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## Subcritical crack growth in zirconia-toughened alumina ZTA ceramics

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A study of subcritical crack growth in zirconia-toughened alumina (ZTA) ceramics was carried. The Al<sub>2</sub>O<sub>3</sub>-10 wt% ZrO<sub>2</sub> ceramics containing two types of ZrO<sub>2</sub> particles: 3mol% yttria stabilized ZrO<sub>2</sub> and pure ZrO<sub>2</sub> were tested. For comparison alumina ceramics was included to examination. The work load-relaxation technique was used for observation subcritical crack growth. The crack length was evaluated by linear-elastic analysis from the compliance of single-edge-notched specimen in three-point bending test. Parameters  $n$ ,  $\log A$  depending on the material, work-of fracture ( $WOF$ ), stress intensity factor at the moment of crack initiation  $K_{Ii}$  and maximum values of stress intensity factor  $K_{I_{max}}$  were presented. The SCG  $v$ - $K_I$  curves in log-log representation has been observed.

### 1. INTRODUCTION

Desirable properties of high-performance ceramics cause their increasing use in areas previously dominated by metals and metallic alloys. Examples of such applications include cutting tools, drill bits, wear parts, structural and electronic components, electrodes, biomechanical devices, lightweight armor, and gas-turbine components [1]. In spite of excellent physical and mechanical properties of ceramics the main drawbacks are their brittleness, large scatter of strength, and subcritical crack growth. Ceramics subjected significant stresses in short time can yield brittle failure however subcritical stresses acting long time carry to fracture too. Subcritical crack growth (SCG) is a time-dependent phenomenon, where a crack is growing at constant load below  $K_I=K_{Ic}$  (where:  $K_I$  is a stress intensity factor but  $K_{Ic}$  is a fracture toughness). A crack of initial depth  $c_i$  propagates slowly until a critical load-dependent size  $c_c$  is attained at which unstable crack extension follows. Crack growth is governed by the stress intensity factor  $K_I$  and for a given material and environment there is an unique relation between the crack growth rate  $v$  and  $K_I$ :

$$v = \frac{dc}{dt} = f(K_I) \quad (1)$$

In Fig.1 a  $v$ -  $K_I$  curve is shown in the log-log representation. A low crack growth rates an extended range (region I) occurs with straight line. In this region the crack growth rate fulfills a power-law relation (2):

$$v = A K_I^n = A^* \left[ \frac{K_I}{K_{IC}} \right]^n \quad (2)$$

with the parameters  $A$ ,  $A^*$  and  $n$  depending on the material, the temperature and the environment. In some case threshold value  $K_{Ith}$  can be detected, below which no subcritical crack growth (SCG) is found. At a relative high crack growth rate a plateau (region II) in  $v$  may occur with crack growth rates independent of  $K_I$  (Fig.1) [2].

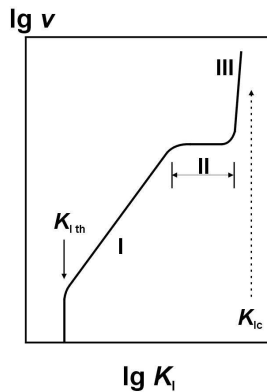


Fig.1. Typical  $v$ - $K_I$ -curve

After a further increase of  $K_I$  high crack growth rates occur (region III), succeeded by unstable crack extension with crack growth rates in the order of the sound velocity. The lowest crack growth rates are of importance for lifetime predictions. Under conditions of subcritical crack growth, finite lifetimes have to be expected. Various methods of determining the  $v$ - $K$ -curves are available in the literature [2,3]. The behavior of macrocracks in the order of several mm can be tested with the double-torsion (DT) method, double-cantilever beam (DCB)

technique, bending test with notched specimens. In presented work load-relaxation technique based on Fett and Muntz [4] method was used for observation subcritical crack growth. The crack length is evaluated by linear-elastic analysis from the compliance of single-edge-notched specimen in three-point bending test.

