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A NEW TYPE OF ALUMINA TOUGHENED ZIRCONIA – MODERN MATERIAL FOR STRUCTURAL APPLICATIONS IN MACHINERY INDUSTRY

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Paper presents selected properties of a kind of alumina toughened zirconia (ATZ) composites manufactured by the method which utilize reactive sintering process. Products made by means of this process show many perspective properties which could improve their usefulness as machinery parts materials. These properties comes from much finer microstructure than this obtained in typical AZT or TZP (tetragonal zirconia) materials.

Special advantages mentioned materials showed in tribological applications connected with sliding cooperation without any lubrication at temperatures exceeded 300 °C like rolling elements of machinery or parts subjected to intensive abrasion. Another potential field of application is area when working elements are subjected to stable load under critical value. Actually, in ever known oxide materials at such conditions slow crack propagation phenomenon occurs. In a new type of ATZ materials this phenomenon could be distinctly limited or even stopped. In the paper we present a results of investigations which confirm high level of useful properties detected in a new type of ATZ composites.

Keywords: alumina toughened zirconia, slow crack propagation, sliding wear

НОВЫЙ ТИП ЦИРКОНИЕВОЙ КЕРАМИКИ, УПРОЧНЕННОЙ ОКСИДОМ АЛЮМИНИЯ – СОВРЕМЕННЫЙ МАТЕРИАЛ ДЛЯ КОНСТРУКЦИОННЫХ ПРИМЕНЕНИЙ В МАШИНОСТРОИТЕЛЬНОЙ ПРОМЫШЛЕННОСТИ

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Статья представляет избранные свойства композитов из оксида циркония, упрочненного оксидом алюминия (ATZ), изготовленных методом, в котором используется процесс реактивного спекания. Изделия, изготовленные с помощью этого процесса, обладают многими перспективными свойствами, которые могут повысить их полезность в качестве материалов для деталей машин. Эти свойства обусловлены гораздо более тонкой микроструктурой, чем у типичных материалов AZT или TZP (тетрагональный диоксид циркония).

Особые преимущества указанные материалы проявили в трибологических применениях, связанных с взаимодействием скольжения без смазки при температурах выше 300 °C, например, в качестве тел качения машин или деталей, подвергающихся интенсивному истиранию. Другой потенциальной областью применения является область, где рабочие элементы подвергаются устойчивой нагрузке ниже критического значения. Действительно, в известных оксидных материалах при таких условиях имеет место явление медленного распространения трещины. В новом типе материалов ATZ это явление может быть четко ограничено или даже остановлено. В статье представлены результаты исследований, подтверждающие высокий уровень полезных свойств, обнаруженных в композитах ATZ нового типа.

Ключевые слова: цирконий, упрочненный оксидом алюминия, медленное распространение трещин, износ скольжения

ALYUMINIY OKSIDI BILAN MUSTAXKAMLASHTIRILGAN YANGI TURDAGI SIRKONIY OKSIDLI KERAMIKA – MASHINASOZLIK SANOATI UCHUN ZAMONAVIY KONSTRUKSION MATERIAL

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Maqolada reaktiv pishirish jarayonidan foydalanib olingan alyuminiy oksidi bilan mustaxkamlangan, sirkon oksididan olingan kompozitlarning tanlangan xossalari taqdim etilgan. Bu jarayon yordamida olingan buyumlar, ko'pgina o'ziga xos xossalari bilan mashina detallarini tayyorlashda qo'llanishi mumkin. Bu xossalalar boshqa materiallarga AZT yoki TZP larga o'ta mayda mikrostrukturaga egaligi bilan asoslanadi.

Bu materiallar 300°C dan yuqori haroratda moylanmagan holatda o'zaro sirpanish ta'sirida bo'lgan tribologik sharoitlarda, masalan, intensiv ishqalanish bilan boradigan tebranish mashinalari yoki detallari sifatida ishlatilganda namoyon bo'ladi. Boshqa potensial ishlatish sohasi kritik qiymatdan past yuklanishda ishlaydigan ishchi elementlar hisoblanadi. Haqiqatan ma'lum bo'lgan oksidli materiallar bunday sharoitlarda yoriqlarning sekin tarqalishi kuzatiladi. Yangi tipdagi ATZ materiallarda esa bu jarayon chegaralangan, xattoki to'xtatilgan bo'ladi. Maqolada ATZ kompozitlarida kuzatiladigan foydali xossalarni yuqoriligini aks ettiruvchi natijalar keltirilgan.

