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Procedia Engineering 100 (2015) 1133 - 1140



www.elsevier.com/locate/procedia

25th DAAAM International Symposium on Intelligent Manufacturing and Automation, DAAAM 2014

Erosion Resistance of Slip Cast Composite Al₂O₃-ZrO₂ Ceramics

Marijana Majić Renjo*, Lidija Ćurković, Krešimir Grilec

Faculty of Mechanical Engineering and Naval Architecture, Ivana Lučića 5, 10000 Zagreb, Croatia

Abstract

In this paper, erosion resistance of slip cast monolithic (Al₂O₃) and composite (Al₂O₃-t-ZrO₂) ceramics was investigated by dry silica sand (SiO₂) erodent. The amount of t-ZrO₂ in powder mixture of Al₂O₃-t-ZrO₂ was 5 wt. % and 10 wt. %. The erosive wear behaviour was studied at following impact angles of erodent: 30° , 60° and 90° . Erosion mechanisms of all prepared ceramic samples were evaluated by measuring the roughness parameters (R_a , R_z , R_{max}) and weight loss, as well as analysis of surface morphology by means of scanning electron microscope (SEM).

Obtained results showed that tribological properties of monolithic Al_2O_3 can be improved with the addition of t-ZrO₂. It was also found that erosion resistance increases with the increasing amount of t-ZrO₂ in the powder mixture. Generally, for all investigated ceramic samples erosion resistance decreases by the increasing erodent impact angle.

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Keywords: alumina (Al2O3); zirconia (ZrO2); Al2O3-ZrO2 composite ceramics; erosion resistance; slip casting

1. Introduction

Advances in materials science drive economic and social development improving the everyday life quality [1]. Due to its advantages, such as high strength and hardness, temperature stability, high melting point, high wear and corrosion resistance, monolithic alumina (Al_2O_3) is a relatively widely used oxide ceramics, suitable for various applications. However, its low fracture toughness makes a great disadvantage. In order to improve the fracture toughness, yttria-stabilized tetragonal zirconia (t-ZrO₂) can be added to monolithic alumina, resulting in composite

^{*} Corresponding author. Tel.: +385 1 6168 304; fax: +385 1 6157 126. *E-mail address:* marijana.majic@fsb.hr

ceramics, tougher and stronger than alumina [2, 3].

Nomenclature

wt.	mass portion, %
η	dynamic viscosity, mPas

Different technologies, such as pressing, casting or spraying can be used to produce composite ceramics. Slip casting is the most adequate technology for production of complex ceramic components of different sizes and shapes. It can be used for both, monolithic and composite ceramics production. It is a simple, reliable, flexible, cost-effective and pollution-free procedure, but it requires adequate understanding of colloid suspensions and their behaviour. In order to use all the benefits of slip casting technology, suspension viscosity, stability, density and composition must be optimal, as well as the casting parameters [4-9]. Various additives are investigated, in order to keep the required suspension density, but lower the viscosity, which causes difficulties in moulding [4, 5, 10]. If a prepared suspension is stable, the slip casting process results in homogenous green and sintered bodies, with required density and adequate properties.

Wear is one of the most commonly encountered industrial problems, leading to frequent replacement of components [11]. Solid particle erosion can be defined as the degradation of material that results from repeated impacts of small solid particles. It occurs in a gaseous or a liquid medium containing solid particles. The present medium can change the velocity and direction of erodents (solid particles) [12]. If the erosion occurs at impact angles between 0° and 30°, it is regarded as abrasive erosion, while the one occurring between 60° and 90° is called impact erosion [13]. Depending on the material and operating parameters, erosive wear can occur with plastic deformation and/or brittle fracture. Ductile materials will undergo wear by a plastic deformation process, where the material is removed by the displacing or cutting action of the eroding particle. On the other hand, eroded brittle material will be removed by the formation and intersection of cracks that cause grain ejection [14].

