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Subcritical crack growth in partially stabilized zirconia (PSZ)

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Studies on the subcritical crack growth behaviour of partially stabilized zirconia (ZrO_2–I, 5 to 10 vol % tetragonal phase; ZrO_2–II, 35%) were carried out using the double-torsion technique and data from the dynamic fatigue of unnotched bend specimens. The results of subcritical crack growth support the model of stress induced transformation from the tetragonal to monoclinic modification. Differences in the crack growth parameter "n" (as-received condition) using the double-torsion technique or bend specimens may be explained by the special nature of subcritical crack extension at stressed surfaces for these different specimen types. The log v–log K_I plot of ZrO_2–I using the double torsion technique shows a plateau of constant velocity, which has to be attributed to a tetragonal–monoclinic transformation. After annealing (1500° C, 5 h) the plateau has vanished and the n value is comparable to bend test in an as-received condition.

1. Introduction

The high strength and the high resitance to fracture of partially stabilized ziconia (PSZ) are attributed to the stress induced transformation of the dispersed tetraonal phase to the room temperature stable phase of monoclinic modification and the accompanying energy absorption process. Recent reports [1–6] strongly support this idea. The aim of this work is to show how the tetragonal to monoclinic transformation can affect the subcritical crack growth in these materials and how crack growth parameters depend upon test procedure and specimen configuration (double-torsion (DT) or bend specimen) as a function of surface condition (as-received and annealed).

With double-torsion specimens (DT) a single well defined macroscopic crack extends in one plane. Crack velocity v and stress intensity K_I are well defined at every point of the log v–log K_I curve.

In the case of bend specimens the microcrack entension from the specimen surface is not explicitly described. The microcrack dimensions are comparable to the microstructure and the extending microcracks are influenced by microcrack density. It is questionable therefore whether continuum mechanics are suitable for the microstructural situation.

2. Materials

Partially stabilized zirconia (PSZ) was used for testing, with the notation ZrO_2–I and ZrO_2–II, where ZrO_2–I has 5 to 10 vol % and ZrO_2–II has 35 vol % of the tetragonal phase. Other properties are given in Table I [1, 5].

Both materials ZrO_2–I and ZrO_2–II were analysed with X-rays in the as-received condition. The crystallographical modifications of the surfaces were carried out using Cu Kα radiation. All three crystallographical modifications (monoclinic, tetragonal and cubic) were present in the as-received condition. Carefully polishing the specimen surface removes the monoclinic phase indicating that no monoclinic phase exists within the interior of the material. The thickness of the monoclinic layer was about 30 μm [7].

3. Experimental procedure

3.1. Double-torsion (DT) technique

The dimension of the DT specimens were $w = 23$ mm, $d = 2$ mm and $L = 80$ mm (Fig. 1) [8]. This technique first proposed by Outwater [9] is very popular for studying crack growth be-

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TABLE I Properties of zirconia material

	ZrO_2–II	ZrO_2–I
Density ($g\,cm^{-3}$)	5.75	5.75
Porosity (%)	1.5	1.5
Grainsize (μm)	60	60
E-Modulus (Pa)	2.4×10^{11} (dynamic)	2.1×10^{11} (dynamic)
Flexural strength ($MN\,m^{-2}$)	448*	280*
K_{IC} (1150° C, 0.5 h) ($MN\,m^{-3/2}$)	7	4.6
K_{IC} (1500° C, 5 h) ($MN\,m^{-3/2}$)	3.5	3.5
Tetragonal Phase (vol%)	35	5–10

* Stress rate 0.7 $MN\,m^{-2}\,min^{-1}$.

haviour owing to its supposed analytical simplicity and the ease with which experiments are carried out.

The load relaxation technique [10] was used taking specimens without a guiding notch because notch width and notch tip geometry influence the crack velocity behaviour [8]. The DT specimens used require, however, a well finished surface, as well as an extremely balanced loading device, a perfectly dimensioned specimen and a homogeneous material along the crack extension. Otherwise the crack will deviate from the midplane.

An initial crack length was introduced by a thin (50 μm) diamond saw. The notched specimen was then precracked by slowly increasing the load. As previous measurements with glass have shown K_I is independent of crack length, a, only over a certain range (Fig. 2). In our experiments the initial crack length was not shorter than $a_i = 20$ mm and the final length not larger than $a_F = 50$ mm [8]. All experiments were carried out in distilled water under the following conditions 1. as-received and 2. after annealing at 1150° C, 0.5 h and 1500° C, 5 h.

