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Crack growth resistance of Al_2O_3-$ZrO_{2(nano)}$ (12 mol% CeO_2) ceramics

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Properties

ABSTRACT

Purpose: Alumina-zirconia ceramics with nano 12 mol% CeO_2 stabilized ZrO_2 were presented as new ceramics with improvement fracture behavior: better crack growth resistance and limited susceptibility to slow crack growth. This ceramics exhibits good cutting properties.

Design/methodology/approach: The controlled crack growth of specimens with double notch was carried with using Zwick testing machine. The specimens with double notch were loaded with rate of $1 \mu m min^{-1}$.The loading of the specimens has been broken at the moment of crack propagation. The procedure has been repeated many times up to obtaining definite increase of crack length. In situ observation of crack growth from notch up to failure was possible due to special device with microscopical objective heads in vertical configuration coupled with CCD camera fitted to Zwick 1446 testing machine. A load-relaxation technique method was used for determination of a relationship of a crack growth velocity (v) versus stress intensity factor (K_I) and calculation the parameters of SCG.

Findings: Many newly developed ceramics that are designed for high-temperature applications or electronic devices possess a low crack growth resistance. It is a drawback in widely usage of these materials in industry. Application of the ZrO_{2nano} stabilized with 12 mol% CeO_2 in alumina–zirconia ceramics improves the fracture toughness and decreases the susceptibility to slow crack growth in comparison to pure alumina ceramics. Observation of the controlled crack growth in the tested ceramics reveals the existence of increasing R-curve in the alumina-zirconia ceramics with nano ceria stabilized zirconia.

Practical implications: Due to improvement of fracture behavior of the alumina-zirconia ceramics with nano 12 mol% CeO_2 stabilized ZrO_2 could be used in “dry cutting” machining.

Originality/value: A combination of in situ microscopic long-through thickness crack growth observation during three point bending (3PB) of a single edge notched beam (SENB) enabled measurement of the R-curve. In presented work a new load-relaxation method was worked out for determination susceptibility tested ceramics to slow crack growth.

Keywords: Ductility and crack resistance; Fracture toughness; R-curve; Slow crack growth

1. Introduction

Many newly developed ceramics that are designed for high-temperature applications or electronic devices possess a low crack growth resistance [1]. It makes difficult for use of these materials, because components may fail as a result of thermal or mechanical loads [2]. The mechanical behavior of ceramics materials such as alumina (Al_2O_3), alumina-zirconia (Al_2O_3-ZrO_2) composites, ZrO_2

and silicon nitride-based (Si_3N_4-based) ceramics are also prone to show stable crack growth when tested under constant or static loads [3]. The phenomenon that induces such crack growth behavior is traditionally called *static* fatigue. About ten percent of the advanced ceramics makes the structural ceramics in which the mechanical properties such as strength, toughness, wear resistance, hardness etc. are of primary interest [4]. Ceramic materials based on alumina are widely used as a cutting material (Fig.1).

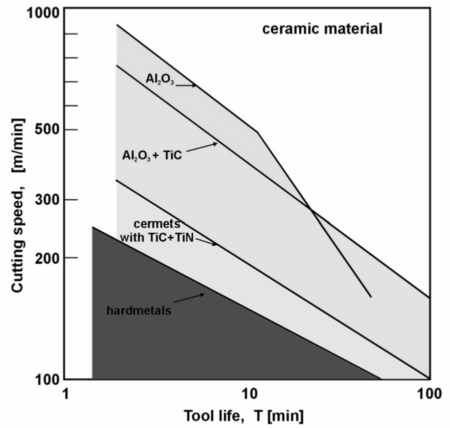

Fig. 1. Comparison of the cutting properties of selected ceramic materials with hardmetals [5]

