Determination of the subcritical crack growth parameters in ZrO_2-Al_2O_3 composite under different environmental conditions

Określenie parametrów pękania podkrytycznego kompozytu z układu ZrO_2-Al_2O_3 w różnych warunkach środowiskowych

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The presence of the water, also in the form of water vapour, can significantly decrease the ceramic material strength. It occurs due to subcritical crack growth phenomenon. Especially susceptible to the stress corrosion are oxide materials. Test were carried out by the Constant Stress Rate method, in the air and the water environment, for the ZrO_2 – 10 vol. % Al_2O_3 composite.
KEYWORDS: subcritical crack growth, Constant Stress Rate test, tetragonal zirconia, corundum

Obecność wody, również w postaci pary wodnej, może znacząco wpływać na obniżenie wytrzymałości materiałów ceramicznych. Ma to miejsce z powodu występowania zjawiska pękania podkrytycznego. Szczególnie podatne na korozję naprężeniową są materiały tlenkowe. Badania przeprowadzono metodą stałego przyrostu naprężeń, w powietrzu oraz środowisku wodnym, dla kompozytu ZrO_2 – 10 obj. % Al_2O_3.
SŁOWA KLUCZOWE: pękanie podkrytyczne, test stałego przyrostu naprężeń, tetragonalny tlenek cyrkonu, korund

The subcritical crack growth of ceramic materials is a particularly dangerous phenomenon because of the material strength reduction. It preludes the material cohesiveness loss and occurs at stresses lower than those when the K_{Ic} is reached, but when they are active for a long time [1]. Water is supposed to accelerate the crack propagation because of chemical reaction which leads to reduction of the surface energy at the crack tip. Oxide materials are especially susceptible to this kind of reaction [2, 3].
Zirconia-alumina composites are known for their very good properties and commonly used as parts of mechanical devices. The aim of the paper was to calculate subcritical crack growth parameters for tetragonal zirconia with 10 vol. % of α-alumina inclusions (ZA10). The experiment was performed by using the Constant Stress Rate test in air and water in order to determine the environmental impact on the crack propagation.

Materials and methods

■ **Samples preparation.** The samples were made by wet mixing of commercial zirconia (TZ-3Y, TOSOH) and α-alumina (TM-DAR, TAIMEI) powders in attritor using 2 mm zirconia grinding media for 30 minutes. Disc-shaped specimens were formed by the uniaxial pressing (50 MPa) and isostatically re-pressed (300 MPa). For the K_{Ic} measurement, bars with notches were prepared. Sintering of composite samples was conducted at 1500 ºC for 1 hour with the temperature increase rate 3 ºC/min.

Results and discussion

■ **Samples characteristics.** Sintering lead to the high densification of samples (98.90%), calculated as a relation of density obtained in Archimedes method at 21 ºC to the theoretical value (assuming that alumina density is 3.99 g/cm^3 and zirconia density is 6.10 g/cm^3). Fracture toughness was determined by three-point bending of notched bars [4] and was 4.27 MPa m$^{0.5}$.

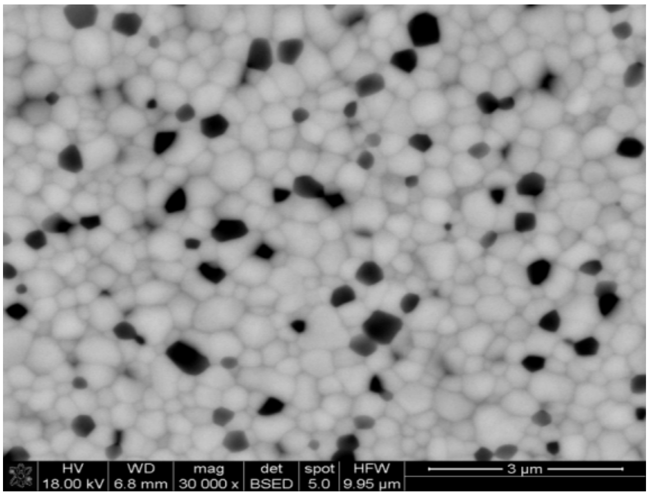

Fig. 1. SEM image of ZA10 composite

Microstructure observation was made using scanning electron microscope Nova Nano SEM 200. SEM image shows good densification and homogenity of composite. Alumina inclusions are seperated in the zirconia matrix. Flexural strength measurements were made by biaxial loading method [5] of disc-shaped specimens (about 13.40 mm diameter and 1.07 mm thickness) at four stress rates for measurements in the air: 0.1, 1, 10 and 200 MPa/s and two extreme stress rates (0.1 and 200 MPa/s) in water. If the strength is growing for each subsequent stress rate, it is certain that the situation would be the same in the aqueous environment. Thus, it is sufficient to make measurements for two extreme stress rates only [6].Tests were conducted at 20 ºC and humidity in the range of

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40÷50%. The amount of thirty samples was found to be sufficient to obtain high statistical accuracy [7]. Results received in biaxial loading method were presented on the flexural strength – stress rate dependence logarithmic graphs (Fig. 2 and 3). Increase of the strength for increasing stress rates proves the occurence of the subcritical crack growth phenomenon, because reducing the measurement time gives flaws less time to grow.

