Processing and Application of Ceramics 9 [4] (2015) 187–191
DOI: 10.2298/PAC1504187W

Subcritical crack growth in oxide and non-oxide ceramics using the Constant Stress Rate Test☆

Agnieszka Wojteczko*, Radosław Lach, Kamil Wojteczko, Paweł Rutkowski, Dariusz Zientara, 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

Received 6 November 2015; Received in revised form 16 December 2015; Accepted 21 December 2015

Abstract

Fracture toughness is one of the most important parameters for ceramics description. In some cases, material failure occurs at lower stresses than described by K_{Ic} parameter. In these terms, determination of fracture toughness only, proves to be insufficient. This may be due to environmental factors, such as humidity, which might cause subcritical crack propagation in a material. Therefore, it is very important to estimate crack growth velocities to predict lifetime of ceramics used under specific conditions. Constant Stress Rate Test is an indirect method of subcritical crack growth parameters estimation. Calculations are made by using strength data, thus avoiding crack measurement. The expansion of flaws causes reduction of material strength. If subcritical crack growth phenomenon occurs, critical value of crack lengths increases with decreasing stress rate due to longer time for flaw to grow before the critical crack propagation at K_{Ic} takes place. Subcritical crack growth phenomenon is particularly dangerous for oxide ceramics due to chemical interactions occurring as a result of exposure to humidity. This paper presents results of Constant Stress Rate Test performed for alumina, zirconia, silicon carbide and silicon nitride in order to demonstrate the differences in subcritical crack propagation phenomenon course.

Keywords: *alumina, zirconia, silicon carbide, silicon nitride, subcritical crack growth, constant stress rate*

I. Introduction

In many ceramics crack does not propagate only at applied stress at K_{Ic}, but well below that value. Crack growth at these values is much slower and difficult to detect, but it leads to decrease in material strength and delayed failure [1–6] even if it was under strength value determined in standard test methods [7–11]. It is an environmentally induced phenomenon. It may vary for different corrosive factors and material types. Water impact on material fracture appears to be particularly dangerous in oxide ceramics used in an environment of high humidity. Water molecules align to the cations at the crack tip surface, with their lone pair orbitals, creating hydrogen bonding. Strained bonds in the material are the enhancing factor. This mechanism leads to bond rupture which causes subcritical crack growth induced by stress corrosion [3,12].

Types of tests, used in subcritical crack growth phenomenon description, can be divided into two groups. The first one is a static loading method based on crack growth observations. In this kind of test, measurements might take long time. In discussed phenomenon cracking might start with velocities about 10^{-12} m/s. It means that crack growth of 1 μm takes over one week. In the second type of tests, subcritical crack growth parameters are determined on the basis of strength data [13]. Strength measurements are performed at different stress rates to allow stress increase in the material for various periods of time. The more time is given, the higher decrease of strength occurs. What is more, there is no need for specific sample preparation, like polishing or introducing cracks. The tests are conducted on sintered ceramics with preexisting flaws (either on the surface or in the material volume) [14]. Therefore, these conditions are closer to the actual in the ceramic components.

☆Paper presented at *11th Conference for Young Scientists in Ceramics - SM-2015*, Novi Sad, Serbia, 2015

*Corresponding author: tel: +48 69 663 7958,
fax: +48 12 633 4630, e-mail: *agdudek@agh.edu.pl*

Relative shortening of testing time and ease of sample preparation (when compared to static fatigue) allows for examination of higher amount of samples, which is statistically advantageous [12]. Studies have shown that the amount of thirty samples is minimal until the result is statistically correct [9,15]. In contrast, the use of larger quantities is not necessary because it does not affect significantly the changes in the accuracy of results [15,16].

The aim of the paper is the presentation of parameters determined by Constant Stress Rate method for oxide and non-oxide ceramics to demonstrate the differences in subcritical crack growth course.

