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FRACTURE TOUGHNESS MEASUREMENTS ON ZIRCONIA TOUGHENED CERAMICS

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Abstract. Three techniques for fracture toughness measurements on zirconia toughened ceramics were evaluated: the notched beam (NB) technique, the indentation fracture (IF) technique and the indentation strength in bending (ISB) technique. Using these techniques comparative measurements were performed on samples prepared by pressing (uniaxial) and sintering of four commercially available powder types. These were: Toyo Soda (Japan) powders with the designations TZ3Y (2.86 mole% Y_2O_3), TZ3YA (2.77 mole% Y_2O_3, 0.1 wt.% Al_2O_3) and TZ3Y20A (2.88 mole% Y_2O_3, 20 wt.% Al_2O_3) and a powder supplied by Viking Chemicals (Denmark) designated as YPSZ-2.5 (2.5 mole% Y_2O_3). The measurements showed that similar K_{IC} values were obtained with the IF- and ISB-techniques, which therefore are recommended for K_{IC} measurements. Too high values were, however, obtained with the NB-technique which therefore cannot be recommended. Finally, the measurements showed that a high temperature annealing is recommended prior to testing for the IF-technique.

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CONTENTS

1. INTRODUCTION

Several methods can be used to determine the fracture toughness of ceramic materials. Among some of the most important methods can be mentioned: (1) the notched beam (NB) technique where a pre-notched sample is fractured using the 3- og 4-point bending technique (3,5) - notching is by this method performed by cutting usually with a high speed diamond saw; (2) the indentation fracture (IF) technique by which the fracture toughness is determined from the length of cracks induced around a Vickers indenter (2), and finally (3) the indentation strength in bending (ISB) technique where microcracks are introduced by a Vickers indenter prior to the 3- og 4-point bending measurement of the fracture strength (1). In the present work these three methods of fracture toughness determination are evaluated and compared by measurements on zirconia toughened ceramic samples prepared from two commercial powders delivered from Toya Soda, Japan and Viking Chemicals, Denmark, respectively.

2. EXPERIMENTAL

Rectangular bars (2x6x25 mm) were prepared by pressing and sintering from the following powders:

Toya Soda, Japan	I	TZ3Y	(2.86 mole% Y_2O_3)
" " "	II	TZ3YA	(2.77 mole% Y_2O_3, 0.1 wt.% Al_2O_3)
" " "	III	TZ3Y20A	(2.88 mole% Y_2O_3, 20 wt.% Al_2O_3)
Viking Chem., DK	IV	YPSZ-2.5	(2.5 mole% Y_2O_3).

All samples were sintered to a density higher than 95% TD.

2.1. Notched beam (NB) measurements

The sintered samples were notched with a diamond saw producing a notch width of about 0.43 mm. The exact width and depth of the notch were measured with an optical microscope equipped with a micrometer eyepiece. The stress intensity factor, i.e. the K_{Ic} values, were determined for samples with different notch depths using the 4-point bending technique. These measurements were performed with 10 mm and 20 mm inner and outer spans, respectively, and with a crosshead speed 0.3 mm/min. The K_{Ic} values were calculated using the following empirical equation (3,5):

$$K_{Ic} = 3\ P_f\ La^{1/2}/4bh^2\ (1.99-2.47(a/h) + 12.97(a/h)^2 - 23.17(a/h)^3 + 24.80(a/h)^4) \qquad (1)$$

Here P_f is the load at fracture whereas the meaning of L, a, h and b are given in Fig. 1, which shows the geometry of the notched samples.

2.2. Indentation fracture (IF) measurements

The samples were polished to nearly mirror finish (diamond paste down to 3 µm-1 µm has been recommended in the literature) prior to the Vickers indentations, which were carried out with different loads in the range 50-500 N. In order to minimize slow crack growth a small drop of silicone oil was placed over the impression immediately after unloading.

The crack radius, c, the indentar diameter, 2a, and the crack length l, are specified in Fig. 2. Average figures of these parameters were determined from ten indentations with the same load in reflected polarized light using an optical microscope. The stress intensity factor, K_{Ic}, was then calculated from the following equations, depending on the c/a ratio:

(1) For $c/a > 2.5$ - Young's modulus not known (2)

$$K_{Ic}\ \Phi/H\ a^{1/2} = 0.15\ k(c/a)^{-3/2} \tag{2}$$

where Φ is the constraint factor $\simeq 3.2$ and H is the hardness. The accuracy obtained with this equation is about 30%.