In this investigation, erosive wear behaviour of slip cast Al_2O_3 and Al_2O_3 -t-ZrO₂ ceramics was studied at the erodent impact angles of 30°, 60° and 90°. Dry silica sand (SiO₂) was used as an erodent. Erosion mechanisms of all prepared ceramic samples were evaluated by measuring the roughness parameters (R_a , R_z , R_{max}) and weight loss, as well as analysis of surface morphology by means of scanning electron microscope (SEM).

2. Materials and methods

2.1. Preparation of monolithic and composite ceramics

Prepared monolithic and composite ceramic samples were formed by slip casting process in plaster mould. A slip is a suspension of fine powder in a liquid with small amounts of additives – dispersants, binders, plasticizers or sintering agents. In this investigation, Al₂O₃ and t-ZrO₂ powders were dispersed in distilled water, with the addition of commercially available dispersant DOLAPIX CE64 (Zschimmer & Schwarz GmbH, Germany). Alumina powder had average particle size of 600-900 nm, according to the manufacturer specification (Alcan Chemicals, USA), while the zirconia powder had particles around 300 nm (SkySpring Nanomaterials, Inc., USA).

Three groups of suspensions were prepared: monolithic Al_2O_3 and composite Al_2O_3 -t-ZrO₂ ceramics (composition: 95 wt. % $Al_2O_3 - 5$ wt. % t-ZrO₂ and 90 wt. % $Al_2O_3 - 10$ wt. % t-ZrO₂). Dry powder content was 70 wt. % in all prepared suspensions. During the preliminary experiments, the amount of DOLAPIX CE 64 was varied within each group, in order to determine its optimal content, which is reflected in obtained minimal viscosity. All 70 wt. % aqueous suspensions were homogenized for 2 hours, at 300 rpm in the planetary ball mill (Retsch PM100, Germany). The grinding jar and ten balls used for homogenization were made of alumina. In order to remove air bubbles and to achieve better homogeneity of prepared suspensions, each of them was treated in the ultrasonic bath. Rheological measurements were conducted on the rotational viscometer Brookfield DV-III Ultra, USA. The chemical composition of prepared suspensions with their measured optimal viscosity is given in Table 1.

<i>wt</i> . (Al ₂ O ₃ + t-ZrO ₂), %	wt. (H ₂ O), %	<i>wt</i> . (Al ₂ O ₃ , in powder mixture), %	<i>wt.</i> (t-ZrO ₂ , in powder mixture), %	<i>wt.</i> * (DOLAPIX CE64), %	η, mPas
70	30	100	0	0.25	6.47
70	30	95	5	0.4	8.54
70	30	90	10	0.5	10.62

Table 1. Chemical composition of 70 wt. % aqueous suspensions for slip casting.

*wt., weight percent based on the applied ceramic dry powder

Slip casting involves preparation of stable highly concentrated water-based slip (suspension), which is poured into microporous plaster of Paris mould. Water is absorbed by the microporous mould through capillary action to produce green ceramic compacts. It results in forming a dense cast form, lacking deleterious air gaps and minimizing shrinkage in the final sintering process [14].

After removing from plaster moulds, green bodies were dried at 100 °C for an hour and then sintered at a temperature of 1650 °C in an electric kiln (Nabertherm P310, Germany).

2.2. Analysis of morphology of sintered composite ceramics by means of SEM-EDS

Surface morphology of sintered composite ceramics samples was determined by SEM – scanning electron microscope (Tescan Vega TS5136LS, Czech Republic), equipped with energy dispersive spectrometer (EDS). Distribution of the aluminium (Al), zirconium (Zr) and oxygen (O) on fracture surface of sintered samples was determined by EDS mapping.

2.3. Analysis of erosion mechanisms of sintered monolithic and composite ceramics

Sintered samples of Al_2O_3 and Al_2O_3 -t-ZrO₂ (with addition of 5 and 10 wt. % t-ZrO₂) ceramics were prepared for erosion test by grinding and polishing. Erosive wear resistance test was conducted on the apparatus, schematically shown in Fig. 1. When using this apparatus, samples are placed in supporters and span at about 1440 rpm through the seeping beam of erodent powder. Two samples can be eroded at the same time. The erodent powder falls down due to the gravitational force.