II. Experimental

The idea was to fabricate two oxide and two non-oxide materials for comparative purposes. In the first group α-alumina samples (TAIMEI Chemicals, TM-DAR) sintered at 1400 °C for 2 hours, and tetragonal zirconia samples (TOSOH, TZ-3Y) sintered at 1500 °C for 2 hours, were prepared. As representatives of non-oxide ceramics, silicon carbide (SIKA FCP 15, Saint-Gobain) sintered at 1800 °C (10 °C/min), 1800–2150 °C (5 °C/min) and 2150 °C for 1 hour, and silicon nitride samples (Si_3N_4 H.C. STARCK and oxide additives in 4 wt.% Y_2O_3 and 6 wt.% of Al_2O_3 TM-DAR), sintered at 1800 °C for 2 hours, were obtained. The sintered samples were disc-shaped with thickness about 1.1–1.2 mm and 13–14 mm in diameter.

Densification of each material was calculated as a reference of density measured in Archimedes method (at 21 °C) to the theoretical values (d_{ZrO_2} = 6.10 g/cm^3; d_{SiC} = 3.21 g/cm^3; $d_{Si_3N_4}$ = 3.21 g/cm^3; $d_{Al_2O_3}$ = 3.99 g/cm^3; $d_{Y_2O_3}$ = 5.01 g/cm^3). Relative density for silicon carbide samples was calculated considering content of phases arising due to oxides addition. Fracture toughness was obtained in SENB method.

Subcritical crack growth parameters were estimated in Constant Stress Rate Test. Calculations were based on power law equation for subcritical crack growth velocity:

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

where v is crack velocity, K_I is stress intensity factor, K_{Ic} is fracture toughness, n is stress corrosion susceptibility parameter, A and A^* are constant depending on material properties and environmental factors, respectively [17,18].

In this method no crack length measurement is required. Estimations are carried out by using strength data. Strength measurements were performed by using Zwick/Roell 2.5 with biaxial loading support. Four stress rates were used: 0.1, 1, 10 and 200 MPa/s. If there is no influence of the environment (no water impact), strength is called inert. It occurs at high stress rates or in inert environments. In this case, the tests were made at 20 °C and humidity in range of 40–50% and the strength results at stress rate of 200 MPa/s were found to be inert [4,19,20]. Dependence between strength and stress rate is shown by:

$$\log \sigma_f = \frac{1}{n+1} \log \dot{\sigma} + \log D \tag{2}$$

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

Strength measurement results are presented on logarithmic dependence graphs, in which the designation of the regression equation allows to calculate n and D parameters. To calculate other parameters from Eq. 1, the following formulas were used:

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

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

where Y is shape factor (for circular samples 1.13 [21]), α is slope, β is intercept and σ_i is inert strength [14,19].

Irwin-Griffith formula allows critical crack length calculation [22]:

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

By expressing crack velocity as a change of crack length in time and knowing needed parameters, calculation of corresponding stress intensity factor is possible [14].

III. Results and discussion

The results of density and fracture toughness were summarized in Table 1. Sintering of the alumina and zirconia lead to high densification, while densification of both non-oxide ceramics was lower, however, it was considered to be sufficient to conduct experiments.

Figures 1 and 2 demonstrate that subcritical crack growth occurs in both oxide materials. It is proved by increase in material strength for increasing stress rates. It can be explained by reduction of testing time for higher stress rates, leading to reduction of crack growth.

For the silicon nitride (Fig. 3) and silicon carbide (Fig. 4) no increase of strength with increasing stress rate was observed. For confidence, additional strength measurements at 0.01 MPa/s for the silicon carbide were

Table 1. Results of relative density and fracture toughness

Material	d_{rel} [%]	K_{Ic} [MPa·m$^{1/2}$]
Al_2O_3	99.28 ± 0.05	4.34 ± 0.20
ZrO_2	99.96 ± 0.01	6.10 ± 1.14
SiC	95.80 ± 0.13	6.36 ± 2.02
Si_3N_4	96.96 ± 0.53	5.26 ± 0.87