(2) For $c/a > 2.5$ - Young's modulus known (7)

$$(K_{Ic}\ \Phi/H\ a^{1/2})\cdot(H/E\Phi)^{0.4} = 0.129(c/a)^{-3/2} \tag{3}$$

where E is Young's modulus. The accuracy obtained by this equation is about 10%.

(3) For $c/a < 2.5$ - Young's modulus known (6)

$$(K_{Ic}\ \Phi/H\ a^{1/2})\cdot(H/E\Phi)^{0.4} = 0.035(l/a)^{1/2} \tag{4}$$

where the radio $l/a = (c/a)-1$.

Finally, by replacing H in Eq. 2 with the empirical Vicker's hardness relation:

$$H = 0.47\ P/a^2 \tag{5}$$

where P is the load and a the half of the indenter diagonal, the following simple equation for K_{Ic} can be obtained:

$$K_{Ic} = 0.0726\ Pc^{-3/2} \tag{6}$$

This equation can be generally used for fracture toughness measurements on ceramics. The accuracy using this equation will also be about 30% as for Eq. 2.

2.3. Indentation strength in bending (ISB) measurements

The sintered samples were indented using Vickers pyramid with different loads. Each sample was indented (only one indentation) at the center of the tensile surface. Immediately after unloading a drop of silicone oil was placed on the indentation site. The samples were then fractured by the 4-point bending technique, the fracture strength, σ_f, was calculated using the equation:

$$\sigma_f = 3P_f\ L/4\ b\ h^2 \tag{7}$$

(P_f, L, b and h are defined in section 2.1.). The corresponding K_{Ic} values were calculated from (1):

$$K_{Ic} = \eta(E/H)^{1/8}\ (\sigma_f \cdot P_i^{1/3})^{3/4} \tag{8}$$

Here E is Young's modulus, H is the hardness, P_i the indentation load and $\eta = 0.59 \pm 0.12$.

If the ratio E/H is not known the product of $\eta(E/H)^{1/8}$ can be replaced by an average value of 0.88. This gives the equation:

$$K_{Ic} = 0.88\ (\sigma_f \cdot P_i^{1/3})^{3/4} \tag{9}$$

which can be used for "well behaved" materials with an accuracy comparable that obtained by the IF-technique (1).

3. RESULTS AND DISCUSSION

3.1. Notched beam (NB) technique

The K_{Ic} values determined by the NB-technique are shown in Fig. 3 as a function of the a/h (notch depth/specimen thickness) ratio. As seen from this figure this technique can give consistent K_{Ic}-values although with some scatter up to an a/h ratio of about 0.36, whereas a large decrease in K_{Ic} is observed at larger a/h-ratios. The reason for the scatter observed at low a/h-ratios could be variations in the notch radius (5), non-uniform notch depth or the rather large notch width (0.43 mm) used in this work. In order to get more accurate data by this technique a better and more reproducible notching technique should thus be developed. It should also be noted here that all samples were annealed at 1450°C before these measurements.

3.2. Indentation fracture (IF) technique

Fig. 4 shows the regular cracking pattern formed by Vickers indentation at a load of 300N on a polished sintered sample (4a) and a polished and then annealed sintered sample (4b) of the material TZ3Y20A. Annealing was also in this case performed at 1450°C. Larger cracks are apparently formed in the as-polished state than when the polished sample is annealed before the measurements. This technique is thus very sensitive to surface stresses created by polishing and in order to get a reliable measurement the polished samples should be annealed before the indentation. This is also clear from Fig. 8, which snows that while the K_{Ic} value in the as-polished state is only about 4 $MN/m^{3/2}$ this value increases up to about 5.8 $MN/m^{3/2}$ by annealing.

Fig. 5 shows the relation between log a (a = half indent diagonal) and log P_i(P_i = indent load) obtained for TZ3Y20A samples. The straight line relationship obtained in this plot indicate that the Vicker's hardness is nearly constant for this material over the load range used. Using Eq. 6 a hardness of 13.635 GPa can be calculated from this line.

Finally, the K_{Ic} values obtained by using Eqs 4 and 6 are compared in Table I. Except for TZ3Y20A the more simple Eq. 6 consistently gives only slightly smaller K_{Ic} values than Eq. 4. The reason for the discrepancy observed for TZ3Y20A could be that the E-values used in the calculation of K_{Ic} according to Eq. 4 were slightly overestimated - for this material the E-value specified by the supplier was used. Considering the uncertainties involved in these measurements, Eq. 6 is therefore believed to be generally acceptable for K_{Ic} calculations from IF-data.