Kalit so'zlar: sirkoniy, alyuminiy oksidi bilan mustaxkamlangan, yoriqlarning sekin tarqalishi, sirpanish edirilishi

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Introduction

Permanent development of structural ceramics products is a necessary condition for further improvement of almost all branches of manufacturing industry [1-4]. Properties of ceramics parts could be spectacular but each type of ceramic product demand a specific technology and has an optimal field of application. Alumina-toughened zirconia (ATZ) materials are relatively well recognized and commercialized due to their low manufacturing costs and good properties, which, in some applications, are much better than the properties of monophase tetragonal zirconia or alumina products. A very good example are knee or hip-joint ceramics endoprosthesis [5-7], but the mentioned ATZ materials have a significantly wider field of application in the machinery industry. Mentioned composites are very often used as an efficient material for parts of machinery subjected to sliding, rolling, or any other movement usually correlated with mechanical loading and the potential abrasive acting of the environmental elements. The applications of ATZ materials are not only limited to room temperature, as they can withstand elevated temperatures (a few hundred Celsius degree). In many previous studies the different aspects of ATZ composite processing, microstructures, and correlations with their final properties were elaborated [8-20]. Usually, attention has been focused on the zirconia/alumina ratio, phase composition, and sintering conditions (or methods). The important issue is also residual stress state caused by coefficients of thermal expansion mismatch of both alumina and zirconia phases. In alumina/zirconia materials, the zirconia phase is always under tension and alumina under compression [21]. Values of these stresses depends on individual phase content and grains size and shape [22]. They also could be introduced to the composite system by additional processes, e.g., ion exchange [23]. However, composite powder processing and, consequently, sintering procedure could also significantly influence the final phase composition, microstructure and residual stress state [24-28]. The aim of the presented paper was to modify a zirconia matrix in the ATZ composite as a specific material with a fine microstructure and high tendency to the tetragonal to monoclinic phase transformation, which could assure a high level of mechanical and tribological properties.

Research methods

An alumina-toughened zirconia (ATZ) ma-

terial, fabricated using a procedure consisting of the common sintering of two different zirconia powders [29, 30]. The general idea was to prepare composite mixing two zirconia powders: pure nanometric ZrO_2 one and a solid solution of 4 mol.% Y_2O_3 in ZrO_2 powder. The raw materials used for the preparation of the powders were zirconyl chloride, yttrium chloride, and ammonia (all delivered by Polskie Odczynniki Chemiczne S.A., Poland), which were also used for the precipitation process. Both zirconia powders (the pure one and the 4 mol.% of yttria solid solution) were obtained separately and homogenized by milling in a rotary-vibratory mill for 2 h in a propyl alcohol environment. The weight ratios of both powders were established on a level which assured 3 mol.% nominal content of yttrium oxide in the fabricated material. The final composition of the material was supplemented with the addition of 2.3 vol.% (1.5 wt.%) of nanometric alumina powder (TM-DAR, Taimicron, Taimei Chemicals Co. Ltd., Japan). The mixing process was performed in a rotary-vibratory mill for 30 min in a propyl alcohol environment. The final material was a composite with a zirconia matrix, with a small addition of nanometric alumina grains. The material was prepared as described and is herein designated as BC. The BC material was designed as composite with a small amount of isolated alumina particles dispersed in the zirconia matrix. The second investigated material, designated as BC10A, was a mixture of BC and 10 vol. % of alumina (TM-DAR, Taimicron). The level of alumina additives was planned to be about an alumina particles percolation threshold to assure different type of microstructure. The third prepared material, designated as BC20A, was a mixture of BC and 20 vol. % of alumina (TM-DAR, Taimicron). In this material alumina content was significantly above the percolation point. The BC20A material was designed as a duplex microstructure material containing two continuous phases.

