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COMPOSITES THEORY AND PRACTICE

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ESTIMATION OF LIFETIME OF ZIRCONIA AND ZIRCONIA-ALUMINA COMPOSITES USING CONSTANT STRESS RATE DATA

Ceramic failure, at stresses lower than those when K_{Ic} is reached, might be an effect of subcritical crack propagation. It occurs when the material is in service in the presence of water (even water vapour) for a long time. If this phenomenon leads to limiting its useful life, it is important to predict the lifetime of a ceramic component under specific conditions. Dynamic tests are suitable to determine subcritical crack growth parameters. Calculations are made to determine material behaviour under static loading on the basis of strength data. The paper presents the results for zirconia-alumina composites with 5, 15 and 50 vol.% inclusions to compare materials with different types of microstructures. Pure zirconia sintered bodies were used as the reference material.

Keywords: **tetragonal zirconia, zirconia-alumina composites, subcritical crack growth, constant stress rate test, lifetime determination**

WYZNACZANIE CZASU ŻYCIA SPIEKÓW Z TLENKU CYRKONU ORAZ KOMPOZYTÓW TLENEK CYRKONU-TLENEK GLINU NA PODSTAWIE WYNIKÓW TESTU STAŁEGO PRZYROSTU NAPRĘŻEŃ

Zniszczenie materiałów ceramicznych, przy obciążeniach niższych niż te, gdy osiągane jest K_{Ic}, może być efektem pękania podkrytycznego. Ma to miejsce, gdy materiał używany jest w obecności wody (również w postaci pary wodnej) przez długi czas. Jeśli to zjawisko prowadzi do ograniczenia czasu użytkowania, istotne jest, aby przewidzieć czas przeżycia elementu ceramicznego działającego w danych warunkach. Odpowiednie do wyznaczania parametrów pękania podkrytycznego są testy dynamiczne. Obliczenia wykonywane są w celu przewidzenia zachowania materiału pod stałymi obciążeniami na podstawie wyników wytrzymałości. Praca przedstawia wyniki dla kompozytów tlenek cyrkonu-tlenek glinu z 5, 15 oraz 50 obj.% wtrąceń w celu porównania materiałów o różnych typach mikrostruktur. Spieki z czystego dwutlenku cyrkonu zostały zbadane jako materiał odniesienia.

Słowa kluczowe: **tetragonalny dwutlenek cyrkonu, kompozyty dwutlenek cyrkonu-tlenek glinu, test stałego przyrostu naprężeń, wyznaczanie czasu przeżycia**

INTRODUCTION

Zirconia-alumina composites are commonly used due to their advantageous properties, for example relatively high strength and fracture toughness. However, in materials exposed a long time to loading, corrosion resistance plays a significant role. Oxide materials are particularly susceptible to the influence of water in a humid environment, which substantially affects the mechanical properties. Water attaches to the atoms at the crack surface and this, connected with stress present during loading, leads to slow crack propagation [1, 2]. Material failure occurs not at the same time as the loading was applied, but at stresses lower than predicted in standard strength tests. Therefore, subcritical crack growth investigations seem to be very important [1, 3-7].

Subcritical crack growth parameters are deduced from experimental data. There are a few methods to determine v-K_I behaviour. If an experiment is conducted at constant stresses the method is called a static fatigue test. Information about subcritical crack growth is received directly from the crack length measurements of materials that have been loaded for some time [6]. The dynamic fatigue method, also known as the Constant Stress Rate test, is an alternative for these time-consuming experiments. The materials are tested at a few stress rates. Differences in measurement time cause differences in strength if the subcritical crack growth phenomenon occurs. Increasing the time gives more time for flaws to grow. As an effect, a decrease in strength is observed, while the stress rate increases.

Subcritical crack propagation parameters are deduced from strength data [8-13]. The results are often presented on plots showing the dependence between the crack velocity and stress intensity factor. Many studies have lead to the creation of three characteristic areas [4, 8], but for lifetime prediction a simplified form with only one straight line is convenient (Fig. 1) [14]. This substantially reduces the time of the experiment.