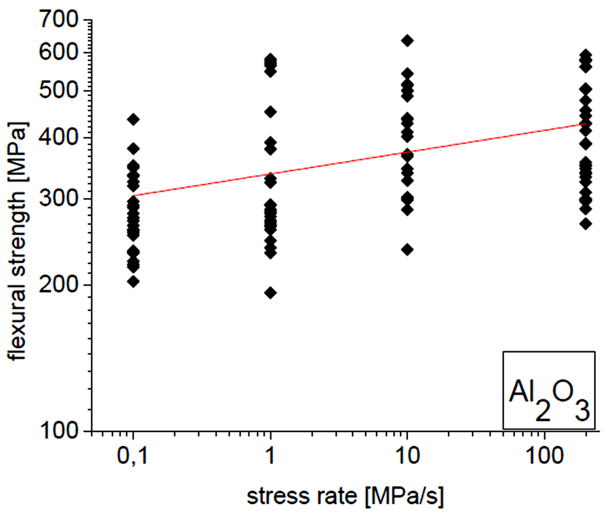

Figure 1. Log flexural strength - log stress rate dependence graph of sintered α-alumina

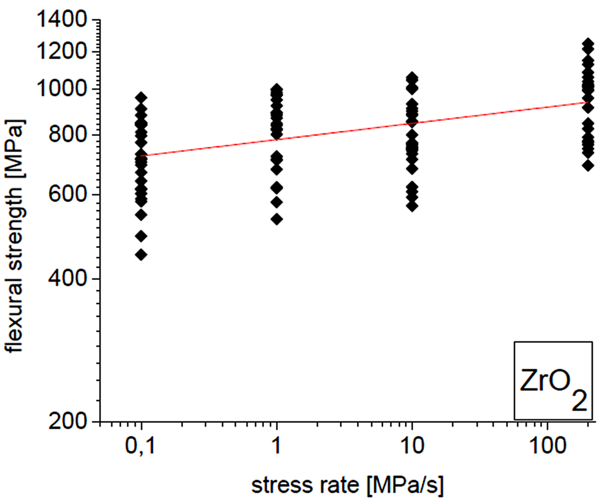

Figure 2. Log flexural strength - log stress rate dependence graph of sintered tetragonal zirconia

made. This was to prolong loading to see if there is any effect of water. The measurements showed that these strengths were on the same level as others. It may be concluded, that for the non-oxide materials tested in presented conditions (20 °C, 40–50% RH) no subcritical crack growth occurred.

Further calculations were possible only for oxide sintered ceramics, in which subcritical crack propagation occurred. Using the slope and intercept of fitting line for strength - stress rate dependences n and D parameters were calculated from Eq. 2 (Table 2). For the zirconia n value was higher. That means that change of material strength for different stress rate was less significant.

Table 2. Values of n and D parameters calculated for alumina and zirconia

Material	n [-]	D [$MPa^{n/n+1} \cdot s^{1/n+1}$]
Al_2O_3	21.83	337.87
ZrO_2	28.30	783.70

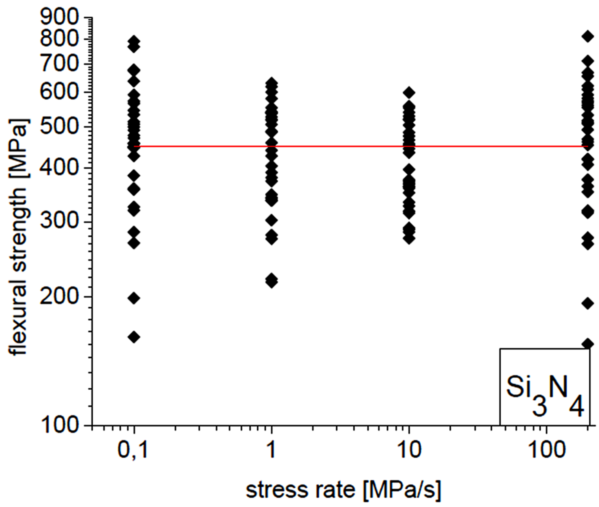

Figure 3. Log flexural strength - log stress rate dependence graph of sintered silicon nitride

