MICROMECHANISMS OF SUBCRITICAL CRACK GROWTH OF PARTIALLY STABILIZED ZIRCONIA (PSZ)

Li-Shing Li and R. F. Pabst

Max-Planck-Institut fuer Metallforschung, Institut fuer Werkstoffwissenschaften, Stuttgart, Federal Republic of Germany

ABSTRACT

Studies on the subcritical crack growth behaviour of PSZ (ZrO_2-I, 5-10% tetragonal phase; ZrO_2-II, 35%) were carried out using the DT-technique and data from the dynamic fatique of unnotched bend specimens. The DT log v-log K_I curve of the as-received and annealed materials (1150 °C, 1/2h, ZrO_2-I) show a plateau of nearly constant velocity which has to be attributed to stress induced phase transformations at higher stress intensities. The effect vanishes when the DT-specimen is annealed at 1500 °C for 5h. The n-value evaluated at low stress intensities decreases from n = 80 for the as-received condition to n = 54 in the annealed condition (ZrO_2-I). The n-values are then comparable to the bend-test data in the as-received conditon.

KEYWORDS

Partially stabilized zirconia; subcritical crack growth; double torsion-test, stress induced phase transformation; surface compression stress.

INTRODUCTION

The fracture toughness of PSZ can be enhanced by the effect of stress induced phase transformation (tetragonal to monoclinic). This creates a zone of microfracture or compression in front of the crack (Li and Dworak, 1978; Dworak and co-workers, 1977, 1978; Garvie and co-workers, 1975; Hannink, 1978; Porter and Heuer, 1979). However, nothing is usually stated about how the transformation process can affect the subcritical crack extension and how the subcritical crack growth parameters depend upon the surface conditions (as-received and annealed), the test procedure and the specimen configuration. This paper, therefore, deals with the crack extension behaviour of a single well defined macroscopic crack within a double-torsion (DT) specimen, as well as with the extension of microcracks within a stressed surface of 4-point-bend specimens.

EXPERIMENTAL PROCEDURE AND RESULTS

The partially stabilized zirconia (PSZ) was used for testing with the notation

ZrO_2-I and ZrO_2-II, where ZrO_2-I had 5-10 vol% and ZrO_2-II 35 vol% of the tetragonal phase. Other properties are given in Table 1 (Stuhrhahn and co-workers, 1975). All three modifications (monoclinic, tetragonal, cubic) were present at the surface in the as-received condition.

The dimensions of the DT-specimens were W = 23 mm, d = 2 mm and L = 80 mm (Fig. 1) (Li and co-workers, 1980). The load relaxation technique was used with specimens without a guiding notch * . An initial crack length was introduced with a thin (50 microns) diamond saw and the notched specimen was then precracked by slowly increasing the load. The initial crack length a_i was not shorter than 20 mm and the final length not larger than a_F = 50 mm (Li and co-workers, 1980). The experiments were conducted in distilled water.

TABLE 1 Properties of Zirconia Material

		ZrO_2-I	ZrO_2-II
Tetragonal Phase	Vol%	5 - 10	35
Density	g/cm^3	5.75	5.75
Porosity		1.5 %	1.5 %
Grain Size	µm	60	60
E-Modulus	Pa	2.1×10^{11} (dyn.)	2.4×10^{11} (dyn.)
Flexural Strength	MN/m^2	280 *	448 *
K_{IC} (RT)	$MN/m^{3/2}$	4.6	7.0
K_{IC} (1500 °C,5h)	$MN/m^{3/2}$	3.5	3.5
n (DT) as received		80	-
n (DT) 1150 °C, 1/2 h		54	-
n(DT) 1500 °C, 5 h		55	50
n (bend test) as received		51	61
test medium **		water	water

* stress rate 0.7 $MN/m^2 s$
** for DT and bend tests
RT = Room Temperature
DT = Double Torsion

* R.F. Pabst and J. Weick "DT-measurements with and without a guiding notch", to be published

The 4-point-bend specimens had dimensions of d = 7 mm, b = 3.5 mm and L = 60 mm. The inner span was 18 mm the outer span 54 mm (Fig. 1). The crack growth parameter "n" was evaluated from a logarithmic plot of an ordered log σ_{F1}-log σ_{F2} relation (Evans and Wiederhorn, 1974), where σ_{F1} and σ_{F2} are the flexural strengths at cross head speeds of $\dot{y}_1$ = 0.025 mm/min and $\dot{y}_2$ = 20 mm/min. All experiments were conducted in distilled water with the PSZ in the as-received condition.