3.2. Bend-test

The 4-point bend specimens had dimensions of $d = 7$ mm, $b = 3.5$ mm and $L = 60$ mm. The inner diameter was 18 mm, the outer diameter 54 mm (Fig. 1). The bend specimens were used to evaluate the crack growth parameter n from a logarithmic plot of an ordered $\log \sigma_{F_2}$–$\log \sigma_{F_1}$ relation [11], where σ_{F_1} and σ_{F_2} are the flexural strengths having cross-head speeds of $\dot{y}_1$ and $\dot{y}_2$

$$\log \sigma_{F_2} = \log \sigma_{F_1} + \frac{1}{n+1} \log \frac{\dot{y}_2}{\dot{y}_1} \quad . \tag{1}$$

The results (Figs. 3 and 4) yield a plot with a slope of one indicating an unimodal flaw distribution. Twenty 4-point bend specimens were used in an as-received condition for each loading rate with a ratio of $\dot{y}_2/\dot{y}_1 = 800$. ($\dot{y}_2 = 20\,mm\,min^{-1}$, $\dot{y}_1 = 0.025\,mm\,min^{-1}$). The experiments were carried out in distilled water in the as-received condition.

4. Results

4.1. Double-torsion test (ZrO_2–I)

Fig. 5 shows the log v–log K_I plot of ZrO_2–I resulting from a relaxation measurement. The curve

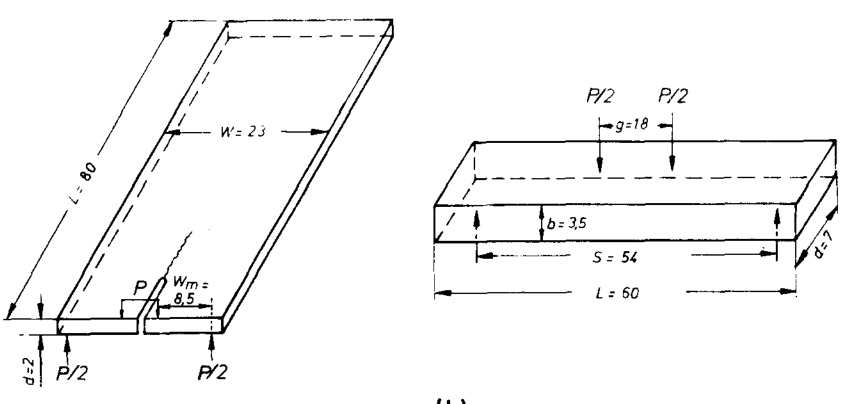

Figure 1 Specimen configurations, (a) DT and (b) 4-point.

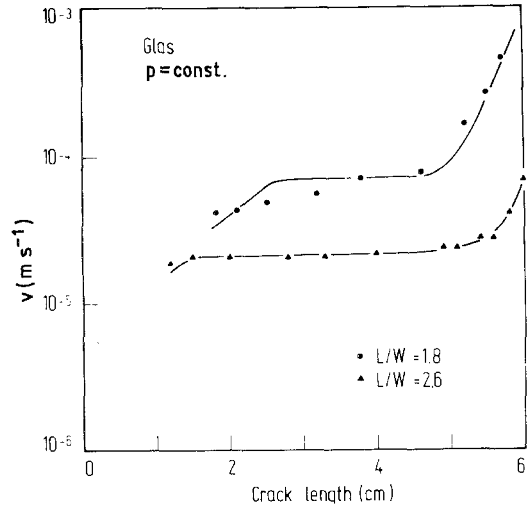

Figure 2 Dependence of K_I on crack tip position, illustrated by the dependence of crack velocity v on crack length. Constant load DT-experiment for specimen ratios $L/W = 1.8$ and $L/W = 2.6$.

exhibits two distinct regions. At a lower K_I the curve corresponds to region I of environmental assisted crack growth ($n = 80$). At higher K_I the crack velocity is nearly constant. Since the experiments were conducted in distilled water it is unlikely that the region corresponds to region II of the classical crack velocity diagram [12].