Fig. 1. Schematical representation of erosive wear resistance testing machine.

The parameters of applied erosive wear resistance test were as follows:

- Sample dimension: 17×17×5 mm
- Erodents: dry silica sand (SiO₂)
- Revolution of sample holder: 1440 rpm
- Sample velocity: 24.3 m/s
- Testing time: 13 min 53 sec (~20 000 impacts)

• Impact angle: 30°, 60°, 90°

Erosion wear mechanisms were monitoring by:

- Measuring of weight loss (samples were weighed on a laboratory balance before and after erosive wear resistance test)
- Measuring of surface roughness parameters (R_a , R_{max} , R_z) before and after erosion at the impact angle of 30°, 60° and 90° by Perthometer S8P (PerthenMahr, Germany)Testing time: 13 min 53 sec (~ 20 000 impacts)
- SEM analysis of surface morphology before and after erosion at 90°

3. Results and discussion

3.1. Surface morphology analysis

The results of SEM-EDS analysis of prepared composite Al_2O_3 -t-ZrO₂ ceramics are shown in Figs. 2 and 3. It can be seen that the t-ZrO₂ particles (the brighter phase) are well dispersed in Al_2O_3 matrix (the darker phase), for both samples (containing 5 wt. % and 10 wt. % of t-ZrO₂). SEM-EDS mapping on the fracture surface of sintered samples showed homogeneous distribution of aluminium (Al), oxygen (O) and zirconium (Zr) in both slip cast composite ceramics.



Fig. 2. SEM images of fracture surface (back-scattered electrons mode) of Al₂O₃-t-ZrO₂ composite ceramics with 5 wt. % of t-ZrO₂; X-ray mapping micrographs of different elements: Al, O and Zr.



Fig. 3. SEM images of fracture surface (back-scattered electrons mode) of Al₂O₃-t-ZrO₂ composite ceramics with 10 wt. % of t-ZrO₂; X-ray mapping micrographs of different elements: Al, O and Zr.

3.2. Erosion mechanisms of monolithic and composite ceramics

All prepared samples (monolithic Al_2O_3 and composite Al_2O_3 -t-ZrO₂, containing 5 and 10 wt. % of t-ZrO₂) were eroded with SiO₂ erodent with different impact angles (30°, 60° and 90°). Each test was conducted twice on each sample. Erosion mechanisms of prepared ceramic samples were evaluated by measuring the weight loss, surface

roughness parameters (R_a , R_z , R_{max}) before and after erosion test at 90° impact angle, as well as analysis of surface morphology by means of scanning electron microscope (SEM).

Average weight loss of each sample, under each testing condition is given in Fig. 4. It can be observed that the weight loss increases with the increase of the impact angle. Other researchers obtained a similar trend of increasing erosion rate with increasing impact angle for brittle materials [15-17]. It can be seen on Fig. 4 that monolithic Al_2O_3 and composite Al_2O_3 -t-ZrO₂ ceramics (composition: 95 wt. % Al_2O_3 – 5 wt. % t-ZrO₂ and 90 wt. % Al_2O_3 – 10 wt. % t-ZrO₂) were more sensitive to the impact erosion (higher impact angles) than to the abrasive erosion (lower impact angles). It can be concluded that monolithic alumina is generally less resistant to the erosive wear than alumina-zirconia composite ceramics. Erosion wear resistance increases with the increase of zirconia.



Fig. 4. Weight loss (in mg) for prepared ceramic samples after erosion at different impact angles.

The investigated samples (monolithic Al₂O₃ and composite Al₂O₃–t-ZrO₂, containing 5 and 10 wt. % of t-ZrO₂) were subjected to the surface roughness measurements before and after erosion with SiO₂ erodent at different impact angles (30°, 60° and 90°). All results of surface roughness parameters (R_a , R_z , R_{max}) measurements are given in Fig. 5. The result analysis didn't show any particular regularity. Generally, all surface roughness parameters (before and after erosion at different impact angles) were higher for monolithic alumina than for alumina-zirconia composite ceramics. In general, surface roughness parameters increased after erosion.

