Effect of basalt and silica additives on erosive wear resistance of cast ceramics

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Abstract. This paper focuses on the erosive wear behaviour of cast ceramics with added basalt (fibres, flakes, and powder) and silica (fine and coarse powder). The objective was to obtain inexpensive cast ceramic compositions with improved erosive wear resistance, which can be useful in the production of supporting and/or protective elements for applications in erosive conditions at room and elevated temperatures. Three commercially available cast ceramics (based on SiO\textsubscript{2}, Al\textsubscript{2}O\textsubscript{3}, and ZrO\textsubscript{2}) without reinforcement as well as reinforced by additives and composites based on liquid glass with additives, were produced and are compared in this work. The obtained samples were assessed for performance in erosive media (particle velocity 20 m s\textsuperscript{-1}; impact angle 30°; temperature 20, 300, and 600 °C) using a high temperature centrifugal erosion tester. Use of the reinforcing additives increased the wear resistance of the obtained commercial composites by up to 54%. Reinforced cast ceramic based on liquid glass exhibited an up to 73% lower wear rate as compared to a sample of commercially available ceramic at temperatures up to 600 °C.

Key words: cast ceramic, erosive wear, reinforcement, basalt, silica, alumina, zirconia, liquid glass.

1. INTRODUCTION

Ceramics are regarded as materials for applications in a wide range of temperatures due to their good thermal and mechanical properties [1]. Quite often it is necessary to obtain ceramic products of complex shape such as restorative materials [2], protective or supporting materials for ovens of irregular shape, and thermal barriers [3], electrical isolators [4], artificial bones [5], etc. Usually it is technologically difficult to apply high temperature and high pressure for the production of these materials. For such needs casting ceramic that cures (hardens) in a temperature range from 20 up to 150 °C and at standard pressure (0.1 MPa) can be applied.

One of the main failure modes of ceramics in industrial, automotive, and aviation use is solid particle erosion. In contrast to the ductile metals that have the highest wear rate at low angles of solid particle impingement (usually between 10° and 45°), for brittle ceramic products the maximum wear rate is mostly observed between 75° and 90° [6,7]. Moreover, the temperature of the test affects the wear behaviour of ceramic materials [8,9]. To improve the mechanical properties and performance of ceramic materials reinforcing additives, such as fibres, second-phase particles, and flakes, have often been used [10,11]. Due to its ceramic nature and low price, basalt can serve as a reinforcing agent in various types of matrices [12].

This paper focuses on the dependence of the erosive wear behaviour of cast ceramics on different reinforcement additives and their concentrations in a wide range of temperatures (from room temperature up to 600 °C). The objective was to obtain cost-effective cast ceramic compositions with high erosive wear resistance necessary in supporting and/or protective elements for applications in erosive conditions at room and high temperatures.
2. MATERIALS AND METHODS

2.1. Description of the used commercially available materials

Three base materials supplied by the Cotronics Corporation (USA) from the Rescor 700 production line were compared: Rescor 750 (hereinafter R750), Rescor 780 (R780), and Rescor 760 (R760), based on SiO₂, Al₂O₃, and ZrO₂, respectively. To obtain a castable mass, liquid activators (Cotronics Corporation) were adapted for each base: Rescor 750 Activator (R750A), Rescor 780 Activator (R780A), and Rescor 760 Activator (R760A). The mass ratio between the selected bases and activators was 78/22 wt%. Since SiO₂ and basalt have similar thermal properties, basalt and silica flour were selected as additional reinforcements of commercial cast ceramic R750. As the selected materials are based on SiO₂, sufficient availability and lower total price can be provided at any region as compared with materials based on e.g. Al₂O₃ and ZrO₂, etc. Table 1 gives the details of the basalt powder and fibres (approximate content of SiO₂ 50 wt% and Al₂O₃ 10 wt%) as well as the activated basalt flakes (ABF) with a similar chemical composition (as shown in Table 2) and fine crystalline silica flour (see Table 1) used as reinforcements. Figure 1 shows the morphology of ABF.