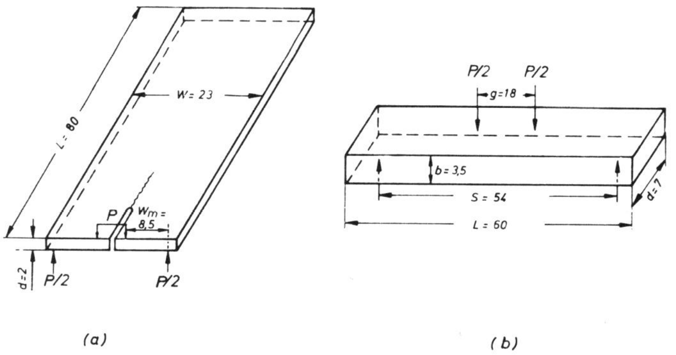

Fig. 1 Specimen configurations, (a) DT, (b) 4-point-bend

Fig. 2 shows a log v-log K_I plot of ZrO_2-I resulting from a DT-relaxation experiment. The curve exhibits two distinct regions. At the lower stress intensities the curve corresponds to the region I of an environmental assisted crack growth described by a relation $v = A\,K_I^n$ (A, n crack growth parameters). Annealing at 1150 °C for 1/2 h decreases n from 80 to 54 and the curve has shifted to the right. With

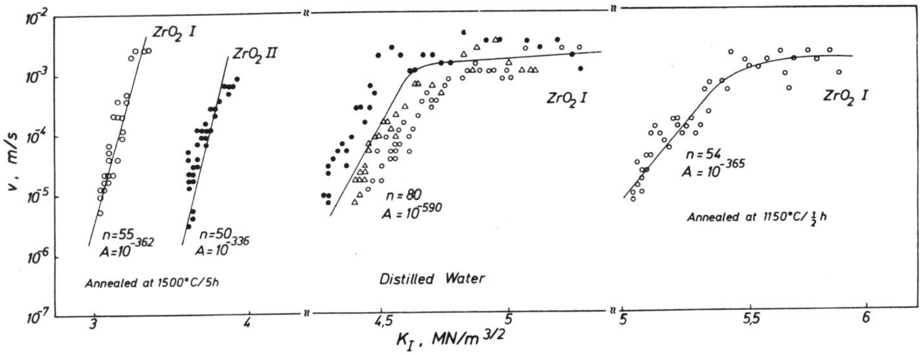

Fig. 2 log v-log K_I plots of ZrO_2-I and ZrO_2-II DT-relaxation, materials functions $v = A\,K_I^n$
(a) as-received condition, n=80, ZrO_2-I (oo) three experiments
(b) annealed 1150 °C - 1/2 h, n = 54, ZrO_2-I
(c) annealed 1500 °C - 5 h, n = 55, ZrO_2-I, n = 50, ZrO_2-II

increasing K_I the crack velocity becomes nearly constant. After annealing at 1500 °C for 5 h the plateau region has vanished. After annealing at 1150 °C and 1500 °C, the n-values are equal, although velocity reduction still exists at 1150°C. Due to a discontinuous behaviour of the load relaxation curve, no evaluation of n and A was possible for ZrO_2-II. Annealing at 1150 °C for 1/2 h had no effect. After annealing at 1500 °C for 5 h the relaxation curve became smooth and the log v-logK_I plot was comparable to that of ZrO_2-I after the same heat treatment (Fig. 2).

The n-values (Table 1) evaluated from bend tests of the PSZ in the as-received condition are comparable to values of the DT-test after annealing. However, the n-values measured from DT-experiments of ZrO_2-I in the as-received condition reveal a great difference in subcritical crack extension behaviour of macro- and microcracks.

DISCUSSION

The existence of a velocity reduction in the DT-test at higher stress intensities relates well to the idea of stress induced transformation in front of the crack tip. Heat treatment at 1150 °C reduces the compression stresses at the specimen surface in the as-received condition and n decreases from n = 80 to n = 54. After annealing at1500 °C the tetragonal phase has transformed to monoclinic and the velocity plateau of ZrO_2-I has vanished. The relaxation curve of ZrO_2-II became smooth and an evaluation of "n" became possible. The fact that the n-values are equal after annealing (Table 1) indicates that the environmental assisted crack growth is affected only by a surface compression stress (as-received condition) and not by the transformation process at the crack tip. The shift to lower intensities of the log v-log K_I curve after annealing at 1500 °C corresponds to a lower fracture thoughness of $K_{IC} = 3.5\ MN/m^{3/2}$ for both ZrO_2-I and ZrO_2-II (Table 1). But it is not yet clear why after annealing at 1150 °C the log v-log K_I curve moves to the right to higher stress intensities.

From the above discussion it may be stated that the differences in "n" for the as-received ZrO_2-I between macrocrack extension (DT-test) and microcrack growth (bend test) is produced by different influences of surface compression stress during crack extension (Li and Pabst, 1980). For bend specimens subcritical crack extension starts after the external load stress at the surface exeeds the induced compression stress. With DT-specimens however, the macrocrack front at its outer parts moves through zones of compressive stresses. Therefore, after the annealing process, the n-values of both specimen configurations are comparable.

CONCLUSIONS

The tetragonal to monoclinic transformation is discussed on the bases of: a) forming a stress induced compression zone at the crack tip or at the as-received surfaces (ground) or b) forming a zone of microcracking at the front of the crack. The subcritical extension parameters in the as-received condition and the annealing reactions indicate that only the influence of compressive stresses may explain the results. The annealing time is much to short to account for microcrack healing. This does not exclude, however, different reactions within other systems like Al_2O_3 with an unstabilized ZrO_2 dispersion.

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