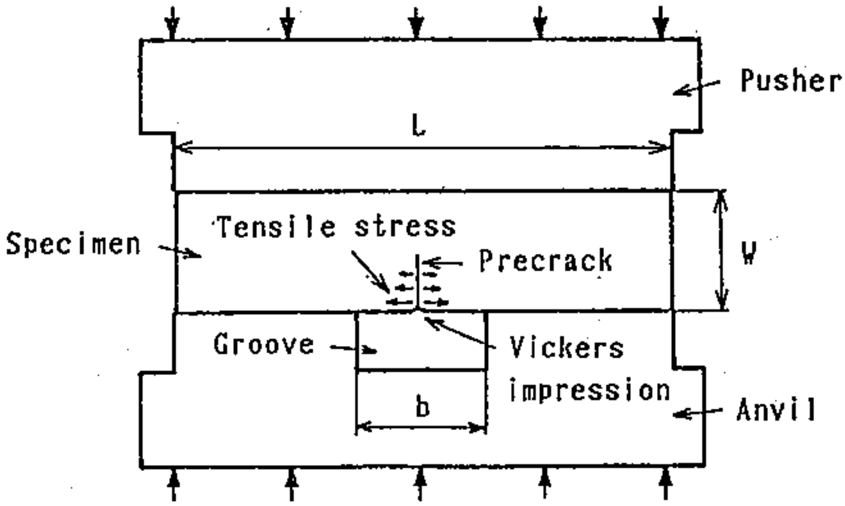

Figure 2 Schematic of SEPB method

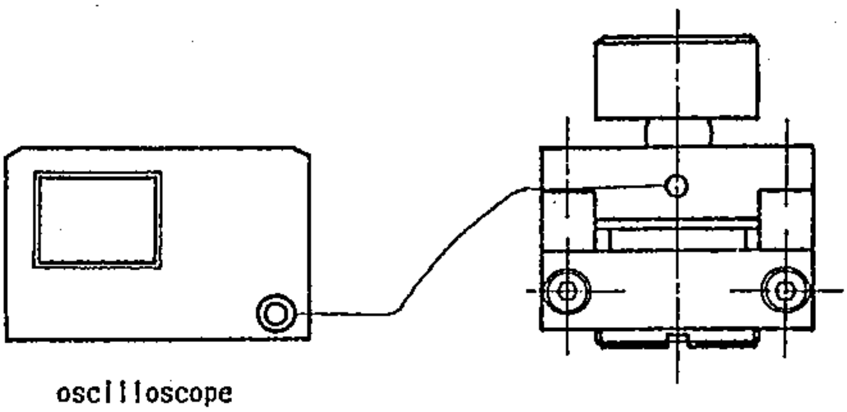

Fig.3

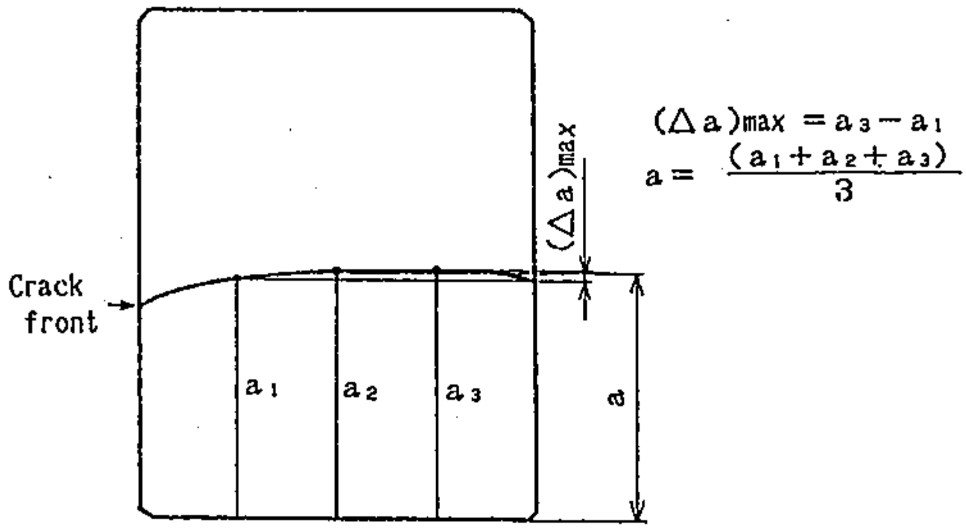

Figure 4 Crack length measurement

IS Method Results Sheet

Materials: ____________________

Young's modulus: ____________________

Test conditions: ____________________

Indented load P ______________ [N]

Cross head speed ______________ [mm/min]

Hardness machine ____________________

Testing machine ____________________

No	W	H	d_1	d_2	d_{mean}	Hv	P_c	σ_c	K_c
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									

Mean ______________

Standerd Deviation ______________

W : Width of the specimen (4mm)
H : Height of the specimen (3mm)
d_1、d_2 : Length of indent
Hv : Vickers hardness
P_c : Maximum load on bending test
σ_c : Bending strength
K_c : Fracture toughness

IF Method Results Sheet

Materials: ______________________

Young's modulus: ____________________

Hardness machine: ____________________

No	P	$2a_1$	$2a_2$	$2a_3$	$2c_1$	$2c_2$	2c	Kc(Eq.1)	Kc(Eq.2)
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									

Mean ______________

Standerd Deviation ______________

P : Indented load
2a : mean of indentation lengths
2c : mean of crack length

Notes : Eq. 1 Miyoshi, sagawa and sassa (1984)
Eq. 2 Marshall and Evans (1981)

SEPB Method Results Sheet

Materials: ____________________

Test conditions:

Cross head speed ____________ [mm/min]

Hardness machine __________________

Testing machine _____________________

Load cell capacity _______________

No	B	W	a_1	a_2	a_3	a	P_c	K_c
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								

Mean ____________

Standerd Deviation ____________

W : Width of the specimen (4mm)

B : Width of the specimen (3mm)

a_1、a_2、a_3 : Pre-crack lengt

Hv : Mean of pre-crack length

P_c : Maximum load on fracture toughness testingng

K_c : Fracture toughness

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