Those the widespread using of ceramics in structural application has been limited by their brittle fracture behavior (low fracture toughness), and low reliability. Apart of brittleness the significant disadvantages of ceramics are: low tensile strength at room temperature for some materials, large scatter of strength and slow-crack growth. Some structural ceramics show an increase of fracture resistance with crack extension under stable crack growth [6]. This phenomenon is called *R-curve* behavior. Increase of fracture resistance with crack extension may occur due to several effects. The most serious influence is friction at the border of the crack tip, which can cause so-called bridging effects. Further possible reasons for the increase of fracture resistance with crack extension are all energy-consuming effects, for example, crack branching. Another reason for *R-curve* behavior is phase transformation effects, which are characteristic of zirconia ceramics. *R-curve* behavior of ceramic materials is a desirable mechanical effect. Oxide ceramics are sensitive to slow crack growth because adsorption of water can take place at the crack tip, leading to a strong decrease of the surface energy in humid (or air) conditions. This is a major drawback concerning demanding, long-term applications. The concept of crack growth resistance curve (K_R curves) has been used to determine the point of crack instability. The failure of ceramics can be caused by slow crack growth of preexisting flaws until the critical dimension is attained. Slow crack growth sometimes called subcritical crack growth (SCG) is a time-dependent phenomenon, where a crack is growing at a load below $K_I = K_{Ic}$ (where: K_I is a stress intensity factor and K_{Ic} is fracture toughness). Crack growth is governed only by the stress intensity factor K_I and for a given material and environment there is an unique relation between the crack growth velocity v and K_I [7]:

$$v = A\,K_I^n = A^*\left[\frac{K_I}{K_{IC}}\right]^n \qquad (1)$$

where: A, A^* are the proportionality constants, n is an empirical parameter depending on the material, the temperature and the environment. The high value of n is found for most ceramics in dry or inert conditions, but for most oxide ceramics in water containing environments the low values of n are determined. In some case threshold value K_{I0} can be detected, below which no slow crack growth (SCG) is found. The present work focuses on the crack growth resistance behavior of alumina-zirconia ceramics with nano ceria–stabilized zirconia. For the investigation of this behavior the *R-curve* as a relationship $K_R = f\ (c)$ and the parameters of the slow crack growth (SCG) were determined.

2. Experimental procedure

Alumina-10 wt% zirconia ceramics with nano 12 mol% CeO_2 stabilized ZrO_2. were tested. A high purity alumina powder α-Al_2O_3 > 99.8 wt% type A16SG produced by the Alcoa firm with an average particle size of below 0.5 µm and nano ceria stabilized zirconia CEZ-12 Daiichi Kicenso Kogyo Co LTD were used to process ceramics samples. Agglomerate of the zirconia stabilized with 12 mol% CeO_2 particles with BET surface areas approximately S_{BET}=10.7 m^2/g and average size of 0.4 µm was disintegrated. Processing steps have included: die pressing the powder at 50 MPa, isostatic pressing at 250 MPa, and sintering in air for 2 h at 1923 K in Seco-Warwick furnace in Ceramics and Materials Engineering Department at University of Science and Technology. The three–point bending test (3PB) was carried on the mechanically notched SENB specimens thinned out to the size 1.5 × 4.0 × 50.0 ±0.1mm Total initial crack length was approximately 1.1 mm (Fig.2).

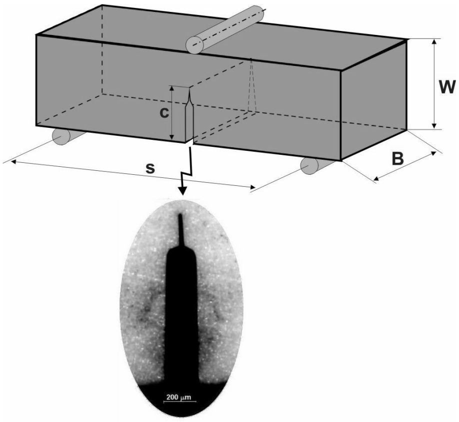

Fig. 2. The SENB specimen with double notch

The controlled crack growth of specimens with double notch was carried with using Zwick testing machine with velocity of 1µm·min^{-1}.The loading of the specimens has been broken at the moment of crack propagation. The procedure has been repeated many times up to obtaining definite increase of crack length.

Individual stages of crack growth at given load has been observed on the computer screen. In situ observation of crack growth from notch up to failure was possible due to special device with microscopical objective heads in vertical configuration [8].