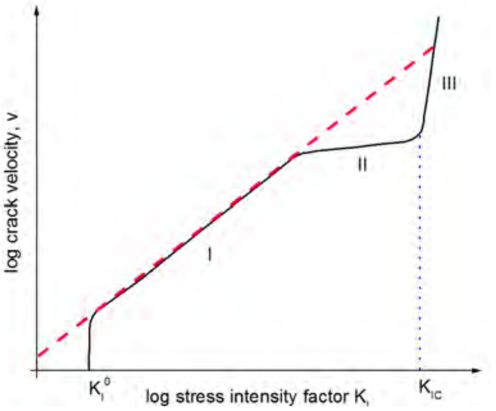

Fig. 1. Simplified form of v vs. K_I plot used in lifetime predictions

Rys. 1. Uproszczona forma wykresu zależności v od K_I używana w przewidywaniu czasu przeżycia

EXPERIMENT

This work presents the results for zirconia (TOSOH, 3Y-TZ)-alumina (TAIMEI, TM-DAR) composites with 5, 15 and 50% volume contents of inclusions and for pure zirconia as a reference. The composite powders were obtained by means of the wet mixing process (iso-propanol) in an attritor for 1 hour with 2 mm grinding balls. Dried powders were formed into disc-shaped specimens using uniaxial pressing at 50 MPa and isostatically re-pressed at 300 MPa. Sintering was conducted at 1500°C for 2 hours at a heating rate 3°C/min. Density was determined using the Archimedes method and relative density was calculated assuming that the theoretical densities were for zirconia $d_{\text{theoZrO2}} = 6.10$ g/cm^3 and for alumina $d_{\text{theoAl2O3}} = 3.99$ g/cm^3. Fracture toughness was measured by the three point bending of beams with narrow notches according to [15]. In this work we made the notches using a diamond wheel 150 μm in thickness. The microstructures were observed using a Nova Nano SEM 200 scanning electron microscope. Strength measurements were carried out by the biaxial loading method at four different stress rates: 0.1, 1, 10 and 200 MPa/s. The tests were conducted at 20°C and 40÷50% air humidity. For facilitation, the composites were designated in the paper as ZA05, ZA15 and ZA50 regarding the alumina inclusion content.

RESULTS

Three types of zirconia-alumina composite microstructures were chosen for the experiment. The difference was due to the alumina inclusion content. The SEM images (Fig. 2) show that for the 5 vol.% content, the alumina is well dispersed in the zirconia matrix, 15 vol.% is where the inclusions start to connect with each other and the addition of 50 vol.% alumina results in creating a duplex microstructure.

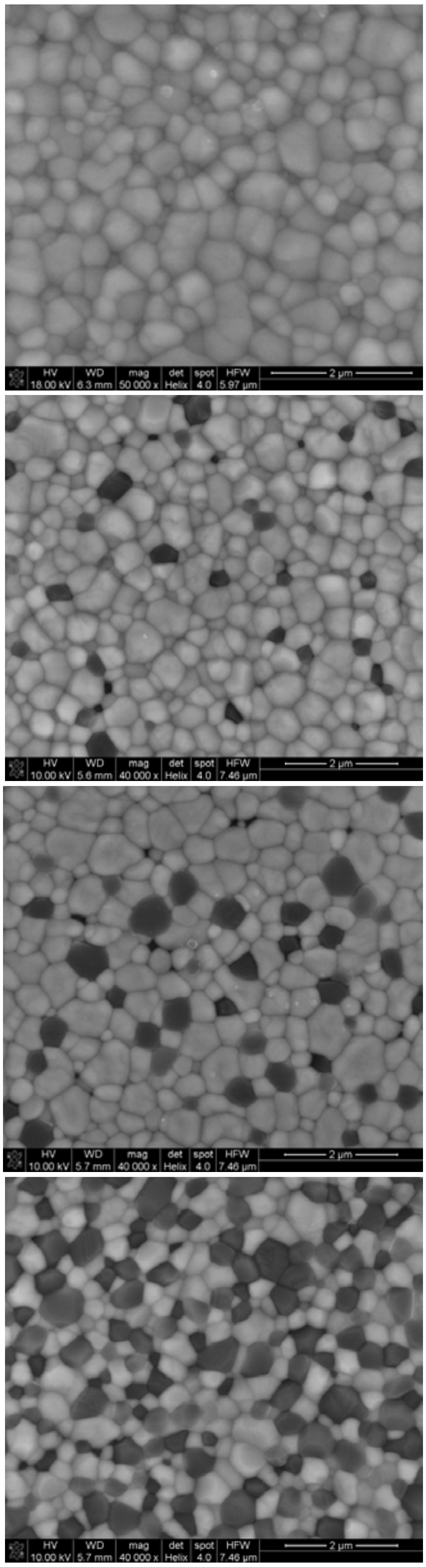

Fig. 2. SEM images of zirconia-alumina composites. In order: ZrO_2, ZA05, ZA15 and ZA50