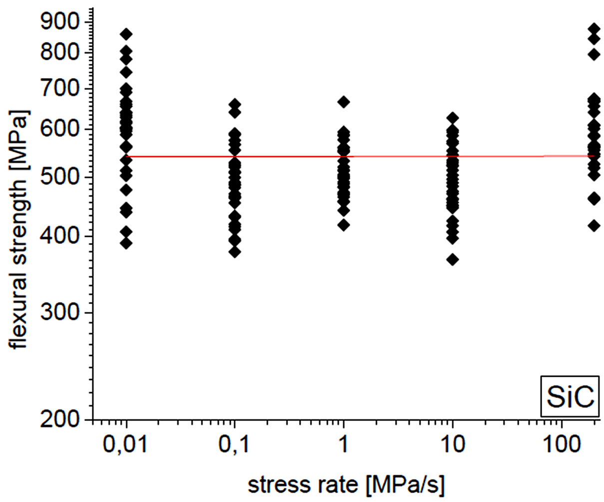

Figure 4. Log flexural strength - log stress rate dependence graph of sintered silicon carbide

Crack velocities and corresponding stress intensity factor values (estimated as it was explained before) combined with the knowledge of n parameters (inducing the slope) allowed to create crack velocity *vs.* K_I/K_{Ic} plot (Fig. 5). It shows that for the alumina, cracking starts at relatively low K_I/K_{Ic} ratio. It may be perceived as a smaller subcritical crack propagation resistance of the alumina compared to the zirconia.

IV. Conclusions

The presented study confirmed that the phenomenon of subcritical crack propagation occurred in sintered oxide ceramic as opposed to non-oxide. This conclusion was made on the basis of log flexural strength - log stress rate dependence graphs. For the investigated alumina and zirconia samples there was noticeable increase of strength for increasing stress rates. It means that when the material is loaded for a longer time in corrosive environment (in this case 20 °C and 40–50% RH)

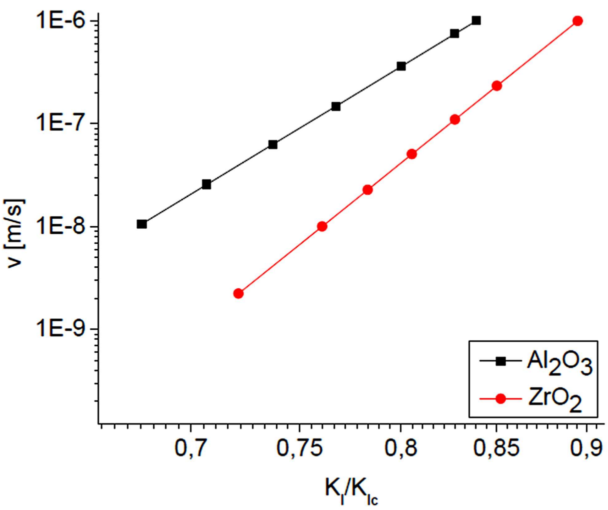

Figure 5. Log crack velocity - log K_I/K_{Ic} dependence plot for sintered alumina and zirconia

its strength decreases as a result of expanded flaw sizes. However, this was not observed for both non-oxide materials - SiC and Si_3N_4. For every stress rate strength value was at the same level. Even if the measurement time was prolonged for SiC, by using 0.01 MPa/s stress rate, no change occurred. This indicates that these materials are subcritical crack growth resistant under presented conditions.

Constant Stress Rate Test allowed estimating subcritical crack propagation parameters using strength data. It was possible for the alumina and zirconia. Change of material strength generated the slope of fitting line which indicated *n* parameter. This case shows that for the material with higher n value (zirconia) cracking starts at higher K_I/K_{Ic} values compared to the alumina. This means that zirconia has lower susceptibility for subcritical cracking. The reason of this phenomenon might be higher energy of chemical bonding in zirconia and well-known input of polymorphic transformation (tetragonal to monoclinic) in energy dissipation on the crack tip.

Acknowledgemets: The work was supported by the Polish State Ministry of Science and Higher Education under AGH University work no. 11.11.160.617.