## 2. EXPERIMENTAL DETAILS

Zirconia-toughened alumina (ZTA) ceramics  $\text{Al}_2\text{O}_3$ -10 wt%  $\text{ZrO}_2$  containing two types of  $\text{ZrO}_2$  particles: 3 mol% yttria stabilized  $\text{ZrO}_2$  and pure  $\text{ZrO}_2$  were tested. For comparison alumina ceramics were included into examination. Concerning the conventional powder-mixing technique, a high-purity alumina powder  $\alpha$ - $\text{Al}_2\text{O}_3$  > 99.8wt% type A16SG produced by the Alcoa firm with an average particle size of below 0.5  $\mu\text{m}$  was used to process ceramics samples. Specific surface of alumina particle determined by nitrogen absorption at the temperature of liquid nitrogen is  $S_{\text{BET}}=4.54 \text{ m}^2/\text{g}$ . Mixture of alumina with pure zirconia and alumina with 3mol% yttria doped zirconia were prepared from two batches of powder. The specific surface of the tested powders for pure zirconia is  $S_{\text{BET}}=4.13 \text{ m}^2/\text{g}$  and for 3mol% yttria doped zirconia is  $S_{\text{BET}}=4.70 \text{ m}^2/\text{g}$  respectively. Next all batches were uniaxially pressed at 50 MPa to a rectangular shape in a die (60×70 mm) to procedure green compacts. These compacts were cold isostatically pressed at 250 MPa. Final composites were sintered in the high temperature electric furnace of Seco-Warwick firm. Maximum temperature reached 1923 K. The specimens thinned out to the size 1.5×4.0×50.0±0.1mm with wide polished side surface and vacuum evaporated thin aluminium layer (about 150 nm thick) were notched. An initial 0.9 mm deep notch was produced by diamond saw (0.20 mm thick) and then the notch tip was sharpened manually using a razor blade (0.025 mm thick) up to deep 1.1 mm. A sharp crack was propagated from the notch tip when the specimens have been subjected to a three-point bending test up to failure. The specimens were loaded using Zwick testing machine with rate of  $1\mu\text{m}\cdot\text{min}^{-1}$ . The PC computer read also the testing machine signal giving the

information about loading force and beam deflection. Hence the crack length  $c$  was calculated from the compliance as a function of time  $t$ . Stress intensity factor  $K_I$  was calculated from the equation (3) [4]:

$$K_I = 1.5 \frac{PL}{W^2 B} Y c^{1/2} \quad (3)$$

where:  $P$  – critical load,  $L$  – roller distance,  $W$ —specimen width,  $B$ -specimen thickness,  $Y$  – geometric function calculated according to [4],  $c$  – crack length.

Crack growth velocity  $v$  was evaluated by differentiating of  $c = f(t)$  and next the dependence (1) was established. The Work-of-fracture ( $WOF$ ) was calculated too as a ratio of the total work of deformation of the notched specimen up to fracture to the double area of the fractured cross-section of the specimen [5,6,7].

### 3. RESULTS AND DICUSSION

Subcritical crack growth parameters  $n, A$ , fracture work  $WOF$ , stress intensity factor for crack initiation  $K_{Ii}$ , maximum stress intensity factor  $K_{I_{max}}$  determined for tested ceramics are presented in Table I.

Table I. Parameters of subcritical crack growth

Material	Notch length $c_0$ (mm)	Crack length after relaxation $c_k$ (mm)	Fracture work $WOF$ ( $J/m^2$ )	Stress intensity factor for crack initiation $K_{Ii}$ ( $MPam^{1/2}$ )	Parameter in formula (2) $n$	Parameter in formula (2) $\log A$	Maximum stress intensity factor $K_{I_{max}}$ ( $MPam^{1/2}$ )
$Al_2O_3$	1.02	1.77	17.8±0.5	3.07±0.15	12.3±2.9	- 12.23±1.71	4.45±0.14
$Al_2O_3$ +10wt% $ZrO_2$ /unstabilized $ZrO_2$ /	1.09	1.80	33.4±0.9	3.94±0.13	9.8±6.37	- 18.42±4.27	5.51±0.08
$Al_2O_3$ +10wt% $ZrO_2$ /3 mol% yttria stabilized $ZrO_2$ /	1.03	1.19	23.9±4.3	3.62±0.22	-	-	3.92±0.23