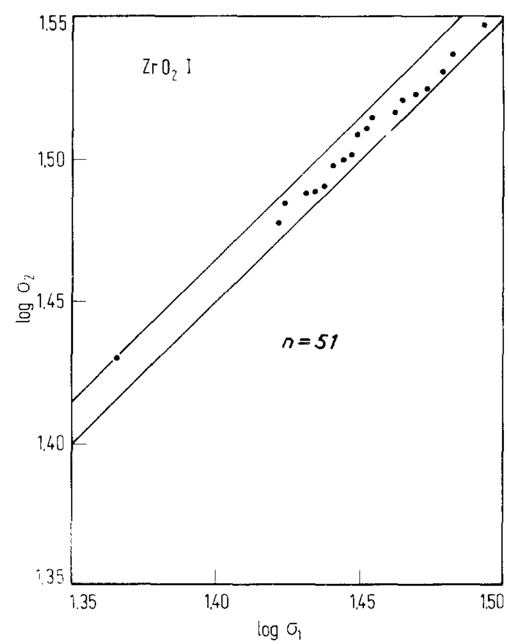

Figure 3 Logarithmic plot of ordered log σ_{F_2}–log σ_{F_1} relation. Cross head speed ratio $\dot{y}_2/\dot{y}_1 = 800$, ZrO_2–I, $n = 51$.

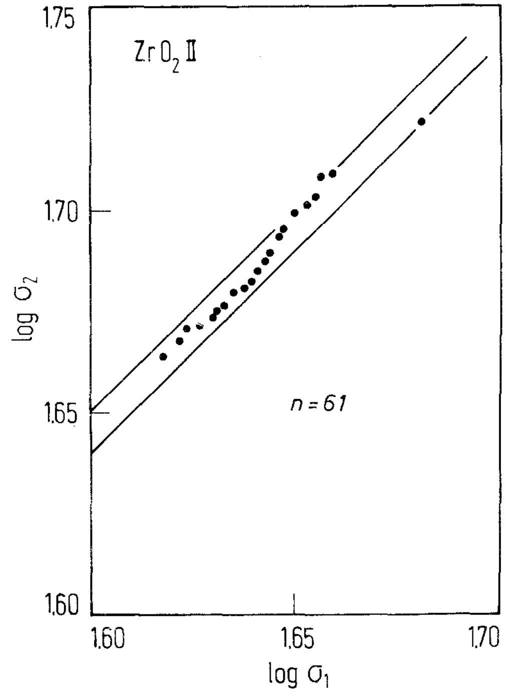

Figure 4 Logarithmic plot of ordered log σ_{F_2}–log σ_{F_1} relation. Cross head speed ratio $\dot{y}_2/\dot{y}_1 = 800$, ZrO_2–II, $n = 61$.

Annealing at 1150° C for 0.5 h results in a similar shape of log v–log K_I (Fig. 6). But n is lowered ($n = 54$) and the curve has shifted to higher stress intensities. However, after annealing at 1500° C for 5 h the plateau region has vanished (Fig. 7). After annealing at 1150° C for 0.5 h and 1500° C for 5 h the n values are equivalent, though the velocity reduction still exists at 1150° C (Figs. 6 and 7).

4.2. Double-torsion test (ZrO_2–II)

Fig. 8 shows a typical relaxation curve of a ZrO_2–II DT specimen. The characteristic discontinuous behaviour of the load relaxation is obviously due to transformation phenomena at the crack tip. It could be that with an inhomogeneous distribution of the tetragonal phase the crack runs through zones of more or less tetragonal fraction resulting in the twisted nature of the curve. Due to that specific nature no evaluation by $v = AK_I^n$ was possible. Annealing at 1150° C for 0.5 h has no effect. But after annealing at 1500° C for 5 h the relaxation curve became smooth and the K_I–v plot was comparable to that of ZrO_2–I after the same heat treatment (Fig. 7).

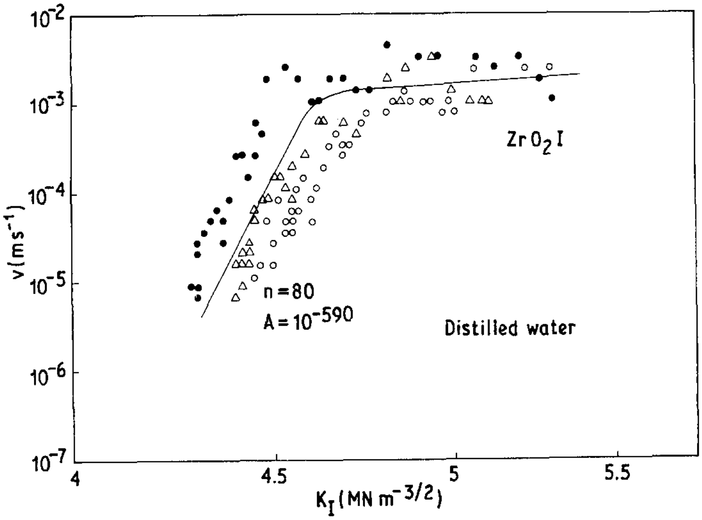

Figure 5 log v–log K_I plot of ZrO_2–I. Results of three DT-experiments, (○ ● △), $v = A\,K_I^n$ as-received condition, $n = 80$.