Compositions of the materials based on Cotronics 750 are specified in Table 3. The composites were prepared according to the following procedure: (1) mixing of dry powders in a blender-type laboratory mixer; (2) adding an activator; (3) mixing in a plastic can; (4) filling of rubber mould with the obtained mixture; (5) densification on a vibrating table for 5 min; (6) drying in a mould at room temperature for 24 h; (7) demoulding of the specimens; (8) drying in an oven (Nabertherm L9/B equipped with a P330 controller) at 120 °C for 4 h and firing in a furnace at 950 °C for 3 h applying a continuously increasing temperature from 120 °C (5 °C min⁻¹); (9) cooling down to room temperature (5 °C min⁻¹). The relatively high processing temperature (950 °C) may affect the content of crystalline phases in all basalt materials by forming new centres of crystallization at temperatures over 600 °C [13].

Table 1. Brief description of the used reinforcements

| Material                  | Specification                                      |
|---------------------------|----------------------------------------------------|
| Basalt powder (BP)        | Particle size range 0–25 μm (produced from wastes by disintegration) |
| Basalt fibres (BF)        | Single filament diameter 13 μm and cut length of 3.2 mm (Kamenny Vek Ltd., Russian Federation) |
| Activated basalt flakes (ABF) | Crystalline phase fraction more than 40% (Otselot, Ukraine) |
| Crystalline silica flour (M4) | Average grain size 54 μm (Sibelco Group, Belgium) |

Table 2. Mineral composition and physical properties of activated basalt flakes [14]

| Component | wt%  | Properties                     |
|-----------|------|--------------------------------|
| SiO₂      | 50.6 | Average size of 0.25 mm × 3 mm flakes |
| Al₂O₃     | 14.0 | Thickness 1–4 μm                |
| FeO⁺      | 16.1 | Moisture content <0.5–0.6%   |
| Fe₂O₃     |      |                                |
| CaO       | 9.6  | Main crystalline phases Silicon, enstatite, |
| MgO       | 4.1  | esseneite, pigeonite,          |
| K₂O       | 3.2  | kyanite, pyroxene,            |
| TiO₂      | 1.6  | parawollastonite               |
| Density   |      | (true/apparent)                |
|           |      | ~2950 kg m⁻³                  |
|           |      | ~515 kg m⁻³                   |

Fig. 1. SEM image of activated basalt flakes.

2.2. Preparation of the liquid glass cast ceramic sample

According to available recommendations for acid-resistant composition production based on liquid glass and high temperature-resistive basalt powder [15], new recipes for castable ceramics were offered as specified in Table 4. Particles of coarse-sized sand (U.S. Silica Co, Ottawa, IL) [16] were used to reveal the effect of sand particle size on the wear behaviour of samples and compare results to a more expensive fine crystalline silica flour (M4). A liquid glass based on sodium metasilicate (Keemia Kaubandus, Estonia) with a hardening accelerator (sodium hexafluorosilicate, Alfa Aesar) [17] was used as the base. The proposed reactive mixture (Na₂SiO₃ + Na₂SiF₆) allows reduction in the time of hardening at room temperature. The reaction, expressed by Eq. (1), proceeds within 10–15 min and is irreversible [18].
where $n$ is the undetermined number of molecules of water, equal on the both sides of the equation.

The newly designed liquid glass cast ceramic composites were prepared according to the composition shown in Table 4 following the above-described procedure. Firing was executed at 90 °C for 4 h with cooling to room temperature at a rate of 5 °C min$^{-1}$.