3.3. Indentation strength in bending (ISB) technique

According to Eq. 9 the product $\sigma_f \cdot P_i^{1/3}$, which is proportional to K_{Ic}, should be constant and independent of the load, P_i, used in the Vickers indentation. This was also found in the present work as shown in Fig. 6 indicating that reproducible results can also be obtained with this technique, which is more simple to perform than the two other techniques. Finally, the SEM picture of a fracture surface shown in Fig. 7 shows that the crack start from the indentation site as it should by this technique.

3.4. Comparison between techniques

The K_{Ic} values obtained by the three techniques are presented in Table I. Furthermore the K_{Ic} values determined by the IF-and ISB-techniques, respectively, for TZ3Y20A are shown in Fig. 8, from which it will be noted that these two techniques give similar results if the IF measurements are carried out on polished and annealed samles. This is also evident from Table I for the three Japanese powders (I, II and III) whereas a somewhat higher K_{IC} value is obtained with the ISB - than with the IF-technique for YPSZ-2.5 (IV - Viking Chemical). The reason for this difference in behaviour between the two powder types is not clear. Finally, it will also be noted from Table I that the NB-technique gives much higher K_{IC} values than the other two techniques. This is in accordance with other investigations (8), which have shown that K_{IC} values obtained with this technique are overestimated because of transformations induced by the notching. Thus the NB-technique apparently is not suitable for K_{IC} measurements. This technique could eventually be improved, however, by using a better notching technique.

4. CONCLUSIONS

Comparative measurements of K_{IC} values were carried out on zirconia toughened ceramics using three techniques: the notched beam (NB) technique, the indentation fracture (IF) technique, and finally the indentation strength in bending (ISB) technique. These measurements showed that similar values can be obtained with the IF- and ISB-techniques, whereas too high K_{IC} values will be determined by the NB-technique. The IF- and ISB-techniques are thus recommended for K_{IC} measurements. In the case of the IF-technique it was further found that a high temperature annealing of the polished samples is necessary prior to testing as this technique is very sensitive towards the surface stresses created by polishing.

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Tabel 1. K_{Ic} values measured by IF-, ISB- and NB-techniques.

Material	IF (300 N) (Eq. 4)	IF (300 N) (Eq. 6)	ISB (Eq. 9)	NB (Eg. 1)
I - TZ3Y E = 200 GPa[a] H = 12.00 GPa[m]	6.56	6.21	6.37	
II - TZ3YA E = 210 GPa[a] H = 12.03 GPa[m]	6.024	5.96	6.04	
III - TZ3Y20A[b] E = 260 GPa[*] H = 13.64 GPa[m]	6.85	5.73	5.8	9.6
IV - YPSZ-2.5 E = 190 GPa[a] H = 9.72 GPa[m]	6.77	6.64	7.31	

a: assumed; m: measured; *: supplier specification.

b: K_{Ic} for this material (TZ3Y20A) is 6.0 $MN/m^{3/2}$ as specified by the supplier.

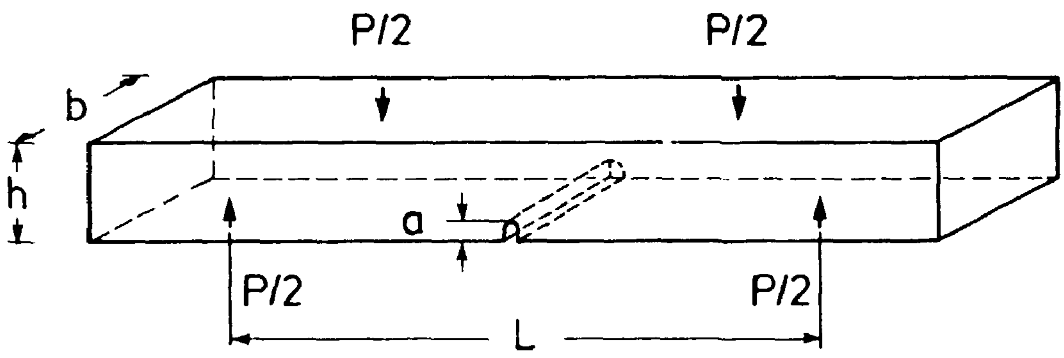


Fig. 1. Sample geometry by notched beam testing.