4.3. Bend test

Figs. 3 and 4 illustrate the evaluation of n from the logarithmic plot of an ordered log σ_{F_2}–log σ_{F_1} relation. The n-values of 51 (ZrO_2–I) and 61 (ZrO_2–II) evaluated from bend tests in an as-received condition are comparable to values of double-torsion tests after the annealing processes, however, the n-values measured from DT experiments for as-received ZrO_2–I ($n = 80$) (Fig. 5) reveals a great difference in subcritical crack extension compared to the bend test in that case.

5. Discussion

5.1. Double-torsion test

The existence of a velocity reduction in the double-torsion test relates well to the idea of stress induced transformation in front of the crack tip.

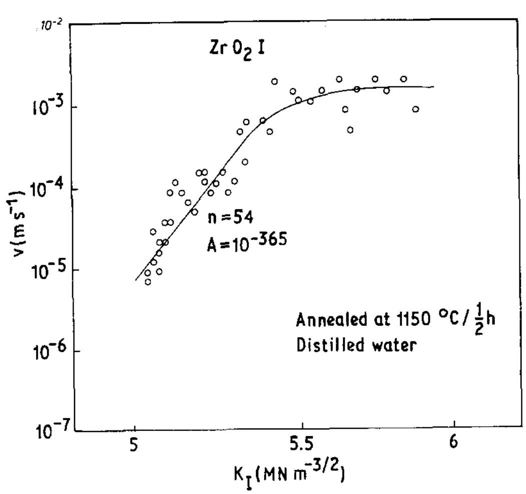

Figure 6 log v–log K_I plot of ZrO_2–I. Annealed 1150° C for 0.5 h, $n = 54$.

At low loads, v increases with stress intensity following the relation $v = A\ K_I^n$, where A and n are the crack growth parameters for environmental assisted crack growth. Above a certain K_I tetragonal to monoclinic transformation is increased to such a degree that the crack velocity is retarded appreciably by transformation and by the compression stress at the crack tip. With further increase of K_I the retarding effect is enhanced, so that an increase in crack velocity is nearly compensated for.

Heat treatment at 1150° C for 0.5 h reduces the compression stress at the surface of the DT-specimen which has formed by surface preparation (monoclinic layer, as-received condition). The change in n ($n = 80$, as-received and $n = 54$, annealed) to lower values indicates that n is strongly affected by surface compression.

After annealing at 1500° C for 5 h the tetragonal phase has transformed to the monoclinic modification [1, 6]. According to this the fracture toughness K_{IC} reaches the lower value of $3.5\,MN\,m^{-3/2}$ for ZrO_2–I and ZrO_2–II (Table I), the velocity plateau of ZrO_2–I has vanished and the relaxation curve of ZrO_2–II became smooth so that an n evaluation was possible (Fig. 7). The log v–log K_I curve changes its position to lower stress intensities which corresponds to a lower fracture toughness K_{IC}.

The fact that the n-value in the absence of the tetragonal phase (annealing at 1500° C) is equivalent to that in the presence of tetragonal phase without surface compression (annealing at 1150° C) means that the environmental assisted n is only affected by surface compression and not by