In presented work, a load-relaxation technique method was used for determination of a crack growth velocity (v) as a function of versus stress intensity factor (K_I).The device coupled with CCD camera was fitted to Zwick 1446 testing machine. When the crack length (including length of double notch) has achieved value 2.5 mm the test has been interrupted. Relationship of $K_R = f(c)$ is given by Eq. 2, 3 [9]:

The stress intensity factor K_R was calculated from the Eq.1:

$$K_R = 1.5 \frac{PS}{W^2 B} Y c^{1/2} \tag{2}$$

where: *P* – critical load, *S* – roller distance, *W*–-specimen width, *B*-specimen thickness, *c* – crack length *Y* – geometric function

$$Y = \frac{\sqrt{\Pi}}{(1-\beta)^{\frac{3}{2}}} \left[0.3738\beta + (1-\beta) \sum_{i,j=0}^{4} A_{ij} \beta^i \left(\frac{W}{S} \right)^j \right] \tag{3}$$

where: β is the c/W and A_{ij} are the coefficients given by Fett [10].

The specimens were loaded during three-point bending test, using Zwick testing machine with rate of $1 \mu m \cdot min^{-1}$. The PC computer read the testing machine signals giving the information about loading force and beam deflection. Hence the crack length *c* was calculated by linear-elastic analysis from the compliance of single-edge-notched specimen in three-point bending test as a function of time (*t*). The work-of-fracture (*WOF*) 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 was calculated too [11].

Usefulness of alumina-10 wt% zirconia ceramics with nano 12 mol% CeO_2 stabilized ZrO_2 as a cutting material in operation cutting condition was tested. The tests were carried on the SNGN 120408 T02020 type of inserts during continuous turning of the 45 steel (185 HB). Taking into account initial tests: type and hardness of machined materials the following cutting parameters was accepted:

- cutting velocity v_c =400 m/min
- feed f= 0,16 mm/rev
- cutting depth a_p=1,6 mm

VB_B=0.30 mm of mean abrasive wear resistance on the main flank face was used. The inserts were tested in Metal Cutting and Tools Department of the Institute of Advanced Manufacturing Technology in Cracow.

3. Results

The set of points received during measurement of fracture resistance (K_R) versus crack extension (*c*) has been described by means of linear equation type *y=ax+b,* where *y=*K_R*, x=c, (a)* is a slope coefficient of a straight line, *(b)* is an intersection of *Y*-axis (Fig. 3). The value of (K_R) was determined for appropriated length crack during in situ observation of crack propagation at the top of the notch according to formula 2,3. The analysis of straight line equations type *y* = *ax*+*b* indicates the pronounced character of the *R-curve* which appoints an increasing dependence on fracture resistance with crack extension.

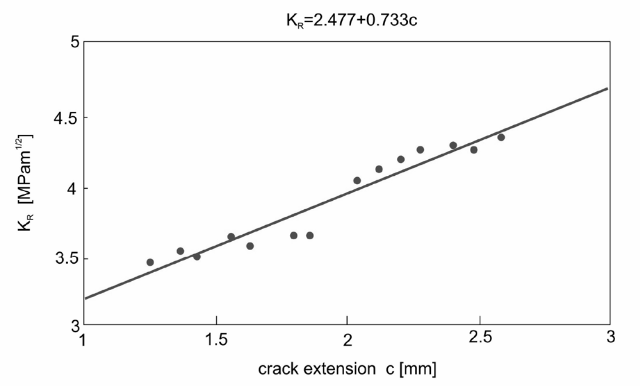

Fig. 3. Crack growth resistance (*R-curve*) for Al_2O_3-10 wt% ZrO_2 ceramics with nano 12 mol% CeO_2 stabilized ZrO_2

Line directional coefficient (a_z) for alumina-10 wt% zirconia ceramics with nano 12 mol% CeO_2 stabilized ZrO_2 is equal 0,733. It can mean that bridging mechanism responsible for existence of R-curve has significant in toughening of the alumina-10wt% zirconia ceramics with nano 12 mol% CeO_2 stabilized ZrO_2. The diagram of the crack growth velocity (v) as a function of stress intensity factor (K_I) in logarithmic system was similar to typical v-K_I curve with three characteristic regions observed for brittle materials (Fig.4).