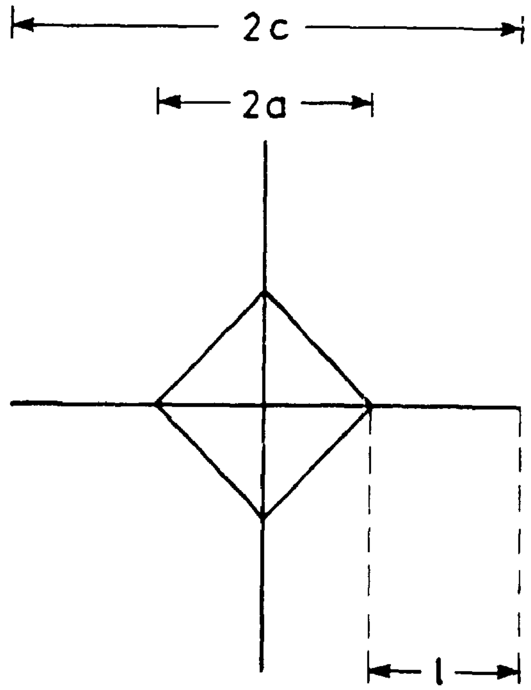


Fig. 2. Schematic representation of impression and radial cracks produced by a Vicker's indenter.