Rys. 2. Obrazy SEM tlenku cyrkonu oraz kompozytów tlenek cyrkonu-tlenek glinu. W kolejności: ZrO_2, ZA05, ZA15 and ZA50

High densification of the zirconia and zirconia-alumina composites (over 98%) was obtained. Tetragonal zirconia is well known for its high fracture toughness thanks to polymorphic transition. The tests show that the 5 vol.% addition of alumina inclusions reduces the resistance to cracking. However, a higher amount of inclusions leads to improvement and the highest result is for the 15 vol.% alumina (Table 1).

TABLE 1. **Densification and fracture toughness of zirconia and zirconia-alumina sintered bodies**

TABELA 1. **Zagęszczenie oraz odporność na kruche pękanie spieków z tlenku cyrkonu oraz kompozytów tlenek cyrkonu-tlenek glinu**

Material	ρ_{rel} [%]	K_{Ic} (SENB) [MPa·m$^{1/2}$]
ZrO_2	99.96 ±0.01	6.10 ±1.14
ZA05	98.23 ±0.13	5.94 ±0.77
ZA15	98.26 ±0.11	7.23 ±0.78
ZA50	98.96 ±0.16	6.57 ±0.11

Estimation of the subcritical crack growth parameters (Table 2) was possible due to the strength measurements at four stress rates: 0.1, 1, 10, 200 MPa/s, thirty samples at each rate (for statistic accuracy). The results of these tests were presented on *log strength* versus *log stress rate* dependence plots (Fig. 3). For each material an increase in strength with an increasing stress rate was observed which proves that subcritical crack growth occurred. The most significant change was observed for the pure zirconia. It suggested that the addition of alumina particles improved subcritical crack propagation resistance.

The value of the *n* parameter is deduced from the slope of the *strength* versus the *log stress rate* dependence using the equation:

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

For major changes in strength, the *n* value is lower, hence this parameter exhibits resistance to subcritical crack propagation. In every composite this parameter is higher than for the pure zirconia. The highest value was obtained by the composite with 15 vol.% alumina inclusions, therefore it seems that for this material the stress corrosion susceptibility is the lowest.

