

# Investigations of the subcritical crack growth phenomenon and the estimation of lifetime of alumina and alumina–zirconia composites with different phase arrangements

Agnieszka Wojteczko\*, Radosław Lach, Kamil Wojteczko, Zbigniew Pędzich

AGH – University of Science and Technology, Faculty of Materials Science and Ceramics, Department of Ceramics and Refractory Materials, Mickiewicza 30, 30-059 Krakow, Poland

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## ABSTRACT

The Constant Stress Rate Test was used to determine subcritical crack propagation of sintered composites in the  $\alpha$ -alumina-tetragonal zirconia system. Strength measurements were conducted at different stress rates. Material strength depends on the flaw size. Increasing stress rates led to strength increase as a result of shortening of the time of crack growth.

The alumina composite with 5 vol% of zirconia inclusions was prepared as a model of particulate composites with isolated grains of the minor phase. The composite with 15 vol% of zirconia was fabricated as a two-phase material with the minor phase grains content near the percolation point. The third type of a composite containing 35 vol% of zirconia additives was prepared as an example of the composite with typical duplex microstructure. Additionally,  $\alpha$  alumina sinters were prepared as a reference material. The performed experiments allowed us to calculate parameters of slow crack propagation and to construct strength-probability-time diagrams for lifetime prediction of ceramic elements.

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## 1. Introduction

Subcritical crack growth is a phenomenon caused by environmental factors. In general, the presence of water in the crack tip might reduce the amount of energy needed to start cracking. It is particularly dangerous in oxide ceramics, where water attaches to the atoms at the crack surface and saturates the energy by chemisorption [1–4]. The effect of subcritical crack growth occurrence is a material failure at stresses lower than deduced from the  $K_{Ic}$  parameter [5–9]. That is why it is so important to estimate crack velocity to predict lifetime of ceramics [1–4,10,11].

Subcritical crack propagation runs at speeds lower than those when the material cracks catastrophically at the critical value of the stress intensity factor. Crack velocity is expressed by the equation:

$$v = AK_I^n = A^* \left( \frac{K_I}{K_{Ic}} \right)^n \quad (1)$$

where:  $v$  – crack velocity,  $K_I$  – stress intensity factor,  $K_{Ic}$  – fracture toughness,  $n$ ,  $A$ ,  $A^*$  – constants depending on material properties and environmental factors [12].

The parameters describing subcritical crack growth can be estimated either by direct and indirect methods. In the direct methods crack length measurements are required. A disadvantage of these methods is that the performed experiments are time-consuming. In the indirect methods subcritical crack growth parameters are deduced from the strength data. At least thirty specimens per experiment are needed to avoid inaccurate estimation [13,14]. The Constant Stress Rate Test is an example of these kind of methods. Flexural strength measurements are obtained at different stress rates. Material strength depends on the flaw size. If subcritical crack growth occurs, there is more time for a crack to grow at lower stress rates, so the material strength is lower than for higher stress rates. The highest strength value is observed at an inert environment, where there is no influence of corrosive factors [11,15].

The dependence between flexural strength and stress rate is expressed by:

$$\log \sigma_f = \frac{1}{n+1} \log \dot{\sigma} + \log D \quad (2)$$

where  $\sigma_f$  – flexural strength,  $\dot{\sigma}$  – stress rate,  $n$  – subcritical crack growth equation exponent (Eq. (1)),  $D$  – subcritical crack growth parameter, depending on material type and environmental factors.

The experimental results are presented in the form of log-log dependence graphs. When the slope and intercept are known,  $n$

\* Corresponding author.

E-mail address: [agdudek@agh.edu.pl](mailto:agdudek@agh.edu.pl) (A. Wojteczko).

**Table 1**  
Relative density and fracture toughness of alumina and AZ composites.

Material	$d_{rel}$ [%]	$K_{Ic}$ [MPam <sup>0.5</sup> ]
Al <sub>2</sub> O <sub>3</sub>	99.28 ± 0.05	4.34 ± 0.20
AZ05	98.55 ± 0.36	4.83 ± 0.40
AZ15	98.99 ± 0.20	5.88 ± 0.14
AZ35	99.04 ± 0.30	4.84 ± 0.34

and  $D$  parameters can be calculated. In addition,  $A^*$  and  $B$  parameters can be determined from the crack velocity equation (Eq. 1):

$$A^* = \frac{2K_{Ic}^2}{B(n-2)Y^2} \quad (3)$$

$$B = \frac{\alpha \left(10^{\frac{\beta}{\alpha}}\right)}{\sigma_i \left(\frac{1}{\alpha} - 3\right)} \quad (4)$$

where:  $Y$  – shape factor (for circular samples 1,13 [16]),  $\alpha$  – slope,  $\beta$  – intercept and  $\sigma_i$  – strength at inert environment (here at 200 MPa/s).

The Irwin–Griffith formula allows the critical crack length calculation:

$$a_c = \frac{1}{\pi} \left( \frac{K_{Ic}}{Y \cdot \sigma_f} \right)^2 \quad (5)$$

Crack growth velocity is determined by the change of crack length to the change of time for different stress rates. Then the calculation of a corresponding stress intensity factor is possible (Eq. (1)).