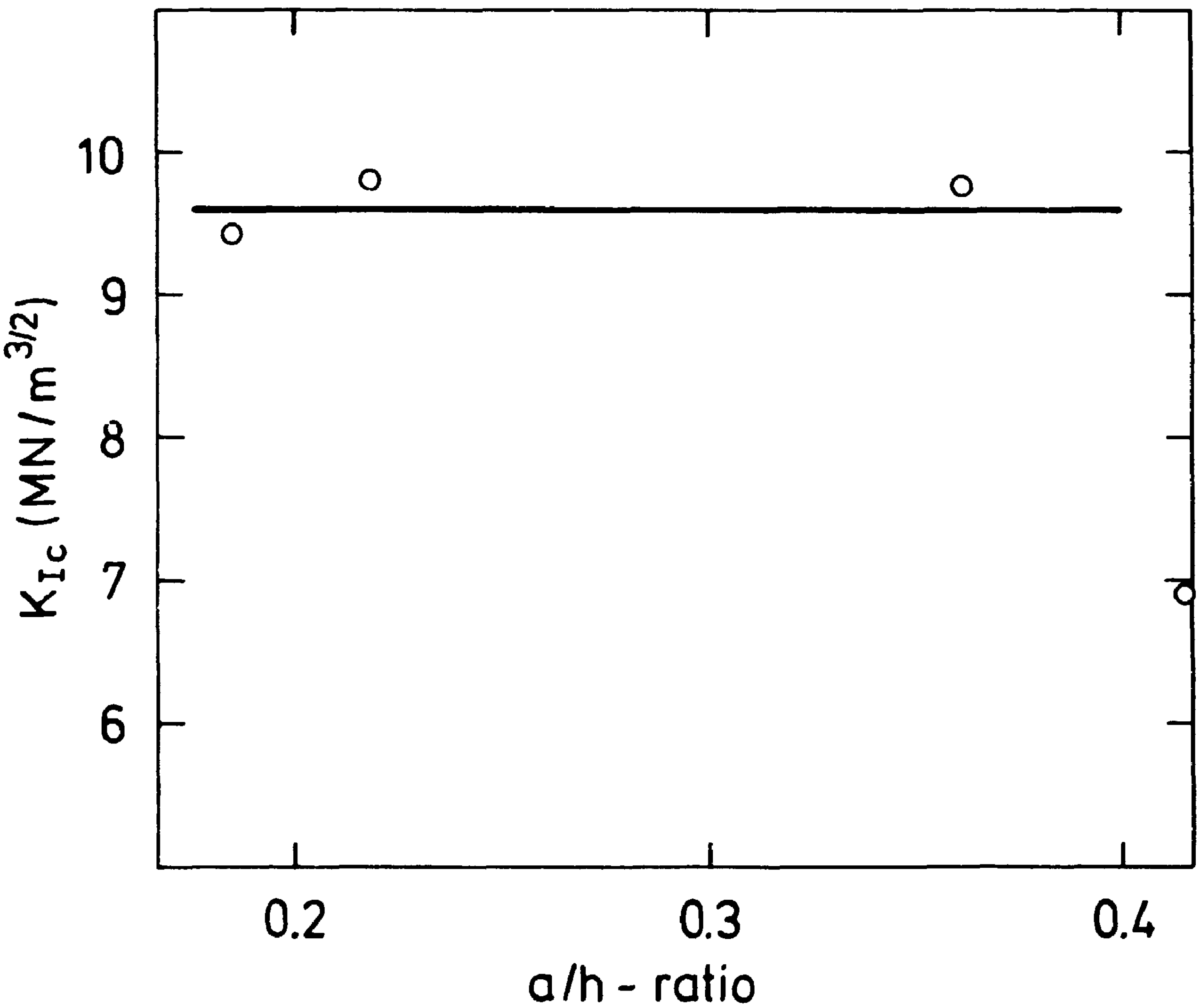
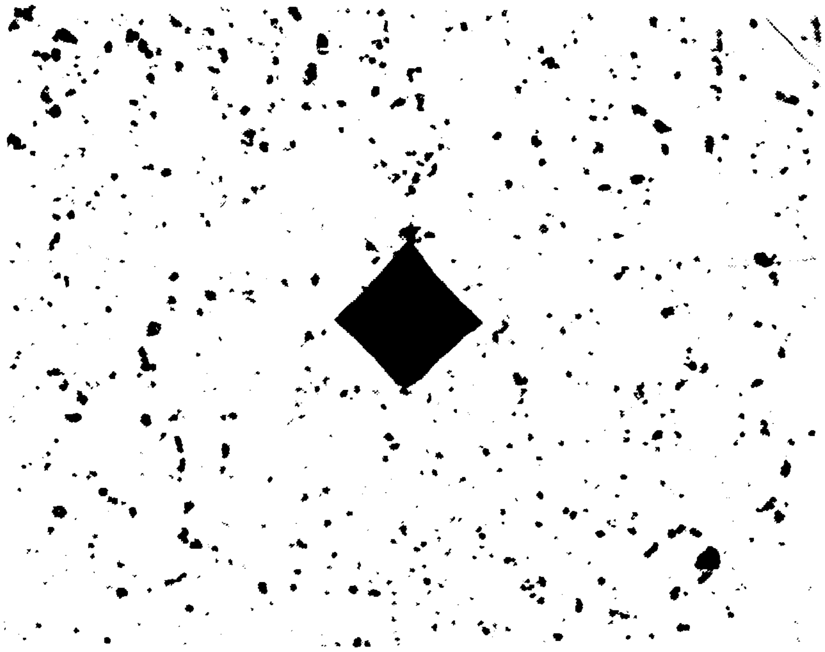
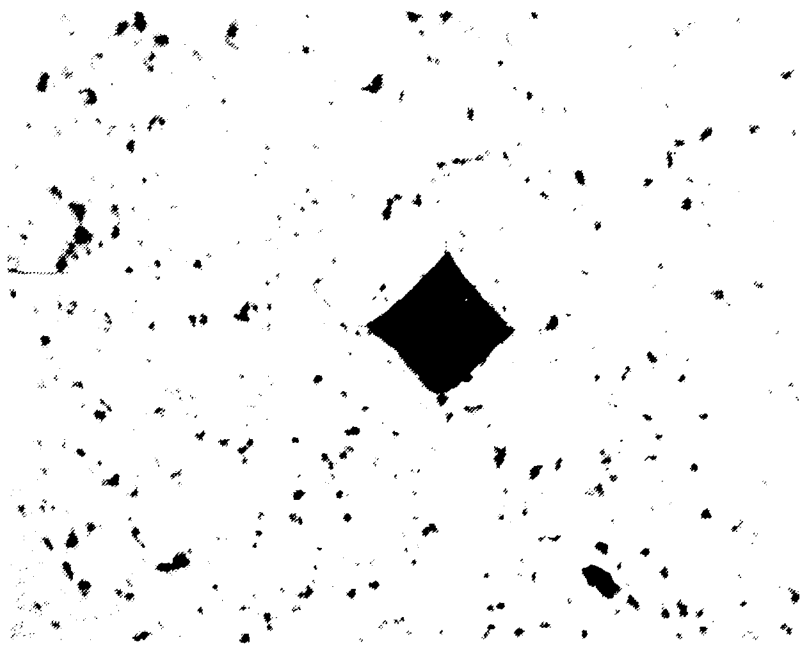


Fig. 3. Fracture toughness determined by the notched beam technique for TZ3YA as a function of the a/h (notch depth/specimen thickness) ratio. All samples were annealed at 1450°C before testing.