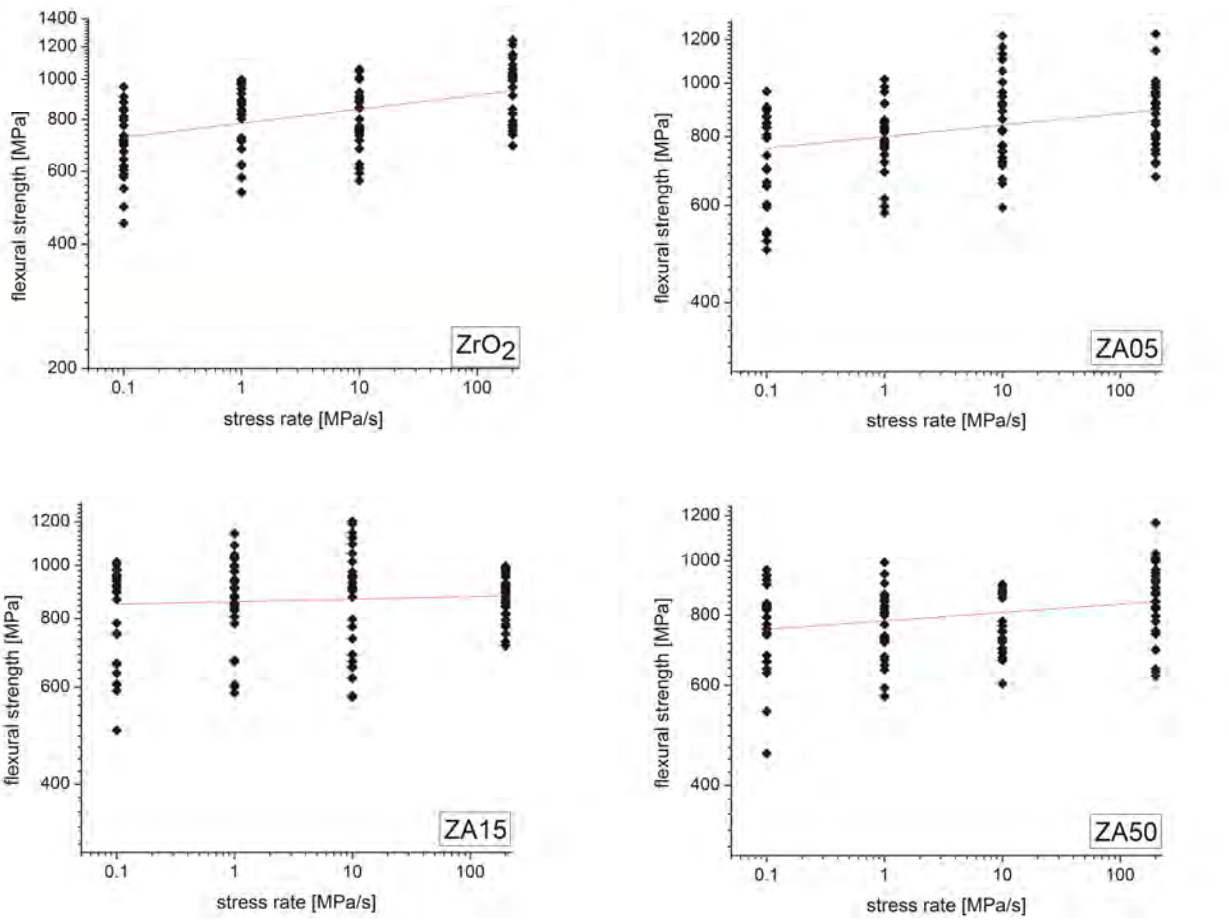

Fig. 3. Double logarithmic flexural strength versus stress rate dependence graphs

Rys. 3. Podwójnie logarytmiczne wykresy zależności wytrzymałości na zginanie od szybkości przyrostu naprężeń

TABLE 2. **Values of stress corrosion susceptibility parameter**
TABELA 2. **Wartości współczynnika podatności na korozję naprężeniową**

Material	*n* [-]
ZrO_2	28.31 (23.88÷34.66)
ZA05	46.39 (36.76÷62.61)
ZA15	84.47 (56.90÷162.13)
ZA50	61.50 (48.33÷106.18)

The slope of the *velocity* vs. K_I/K_{Ic} graph depends on the *n* parameter. By knowing the velocity (calculated as a change in crack length in time) and the corresponding stress intensity factor (Table 3), it is possible to create a part of the *v* vs. K_I/K_{Ic} dependencies (Fig. 4) [10].

TABLE 3. **Crack length velocities at presented stress intensity factors**
TABELA 3. **Szybkości rozprzestrzeniania się pęknięć dla prezentowanych współczynników intensywności naprężeń**

Material	*v* [m/s]	K_I [MPa·m$^{1/2}$]
ZrO_2	$2.23 \cdot 10^{-9}$	4.40
ZA05	$1.65 \cdot 10^{-9}$	4.96
ZA15	$1.08 \cdot 10^{-9}$	5.76
ZA50	$5.71 \cdot 10^{-10}$	5.48

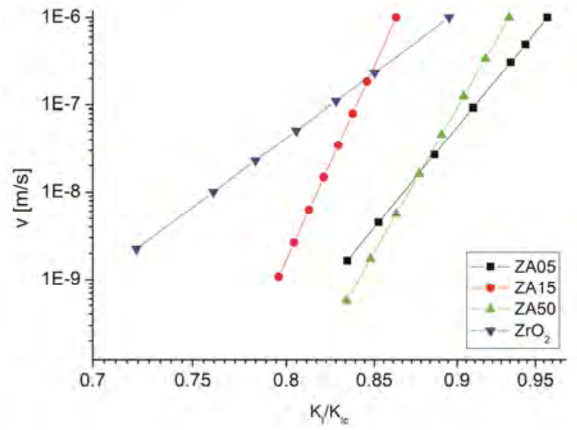

Fig. 4. Crack length velocity at stress intensity factors lower than K_{Ic}

Rys. 4. Szybkości rozprzestrzeniania się pęknięć dla współczynników intensywności naprężeń niższych od K_{Ic}

The *n* parameter also heavily influences determining the time to failure. Despite the fact that the collected results relate to the dynamic conditions, conversion to static ones is possible [13]. It is an easier way to present the lifetime of a material and a case which corresponds to most real applications. Figure 5 shows the strength-probability of survival-time diagrams. The chosen times of service were 1 second, 1 day, 1 month and 1 year. For pure zirconia the difference between the strength for 1 second of service (where the subcritical crack growth does not play a big role) and 1 day and longer times is the most significant. For the composites this difference is smaller (the best case is for the ZA15 composite).