One shape of samples were plates of $60 \times 60 \times 6$ mm and the second one were discs of 16 mm in diameter and ~ 1.5 mm in height. Samples were first uniaxially pressed (50 MPa) and then isostatically re-pressed at 200 MPa. Then, the samples were sintered at $1450^\circ C$ for 2 h.

After sintering densities of samples were determined by Archimedian method at $21^\circ C$ and related to their theoretical values (assuming that $dAl_2O_3 = 3.99 \text{ g/cm}^3$ and $dZrO_2 = 6.10 \text{ g/cm}^3$). Us-

ing the rule of mixtures assuming predicted zirconia and alumina content in the composite BC, BC10A and BC20A theoretical densities were calculated as $d_{BC} = 6.01 \text{ g/cm}^3$, $d_{BC20A} = 5.81 \text{ g/cm}^3$ and $d_{BC10A} = 5.61 \text{ g/cm}^3$.

Sintered samples was tested using the ball-on-disc method in a temperature range between 20 and 400 °C. The friction coefficient (CoF) and wear rate values were obtained based on the proper standard [31] using a Tribotester T-21, manufactured in the Institute for Sustainable Technologies (Radom, Poland). Wear rates for flat samples and counterparts (balls) were designated as WV and WVB, respectively. Normal load F was established on 10 N, the sliding speed was 120 rpm, and number of cycles was 30,000. The applied temperatures ranged from 20 (RT) to 500 °C. The radius of the wear trace was 5 mm. Zirconia balls (5 mm in diameter) were used as the counterparts. In this role, we used a commercially available grinding media manufactured by Tosoh Comp. usually used in attritor-type mills.

After the sliding wear tests, the worn surfaces were examined with an interferometric profilometer ProFilm3D (Milpitas, USA) to estimate the wear rates for the samples and counterparts (WV, WVB) according to the procedure described by the authors of [32].

Images of the materials' microstructures were obtained with a scanning microscope Apreo 2 (Thermo Fischer Scientific) on polished samples.

Slow crack propagation rate was determined with procedure described in details in [33, 34] utilizing Constant Stress Rate Test applied under biaxial bending mode. Such procedure could detect occurrence and intensity of phenomenon of strength decrease under long-lasting load which is typical for oxide materials.

Results and discassen

Densities of sintered samples measured by Archimedian method showed a similar densification level for both tested materials. Measures density of BC samples was $5.97 \pm 0.01 \text{ g/cm}^3$ what mean $99.3 \pm 0.02\%$ of theoretical density. Densities of BC10A samples were $5.78 \pm 0.03 \text{ g/cm}^3$ and 99.5 ± 0.05 , respectively. Densities of BC20A samples were $5.58 \pm 0.05 \text{ g/cm}^3$ and 99.4 ± 0.09 , respectively. These data confirm very good level of densification what is very profitable for tribo-

logical properties and generally for mechanical ones.

Scanning electron microscopic (SEM) images in Figure 1 illustrate a typical microstructures of BC (top) BC10A (middle) and BC20A (bottom) materials. Lighter grains compose zirconia matrix one and darker once are alumina as minority phase. Micrographs confirm very fine and uniform microstructures with inclusions grains smaller than 500 nm.