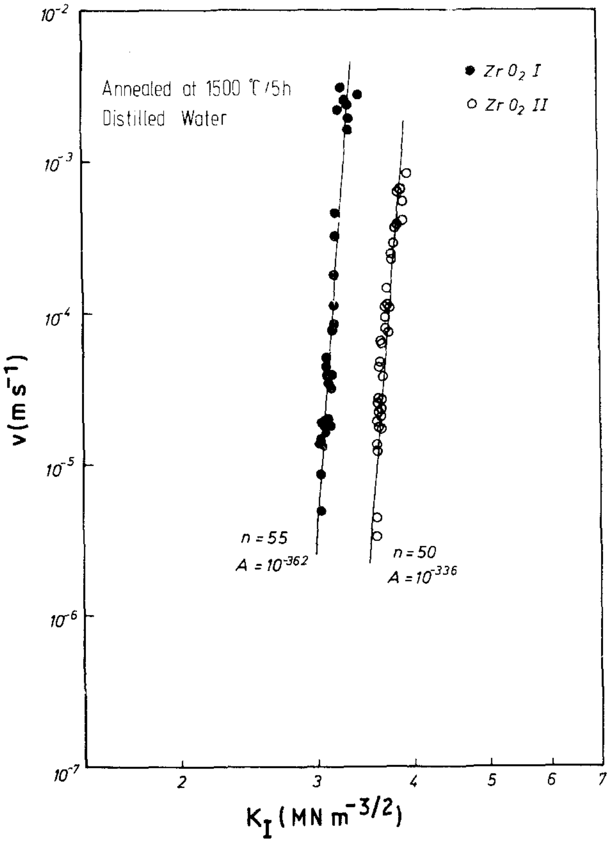

Figure 7 log v–log K_I plot of ZrO_2–I and ZrO_2–II, annealed 1500° C for 5 h, ZrO_2–I, $n = 55$, ZrO_2–II, $n = 50$.

a transformation process. The velocity plateau of ZrO_2–I, however, and the twisted nature of the relaxation curve of ZrO_2–II in the as-received condition and after annealing at 1150° C results from a stress induced tetragonal–monoclinic transformation at the crack tip.

5.2. Bend test

Taking the above assumption that the n evaluation from the DT-experiment is only affected by surface compression and that the transformation process occurs at higher stress intensities the following may be stated: The difference in n between DT- and bend-tests for as-received ZrO_2–I is produced by different influence of surface compression during crack extension [7]. For bend specimens subcritical crack extension starts after the external load stress at the surface exceeds the induced surface compression stress. With DT-specimens however, the macrocrack front at its outer parts moves continuously through zones of compression stress (as-received condition). So, n is increased. In consequence after removing the surface compression by annealing the n-values of both specimen configurations are comparable.

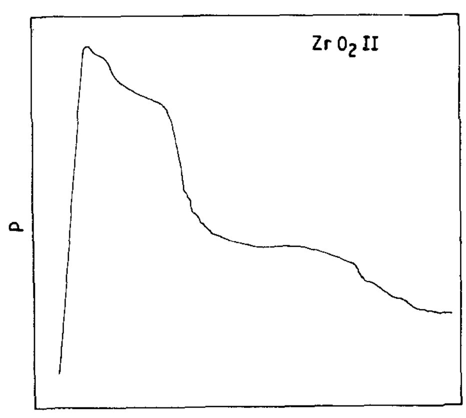

Figure 8 Discontinuous load–relaxation curve of ZrO_2–II. As-received condition.

In Table II n-values from DT- and bend tests are summarized from the literature comprising the data of this work. Agreement between these tests is not always found. Differences and accordance between our measurements may be found in the above discussion.

6. Conclusions

Double-torsion measurements and dynamic fatigue data of bend specimens reveal how the tetragonal particles interact with the propagating crack giving

TABLE II

Material	Test conditions	n	n	Reference
		DT	dynamic	
SiC	1400° C, in air	21	21	[13]
Mortar	water	17,8	14.9	[14]
Cement	water	34,2	17.7	[14]
Piezoelectric ceramic	air	56	63.5	[15]
Float glass	41–67% humidity	17,2	18	[16]
Cithium alumino-silicate	water	46	37	[17]
Magnesium alumino-silicate	water	84	63	[17]
ZrO_2–I	water	80	51	this work
		54*		this work
		55†		this work
ZrO_2–II	water	50†	61	this work

*Annealed, 1150° C, 0.5 h.
†Annealed, 1500° C, 5 h.

evidence to a stress induced transformation in partially stabilized zirconia. The DT K_I–v curve of as-received and annealed material (1150° C, 0.5 h, ZrO_2–I) shows a plateau of nearly constant velocity which has to be attributed to a tetragonal–monoclinic transformation at a certain K_I. The effect vanishes when the DT-specimen is annealed at 1500° C for 5 h. The n-value evaluated by $v = A\ K_I^n$ at low stress intensities decreases from $n = 80$ for the as-received condition to $n = 54$ in the annealed condition (ZrO_2–I). A DT-evaluation of n for ZrO_2–II, which has a higher content of tetragonal phase, was not possible in the as-received condition. Annealing at 1500° C for 5 h yields an n-value of 50.

The difference in crack growth parameter n between bend strength ($n = 51$, $n = 61$) and DT-measurement ($n = 80$) in the as-received condition results from a different reaction of the specimen surface on subcritical crack extension. If double torsion specimens are annealed the n-value is comparable to that of an as-received bend specimen.

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