Subcritical crack growth parameters are used to predict the lifetime of ceramics. In most cases, ceramic components are used under static load, while the Constant Stress Rate method is a dynamic test. Therefore, the calculations between dynamic and static conditions are required. Time to failure ( $t_f$ ) for static load is determined from the relation:

$$t_{f,static} = \frac{t_{f,dynamic}}{(n+1)} \quad (6)$$

Then the estimation of stress in static load at a given time ( $t$ ) is possible:

$$\sigma_{f,static} = \sigma_{f,dynamic} \left( \frac{t_{f,static}}{t} \right)^{\frac{1}{n}} \quad (7)$$

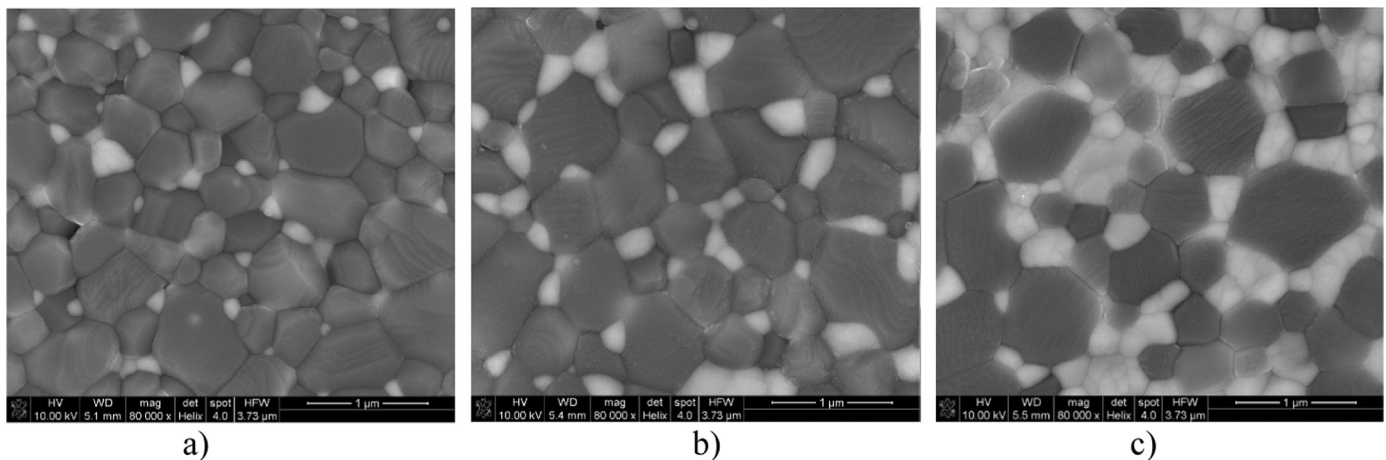
To predict the material behavior under service, the strength-probability of failure-time diagrams (SPT) have been constructed. It is possible to estimate lifetime by means of proof testing, but when the ceramic element is going to be used for a long time, it would be very time-consuming, so taking more measurements could be problematic. Thus, using a larger amount of samples, with preexisting flaw distribution, in Constant Stress Rate method more reliable results can be statistically obtained [17].

## 2. Experiment

The materials investigated in presented work were:  $\alpha$ -alumina (TAIMEI Chemicals, TM-DAR) sinters and  $\alpha$ -alumina–tetragonal zirconia (TOSOH, TZ-3Y) composites with 5 (AZ05), 15 (AZ15) and 35 vol% (AZ35) of inclusions. The experiment conditions were established at 20 °C and in humidity in the range of 40–50%. Disc-shaped specimens (diameter 18 mm, thickness 1.5 mm) were formed by uniaxial pressing at 50 MPa and then isostatically repressed at 300 MPa. Sintering of pure alumina and AZ05 composite was conducted at 1400 °C and for AZ15 and AZ35 at 1500 °C to gain high densification which was determined as a reference of density measured by the Archimedes method (at 21 °C) to the theoretical values (assuming that for alumina  $d_{theoAl_2O_3} = 3.99$  g/cm<sup>3</sup> and for zirconia  $d_{theoZrO_2} = 6.10$  g/cm<sup>3</sup>). Density measurement were performed using 10 samples for each type of investigated materials. Fracture toughness was determined in three-point bending of notched beams, utilizing 6 samples for each type of material. Microstructure observation was obtained at Nova Nano SEM 200 scanning electron microscope. Strength measurements were performed with Zwick/Roell 2.5 device, applying the biaxial-loading method [18] at four stress rates: 0.1, 1, 10 and 200 MPa/s. The number of samples used for strength tests was 30 for each stress rate.

## 3. Results

The applied sintering procedures allowed a relatively high densification of investigated materials to be achieved. Fracture toughness was higher for the composites than that measured for the pure alumina. The highest value was determined for the composite with 15 vol% of inclusions (Table 1). It corresponds to the literature data [19,20] which indicated that transformability of zirconia from tetragonal phase to the monoclinic one is the highest for the materials containing 10–15 vol% of zirconia in the alumina matrix. This phenomenon increases most effectively fracture



**Fig. 1.** SEM images of alumina (dark phase)-zirconia (light phase) composites with 5 (a), 15 (b) and 35 vol% (c) of inclusions, respectively (from left to right side).

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