4 a



4 b

Fig. 4. Radial cracks around Vicker's indenter in sintered TZ3Y20A samples as polished (a) and as polished and then annealed (b). Annealing temperature: 1450^{o}C. Load: 300N.

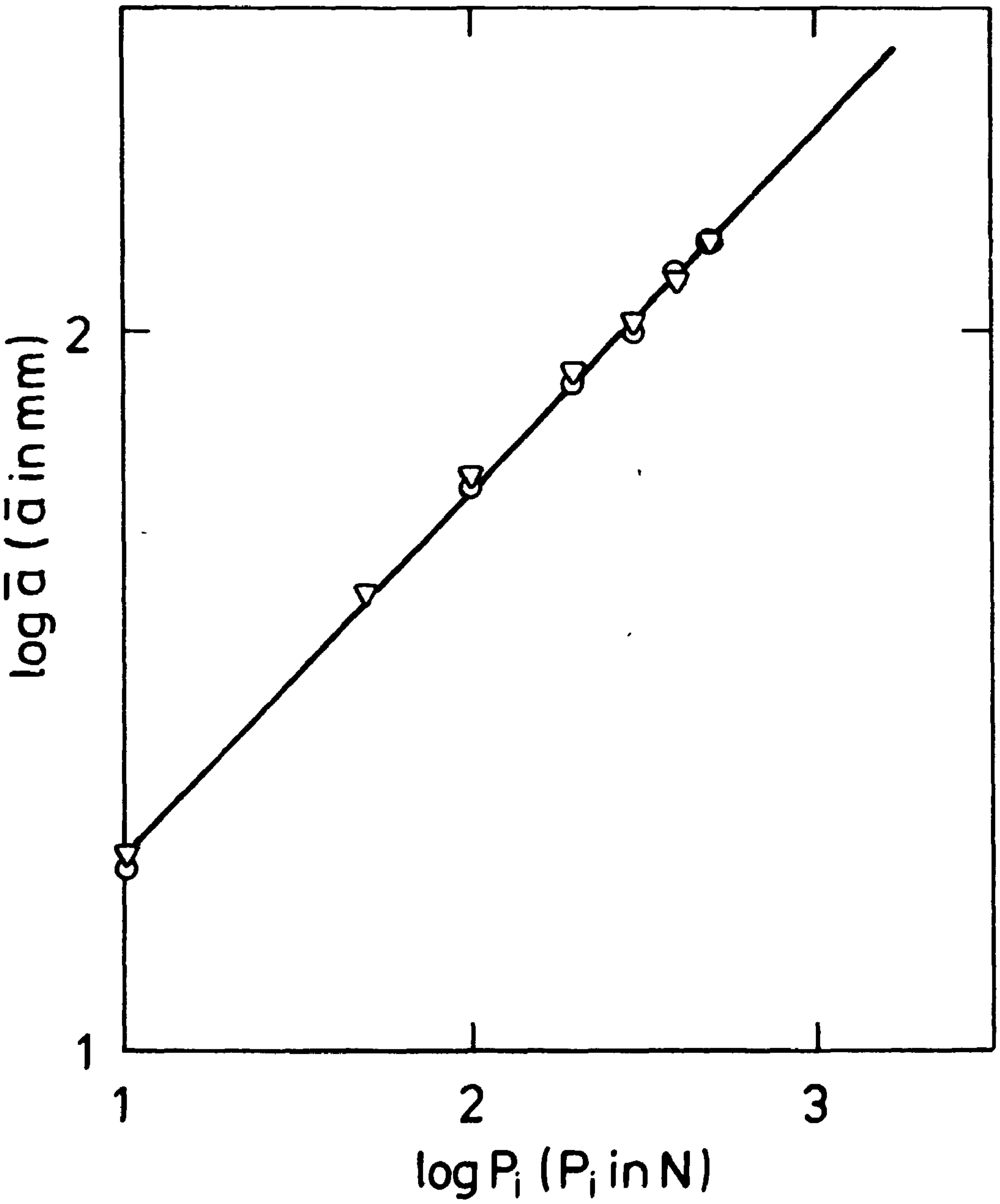


Fig. 5. Relation between log a (a = average value of half indent diagonal) and log P_i (P_i = indent load) determined by Vicker's indentation measurements on TZ3Y20A.