References

1. A.H. De Aza, J. Chevalier, G. Fantozzi, M. Schehl, R. Torrecillas, "Crack growth resistance of alumina, zirconia and zirconia toughened alumina ceramics for joint prostheses", *Biomater.*, **23** (2002) 937–945.
2. Ch. Benaqqa, J. Chevalier, M. Saadaoui, G. Fantozzi, "Slow crack growth behaviour of hydroxyapatite ceramics", *Biomater.*, **26** (2005) 6106–6112.
3. J.A. Salem, M.G. Jenkins, "The effect of stress rate on slow crack growth parameter estimates", pp. 213–227 in *Fracture Resistance Testing of Monolithic and Composite Brittle Materials, ASTM STP 1409*, American Society for Testing and Materials, 2002.
4. J.A. Griggs, S.M. Alaqeel, Y. Zhang, A.W. Miller, Z. Cai, "Effects of stress rate and calculation method on subcritical crack growth parameters deduced from constant stress-rate flexural testing", *Dental Mater.*, **27** (2011) 364–370.
5. B. Taskonak, J.A. Griggs, J.J. Mecholsky, J.H. Yan, "Analysis of subcritical crack growth in dental ceramics using fracture mechanics and fractography", *Dental Mater.*, **24** (2008) 700–707.
6. Z. Pędzich, "Sub-critical crack propagation in particulate composites with Y-TZP matrix" (in Polish), *Compos. Theory Practice*, **5** (2005) 86–90.
7. ASTM Standard, F-394-78, *Standard Test Method for Biaxial Flexure Strength (Modulus of Rupture) of Ceramic Substrates.*
8. ASTM Standard, C1499-08, *Standard Test Method for Monotonic Equibiaxial Flexural Strength of Advanced Ceramics at Ambient Temperature.*
9. ASTM Standard, C1161-02c, *Standard Test Method for Flexural Strength of Advances Ceramics at Ambient Temperature.*
10. D.K. Shetty, A.R. Rosenfield, G.K. Bansal, W.H. Duckworth, "Biaxial fracture studies of a glass-ceramic", *J. Am. Ceram. Soc.*, **64** [1] (1981) 1–4.
11. A. Börger, P. Supancic, R. Danzer, "The ball on three balls test for strength testing of brittle discs: Stress distribution in the disc", *J. Eur. Ceram. Soc.*, **22** (2002) 1425–1436.
12. T.A. Michalske, S.W. Freiman, "A molecular mechanism for stress corrosion in vitreous silica", *J. Am. Ceram. Soc.*, **66** [4] (1983) 284–288.
13. S.M. Wiederhorn, "Subcritical crack growth in ceramics", pp. 613-646 in *Fracture Mechanics of Ceramics*, Eds. R.C. Bradt *et al.*, Plenum Press, New York, 1974.
14. R. Bermejo, P. Supancic, C. Krautgasser, R. Morrell, R. Danzer, "Subcritical crack growth in low temperature co-fired ceramics under biaxial loading", *Eng. Fract. Mech.*, **100** (2013) 108–121.
15. J.I. McCool, "Statistical error in crack growth parameters deduced from dynamic fatigue tests", *Int. J. Fatigue*, **26** (2004) 1207–1215.
16. M. Cheng, W. Chen, "Measurement and determination of dynamic biaxial flexural strength of thin ceramic substrates under high stress-rate loading", *Int. J. Mech. Sci.*, **47** (2005) 1212–1223.
17. ASTM Standard, C1368-06, *Standard Test Method for Determination of Slow Crack Growth Parameters of Advanced Ceramics by Constant Stress-Rate Flexural Testing at Ambient Temperature.*
18. M. Steen, "Fractography and fracture mechanics property assessment of advanced structural ceramics", *Int. Mater. Rev.*, **44** [5] (1999) 165–216.
19. R. Lach, A. Dudek, Z. Pędzich, "Determination of subcritical crack propagation parameters for ZrO_2 sinters using Constant Stress Rate test" (in Polish), *Materiały Ceramiczne*, **67** [1] (2015) 56–61.
20. J.B. Wachtman, W.R. Cannon, M.J. Matthewson,

Subcritical Crack Propagation, Mechanical Properties of Ceramics, Second Edition, John Wiley & Sons, 2009.
21. G.D. Quinn, *Fractography of Ceramics and Glasses*, NIST, Special Publication 960-16, 2007.
22. A.A. Griffith, "The phenomenon of rupture and flow in solids", *Philos. Trans. Roy. Soc. London*, **221** (1921) 163–198.