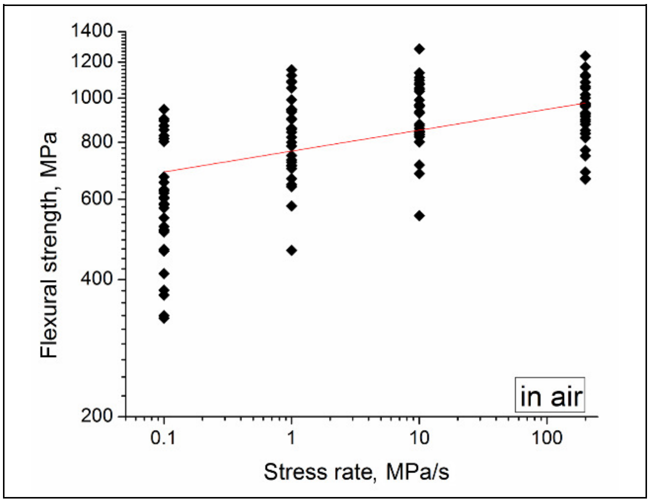

Fig. 2. Log flexural strength – log stress rate dependence graph of ZA10 in air

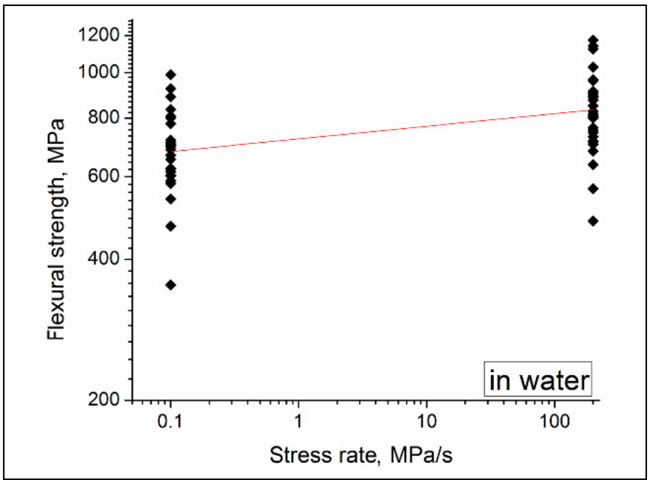

Fig. 3. Log flexural strength – log stress rate dependence graph of ZA10 in water

That characteristic change of strength was observed in both environments. It means that 40÷50% humidity is enough to have an impact of the environmental factors on the crack propagation.

Dependence between flexural strength and stress rate is expressed by:

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

where σ_f – flexural strength, σ – stress rate, n – subcritical crack growth equation exponent, D – subcritical crack growth parameter depending on material type and environmental factors [3].

TABLE. The exponent of subcritical crack propagation equation (*n* parameter)

Environment	n [-]	D [MPa$^{n/n+1}$s$^{n/n+1}$]
air	22.26	763.84
water	37.46	719.45

The *n* value, shown in the table, gives the information about the subcritical crack growth susceptibility. It determins the slope of the crack velocity (v) vs. K_I/K_{Ic} dependence (Fig. 4).

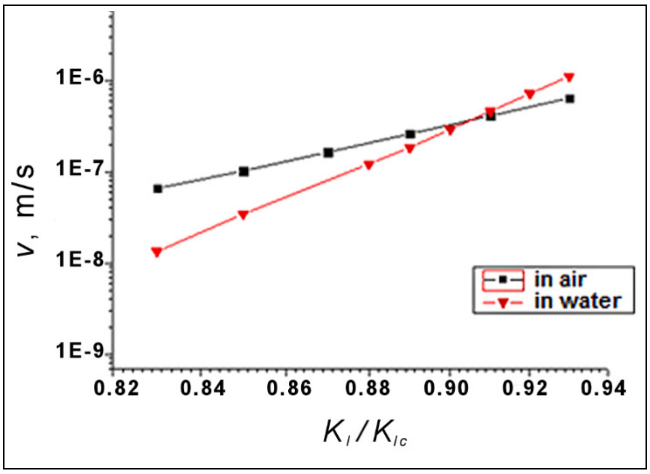

Fig. 4. A plot of crack velocity vs. K_I/K_{Ic} dependence

Conclusions

Calculations performed for data obtained in the experiment show that in the range of stress intensity factor K_I close to the K_{Ic} the tested composite cracks in a similar manner in the aqueous medium and in the air. What is more, it can be even observed that the exponent *n*, providing a tendency to the subcritical crack growth, in the presented range is higher for the water (so its tendency to crack is lower) than for the air. However, this difference is small. The results are important for practical applications and can be used in a lifetime prediction of zirconia – 10 vol.% of alumina composite parts.

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