Surface topography plays an important role in surface analysis after solid particle erosion [18]. SEM images of prepared samples before and after erosion with SiO_2 particles at the impact angle of 90° are presented in Figs. 6 and 7. It can be noticed that monolithic alumina sample shows the roughest surface on both figures, which is consistent with measured surface roughness parameters. Also, the sample with highest amount of zirconia has the smoothest surface.





Fig. 5. Surface roughness parameters for all prepared samples before and after the erosion at all impact angles: (a) R_{a_2} (b) R_{z_2} (c) R_{max} .



Fig. 6. SEM images of prepared samples before erosion: (a) monolithic Al₂O₃; (b) composite Al₂O₃–t-ZrO₂ containing 5 wt.% of t-ZrO₂; (c) composite Al₂O₃–t-ZrO₂ containing 10 wt.% of t-ZrO₂.

When the impact angle is close to 90°, the dominant material removal mechanism was grain ejection and low, but existing, plastic deformation. Due to Al_2O_3 low fracture toughness, cracks propagate rapidly across the grain boundaries. The subsequent erodent impacts easily remove the surface material via the ejection of the upper layer grains. It can be observed that samples containing only Al_2O_3 were the most damaged ones.

The visible difference among the samples is observed in Fig. 7 (after erosion at 90°), where it is clearly seen that the SiO₂ erodent at this impact angle significantly damages the alumina surface, in which the craters of ejected grains are visible. With the addition of ZrO_2 in Al_2O_3 matrix, the wear scars are reduced, which indicates the grain ejection has been slowed down.



Fig. 7. SEM images of prepared samples after erosion at 30°: (a) monolithic Al₂O₃; (b) composite Al₂O₃-t-ZrO₂ containing 5 wt.% of t-ZrO₂; (c) composite Al₂O₃-t-ZrO₂ containing 10 wt.% of t-ZrO₂.

Conclusion

Green bodies of monolithic Al_2O_3 and composite Al_2O_3 -t-ZrO₂ ceramics (composition: 95 wt. % Al_2O_3 – 5 wt. % t-ZrO₂ and 90 wt. % Al_2O_3 – 10 wt. % t-ZrO₂) were formed by slip casting process in plaster mould. Optimal amount of dispersant DOLAPIX CE 64 was determined in preliminary investigation. After drying, green bodies were sintered at a temperature of 1650 °C. Sintered samples were subjected to the test of the erosion resistance to the SiO₂ under different impact angles (30°, 60°, 90°).

Weight loss was observed on all samples, but it has been two to three times lower for composite than for monolithic samples. It also showed that investigated samples were more sensitive to the impact erosion (at higher impact angles) than to the abrasive erosion (at lower impact angles). Weight loss measurements showed that erosion wear resistance increases with the increase of zirconia.

SEM-EDS analysis of prepared composite ceramics showed that t-ZrO₂ particles are homogenously dispersed in Al₂O₃ matrix, for both samples (containing 5 wt. % and 10 wt. % of t-ZrO₂).

The SEM images of tested samples before and after erosion show larger wear scars on the surface of monolithic Al_2O_3 than on the composite Al_2O_3 -t-ZrO₂ ceramics. The wear scars were in the form of ejected grains. The increase in the t-ZrO₂ amount resulted in reduced wear scars of composite ceramics, indicating reduced grain ejection.

Surface roughness parameters (R_a , R_z , R_{max}) increased after erosion for all investigated samples (monolithic Al₂O₃ and composite Al₂O₃–t-ZrO₂ ceramics). The largest increase was observed for monolithic Al₂O₃. Surface roughness parameter values generally decrease with the increase of t-ZrO₂ content.

All conducted tests lead to a conclusion that tribological properties of monolithic Al_2O_3 can be improved with the addition of t-ZrO₂. Erosion resistance of composite Al_2O_3 -t-ZrO₂ ceramics increases with the increase in the amount of t-ZrO₂.

The future erosion experiments will be performed on nanostructured alumina-zirconia ceramic composite.

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