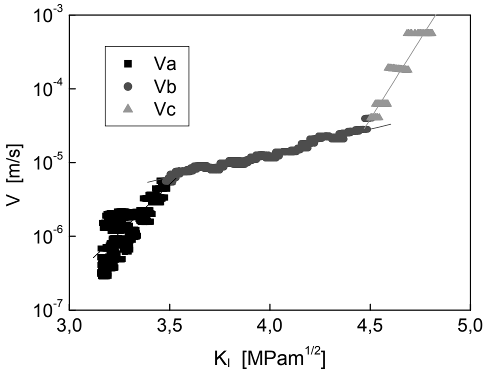

Fig. 4. Stress-intensity factor (K_I) as a function of the crack growth velocity (v) of the Al_2O_3-10 wt% ZrO_2 ceramics with nano 12 mol% CeO_2 stabilized ZrO_2

The curves of alumina ceramics exhibit an increasing character of relationship without ”plateau”. One of the suggestions of a steady-state “plateau” toughness may be reached under conditions where bridges are created and destroyed at the same rate [3].Slow-crack-growth empirical parameter *n* and proportionality constant *logA*, fracture work *WOF*, stress intensity factor for crack initiation K_{I0}, maximum stress intensity factor K_{Imax} determined for the tested composite were presented in Table 1.The Al_2O_3–10 wt% ZrO_2 with

Table1.
Slow crack growth parameters (*n, logA*), fracture work *WOF*, stress intensity factor for crack initiation K_{I0}, critical stress intensity factor K_{Ic} of the tested ceramics

Ceramic material	Fracture work, *WOF* [J/m^2]	Stress intensity factor for crack initiation, K_{I0} [MPa m$^{1/2}$]	Maximum stress intensity factor K_{Imax} [MPa m$^{1/2}$]	*n* Parameter in Eq.3 for region I(v_a),II(v_b),III(v_c) (see Fig.5)	*logA* Proportionality constant in Eq.3 for region I(v_a),II(v_b),III(v_c)	Critical stress intensity factor, K_{Ic} [Mpa m$^{1/2}$]
Al_2O_3–10 wt % $ZrO_{2(nano)}$ with nano 12 mol% CeO_2 stabilized ZrO_2	23.5±5.2	3.21±0.04	5.08±0.09	20.27 (v_a)* 5.87 (v_b) 50.75 (v_c)	-16.33 (v_a) -8.40 (v_b) -37.64 (v_c)	3.96±0.08

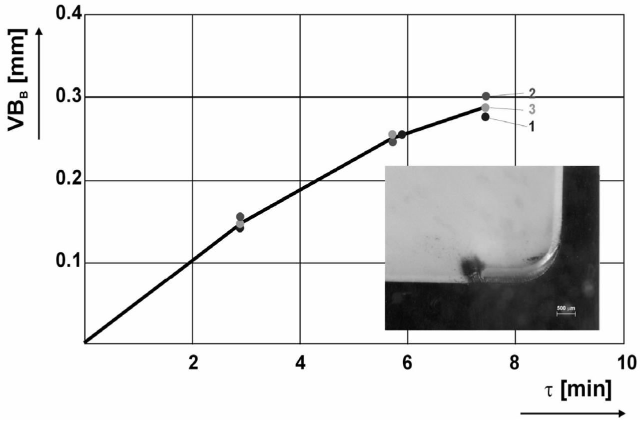

Fig. 5. Mean abrasive wear resistance VB_B measured in defined time distance τ

nano CeO_2 stabilized ZrO_2 ceramics reveal higher value of *WOF* and critical stress intensity factor K_{IC} comparable with the Al_2O_3–10 wt% ZrO_2 with 3 mol% yttria stabilized ZrO_2. However values of K_{I0}, K_{Imax} indicate the higher values than alumina ceramics [12]. Good cutting properties of the tested ceramics based on mean abrasive wear resistance VB_B measured in defined time distance τ allow to use this material as a tool ceramics (Fig.5). The values of slow crack growth parameters *n* show a moderate susceptibility of the Al_2O_3–10 wt% ZrO_2 with nano CeO_2 stabilized ZrO_2 ceramics to SCG.

4. Conclusions

1. Application of the ZrO_{2nano} (12 mol% CeO_2) in alumina–zirconia ceramics improve the fracture toughness in comparison to pure alumina ceramics.
2. Observation of the controlled crack growth in the tested ceramics reveals the existence of increasing *R-curve* in the alumina-zirconia ceramics with nano ceria stabilized zirconia.
3. Alumina-zirconia ceramics with ZrO_{2nano} (12 mol% CeO_2) can be used as a tool materials.

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