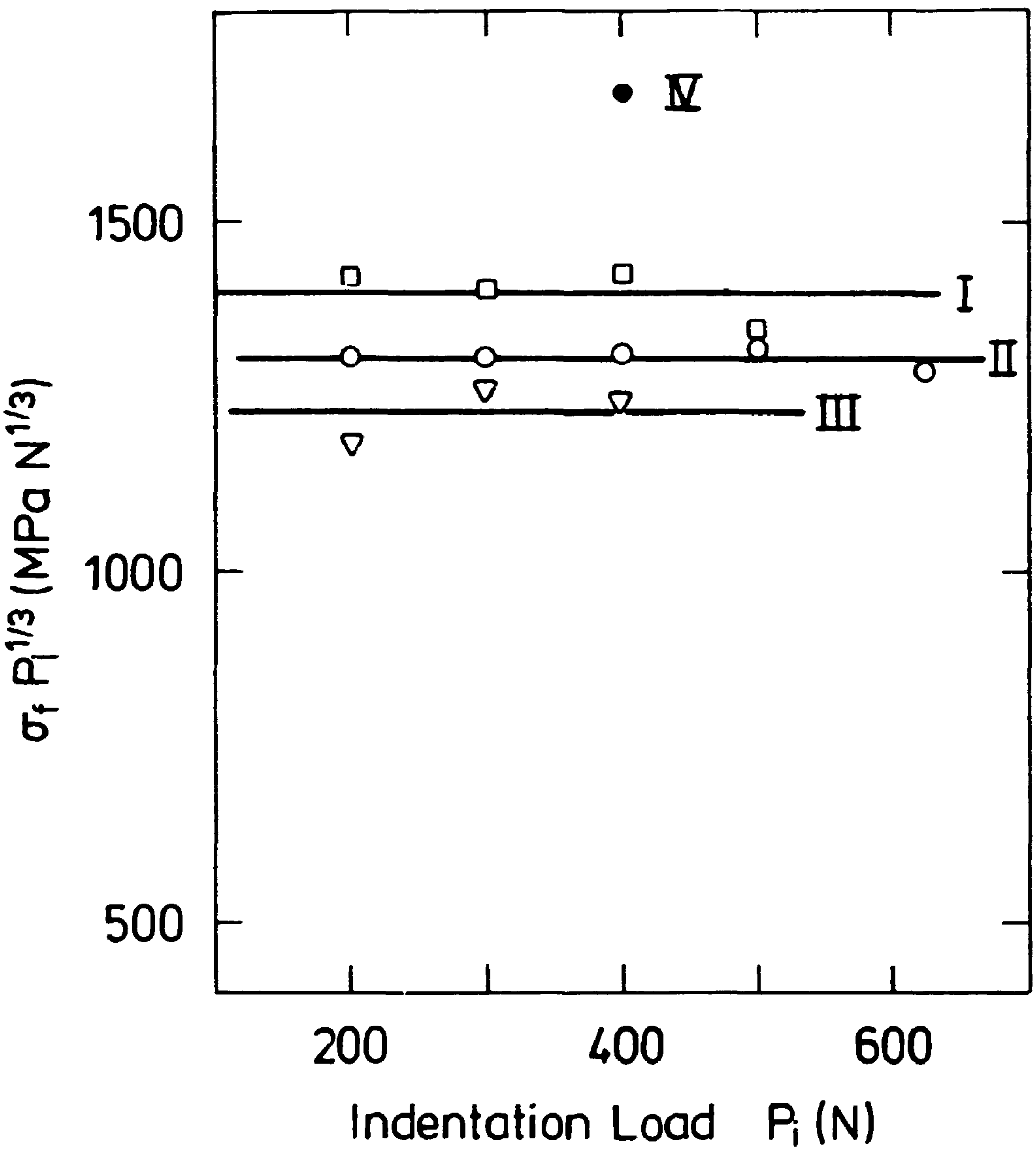


Fig. 6. Plot of $\sigma_f P_i^{1/3}$ versus indentation load, P_i, for ISB-measurements on samples prepared from TZ3Y (I), TZ3YA (II), TZ3Y20A (III) and YPSZ-2.5 (IV). Samples from I, II and IV were sintered at 1535°C (3 hrs) whereas samples from III were sintered at 1635°C (30 min.).

Fig. 7. SEM photograph of fracture surface of TZ3Y20A sample fractured by the ISB-technique.