On the basis data obtained from subcritical crack growth tests higher values of: fracture work  $WOF$  (twice value than pure alumina), stress intensity factor for crack initiation  $K_{Ii}$ , and maximum stress intensity factor  $K_{I_{max}}$  have been observed for alumina-zirconia ceramics with unstabilized  $ZrO_2$  than for other ceramics. It can be explain by stress-induced phase transformation toughening and microcrack toughening in  $Al_2O_3$ - $ZrO_2$  composite [8]. Phase transformation of  $ZrO_2$  from tetragonal ( $t$ ) to monoclinic ( $m$ ) has been widely used to improve the toughness of brittle ceramic matrices. The improvement is understood as a result of volume expansion during the  $t \rightarrow m$  transformation of  $ZrO_2$  grains dispersed in the matrix. In

the stress field of propagating cracks,  $t$ -ZrO<sub>2</sub> grains in an Al<sub>2</sub>O<sub>3</sub> matrix undergo the  $t \rightarrow m$  transformation (stress-induced phase transformation) and the residual stresses around already transformed  $m$ -ZrO<sub>2</sub> particles can cause microcracking. Studies in [8] showed also that unstable (pure) ZrO<sub>2</sub> particles in alumina matrix caused higher toughening than 3mol% Y<sub>2</sub>O<sub>3</sub> stabilised ZrO<sub>2</sub>. It was because the monoclinic fraction on the fractured surface was in the first case higher than in the second. In our case is probably the same situation.

Determination of subcritical crack growth parameters  $n$ ,  $A$  for Al<sub>2</sub>O<sub>3</sub> +10wt%ZrO<sub>2</sub> / 3 mol% yttria stabilized ZrO<sub>2</sub>/ were impossible because of too short time of relaxation load. For this reason it was difficult to obtain data necessary to draw SCG curve  $v$  versus  $K_I$ . The plots  $K_I$  versus crack length  $c$  and  $\log v$  versus  $\log K_I$  for Al<sub>2</sub>O<sub>3</sub> +10wt%ZrO<sub>2</sub> /unstabilized ZrO<sub>2</sub>/ are presented in Fig.2.

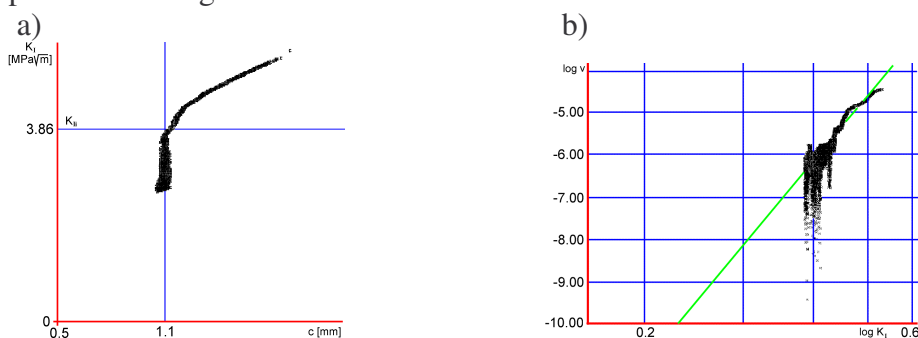


Fig.2. The curves obtained from subcritical crack growth (SCG) for Al<sub>2</sub>O<sub>3</sub> +10wt% ZrO<sub>2</sub> /unstabilized ZrO<sub>2</sub>/ a)  $K_I$  vs. crack extension  $c$  b)  $\log v$  vs.  $\log K_I$ .

## CONCLUSIONS

- Subcritical crack growth is observed in alumina-zirconia ceramics with unstabilized zirconia and in alumina ceramics.
- Determination of subcritical crack growth parameters  $n$ ,  $A$  for Al<sub>2</sub>O<sub>3</sub> +10wt%ZrO<sub>2</sub> / 3 mol% yttria stabilized ZrO<sub>2</sub>/ were impossible because of too short time of relaxation load.
- Alumina-zirconia ceramics exhibit higher values of stress intensity factor for crack initiation and WOF in comparison with alumina ceramics.

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