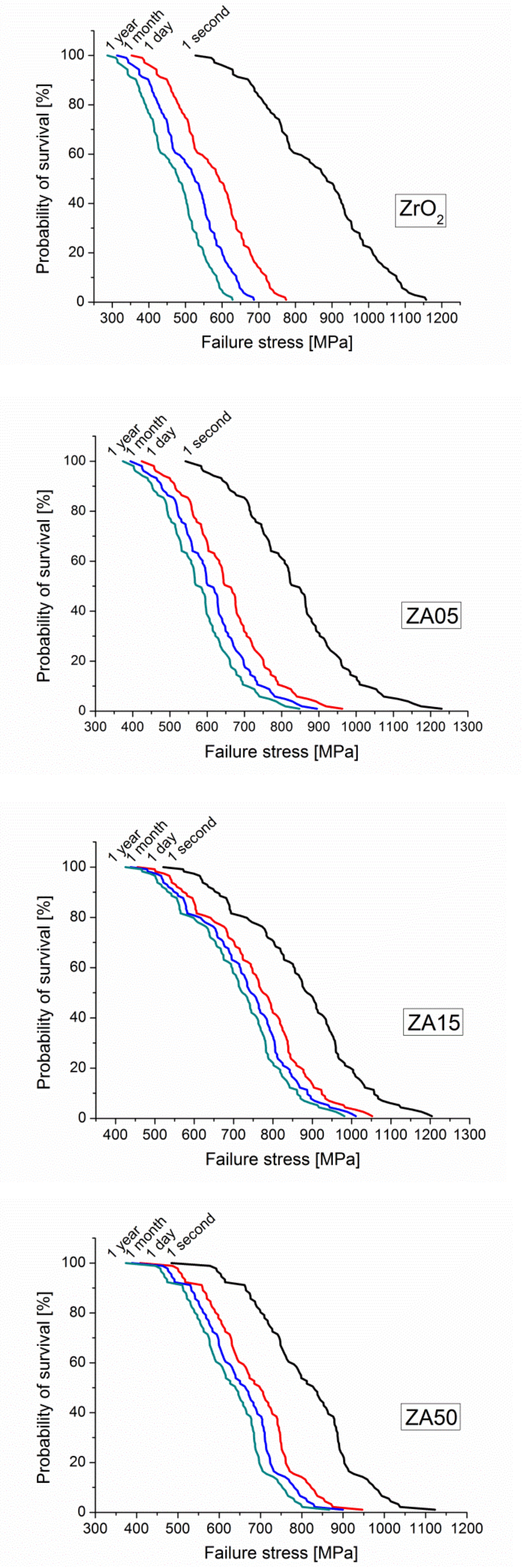

Fig. 5. Strength-probability-time diagrams

Rys. 5. Wykres zależności wytrzymałość-prawdopodobieństwo-czas

CONCLUSIONS

The presented work shows that the Constant Stress Rate test is a useful method for subcritical crack growth detection and lifetime predictions. It seems that determining the whole v vs. K_I plot with known areas [4, 8] is not obligatory and a simplified form is sufficient. Most cases of material service are rather under static conditions. The results presented in our work were achieved under dynamic ones, but this inconvenience could be solved by adopting a few simple calculations [10].

For every tested material, subcritical crack propagation was observed as an increasing resistance to increasing stress rates. On the basis of the obtained results, the n parameter - showing the stress corrosion resistance, can be easily calculated. A higher n value means that material shows a lower tendency for subcritical crack propagation. The addition of alumina inclusions in zirconia matrices decreases this tendency in every case. The most effective improvement is observed for the 15 vol.% alumina additions. In this composite the n value is the highest and there is a smaller decrease in strength between the 1st second of service and longer times.

Acknowledgements

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