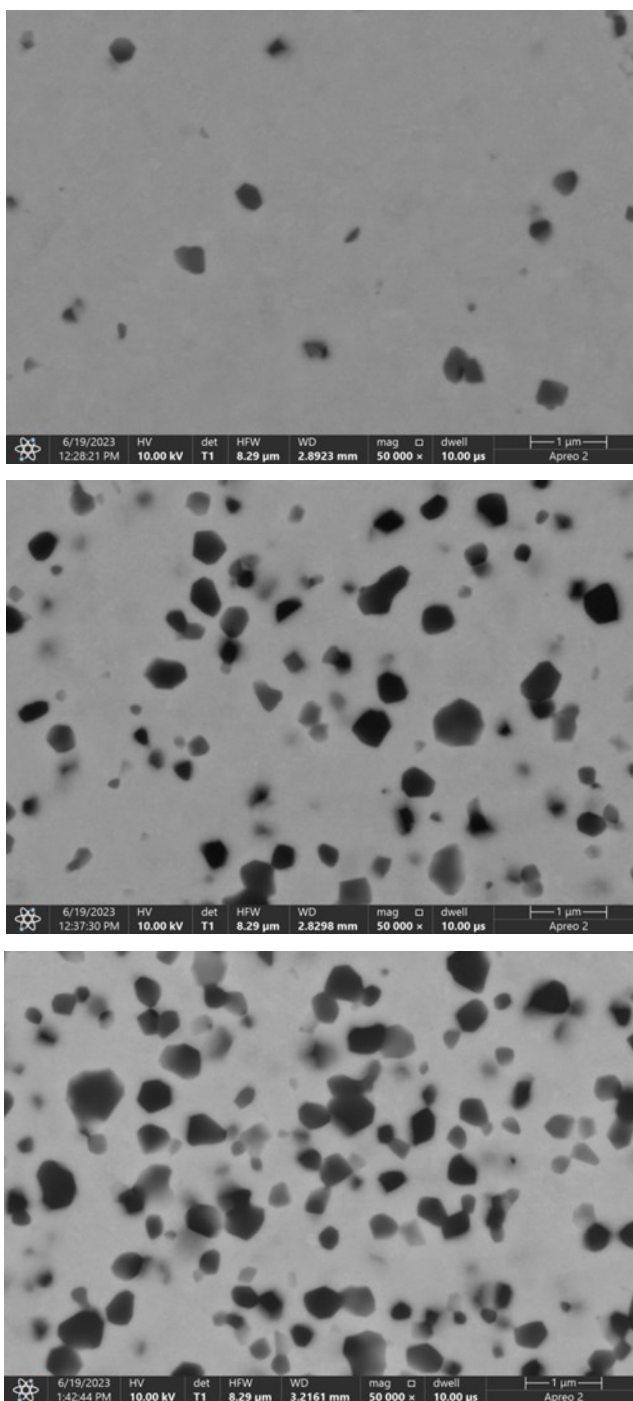


Figure 1. Microstructures of polished surfaces of investigated composites (top – BC; middle – BC10A; bottom – BC20A).

Figure 2 shows the aggregated wear test results for the investigated samples. It can be observed that temperature at which sliding wear is minimal is different for BC material when compared with BC10A and BC20A ones. For BC material it is 300 °C and for BC10A and BC20A it is 400 °C. This means that the most favorable conditions for cooperation with a zirconia counter-sample are slightly different for a material with a minimum content of inclusions (lower temperature) and different for a material enriched with alumina inclusions (higher temperature). Figure 3 shows the collected results of coefficients of friction (CoF) measurements. It is easy to state from this plot that CoF's for all types of materials decrease with temperature of test increase. Actually, for all investigated materials these relation and CoF values are similar.

Investigations of subcritical cracking susceptibility performed using Constant Stress Rate Test applied to biaxial bending procedure, proved that these materials are fully resistant for slow crack propagation phenomenon, Figure 4 presents comparison of constant stress rate for typical 3Y-TZP material and BC20A composite. Very high value of n parameter (>300) present in the Figure confirms that in BC20A material phenomenon of subcritical crack propagation was definitely stopped. Usually it is believed that values higher than 100 are typical for material non fragile for slow crack propagation.

Conclusion

Generally, results of conducted experiments confirm that the ATZ composite materials manufactured by the proposed technique had a strong potential to be used for reliable machinery parts working in the sliding regime at elevated temperatures or working under subcritical stresses

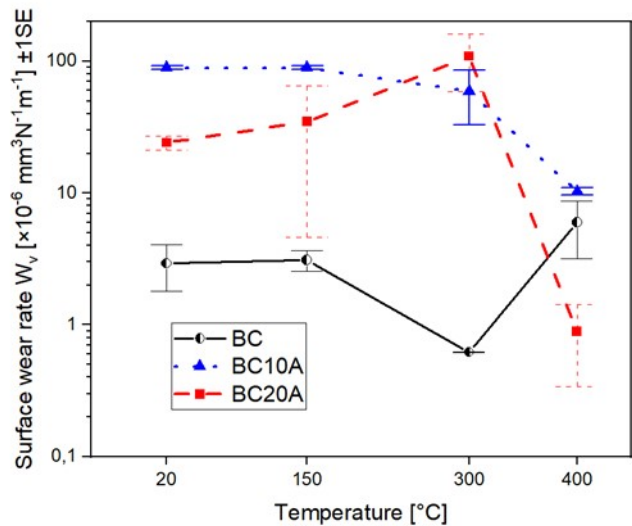


Figure 2. Sliding wear rates W_v at different temperatures for all investigated materials.