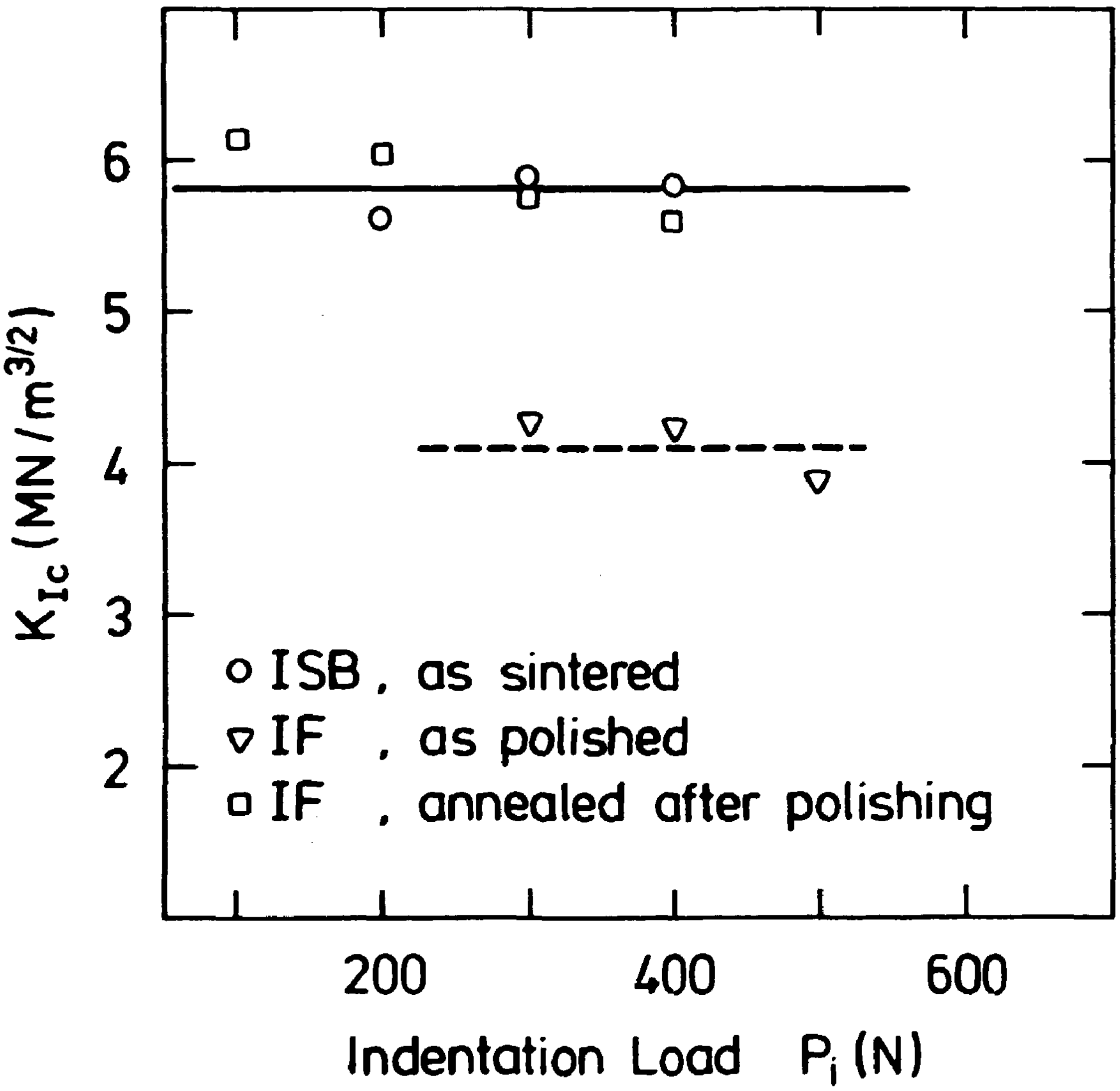


Fig. 8. K_{Ic} values versus indentation load determined by the IF- and ISB-techniques on TZ3Y20A (sintered at 1635°C for 30 min.).

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Fracture toughness measurements on zirconia toughened ceramics

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Descriptors - INIS:

BENDING; CERAMICS; COMPARATIVE EVALUATIONS; DOPED MATERIALS; FRACTURE PROPERTIES; HARDNESS; MEASURING METHODS; MECHANICAL TESTS; NOTCHES; SINTERED MATERIALS; YTTRIUM OXIDES; ZIRCONIUM OXIDES