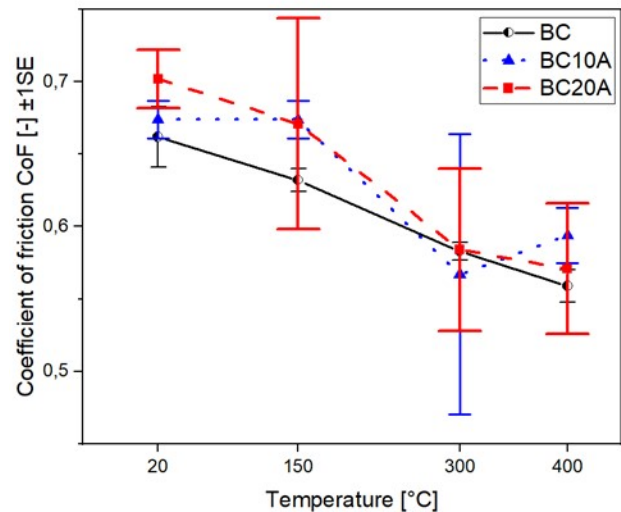


Figure 3. Coefficients of friction (CoF) vs. temperatures of tribological pairs defined by investigated ATZ materials and zirconia ball.

during long term loading.

Tribological pairs composed of ATZ composites and tetragonal zirconia counterpart behave in similar way independently of ATZ composite phase arrangement. Typical particulate

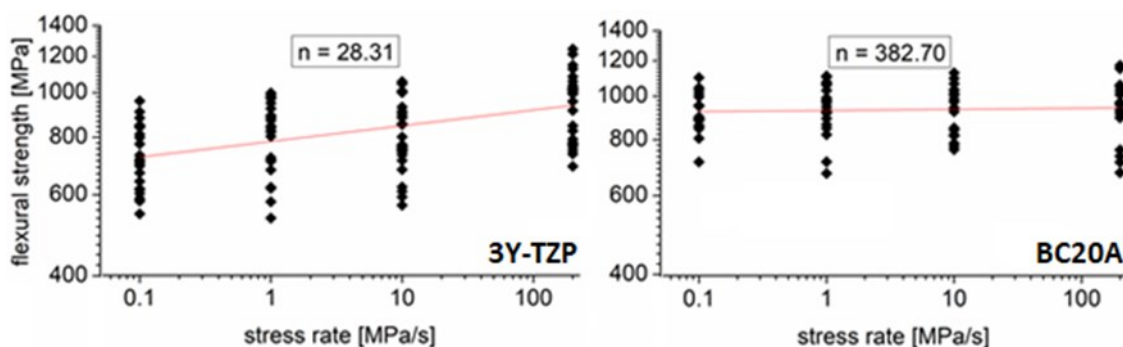


Figure 4. Results of constant stress rate in biaxial bending test confirming the occurrence of the subcritical crack propagation phenomenon in commercial 3Y-TZP ceramics (left according to [35]) and lack of this phenomenon in BC20A composite (right).

composite with isolated alumina particles or typical duplex microstructure composed of two interpenetrating continuous phases show wear rate and coefficient of friction significantly decrease with temperature increase. Observed differences concern a temperature value at which minimal wear rate is detected. In a typical particulate composite (BC) temperature of forming of stable layer which supporting sliding co-operation is lower than in material with higher alumina content (BC10A, BC20A).

Proper microstructure of composite and introduction of higher amounts of alumina fine grained phase could affect subcritical crack propagation even stop this phenomenon what is very rare in the case of fully oxide ceramics.

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