### 2.3. Characterization

Microstructural examination was conducted by using a scanning electron microscope (SEM) HITACHI TM-1000. For erosive testing of samples, an elevated temperature particle accelerator (model CAK-HET) produced in Tallinn University of Technology was used (6 kg of sand abrasive particles per test; particle velocity 20 m s$^{-1}$ and impact angle 30°) [19]. To estimate the influence of temperature on the wear properties of the obtained composites, three temperature regimes (20, 300, and 600 °C) were selected. A sample with the dimensions of 23 mm × 23 mm × 8 mm was tested at least twice. The wear rate was calculated according to the method given in [20]. The following formula was used for the calculation of the loss of weight of the tested samples:

$$\Delta M = (m_1 - m_2)1000,$$

where $\Delta M$ – loss of weight during the erosion test, mg; $m_1$ – weight of the sample before the erosion test, g; $m_2$ – weight of the sample after the erosion test, g.

To calculate the volumetric wear Eq. (3) was applied:

$$V = \frac{\Delta M}{\rho},$$

where $V$ – volumetric wear, mm$^3$; $\rho$ – material density, mg/mm$^3$.

The relative uncertainty of erosive testing was less than ±10%.

### Table 3. Mix design of the samples based on Cotronics 750, wt%

| Material | Base | Activator | Reinforcement |
|----------|------|-----------|---------------|
| R750     | 78   | 22        | –             |
| R750 + 7 wt% BP | 69   | 24        | 7             |
| R750 + 19 wt% BP | 59   | 22        | 19            |
| R750 + 1 wt% BF | 75   | 24        | 1             |
| R750 + 5 wt% BF | 69   | 26        | 5             |
| R750 + 11 wt% BF | 58   | 31        | 11            |
| R750 + 11 wt% M4 | 67   | 22        | 11            |
| R750 + 28 wt% M4 | 50   | 22        | 28            |
| R750 + 38 wt% M4 | 40   | 22        | 38            |
| R750 + 2 wt% ABF | 76   | 22        | 2             |
| R750 + 6 wt% ABF | 69   | 25        | 6             |
| R750 + 12 wt% ABF | 59   | 29        | 12            |

BP – basalt powder, BF – basalt flakes, M4 – crystalline silica flour (Sibelco M4), ABF – activated basalt flakes; – none.

### Table 4. Composition of the samples based on liquid glass, wt%

| Material | Base | Activator | Reinforcement |
|----------|------|-----------|---------------|
| Liquid glass + BP | 22   | 2         | 65            |
| Liquid glass + ABF | 22   | 2         | –             |
| Liquid glass + M4 | 22   | 2         | 65            |
| Liquid glass + sand (coarse, Ottawa) | 22   | 2         | –             |

BP – basalt powder, ABF – activated basalt flakes, M4 – crystalline silica flour, – none.
3. RESULTS

3.1. Commercially available materials

The wear rates of commercially available ceramics are shown in Fig. 2. The zirconia-based cast ceramic (R760) demonstrated the highest wear rate at all temperature regimes. The cast ceramic based on SiO₂ (R750) exhibited a significantly lower (up to ~22 times) wear rate as compared to ZrO₂ based ceramic. The lowest wear rate at all temperature regimes was shown by the Al₂O₃ based material (R780). Due to the higher concentration of fine particles in the raw powders, visually coarse particles on the surfaces of R750 and R780 are surrounded by finer particles as compared to R760 (Fig. 3). Generally, the

![Graph showing wear rates of commercially available ceramics.](image)

**Fig. 2.** Wear rates of commercially available ceramics.

![SEM images of the surfaces of commercially available ceramics after the wear rate test at 600°C.](image)

**Fig. 3.** SEM images of the surfaces of commercially available ceramics after the wear rate test at 600°C: (a) R750 (based on SiO₂), (b) R780 (based on Al₂O₃), and (c) R760 (based on ZrO₂).
decrease in the wear rate at increasing the temperature (from 20 to 600 °C) can be related to the embedment of sand particles into the surface and reduction of brittleness. A deeper investigation of this effect is necessary.

3.2. Cotronics 750 cast ceramic composite samples

The addition of 7 wt% BP to SiO₂-based cast ceramic (R750) decreased the wear rate by 5% at 20 °C, 22% at 300 °C, and had no effect at 600 °C (Fig. 4). When the concentration of BP was raised to 19 wt%, the wear rate increased by 30% at 20 °C, 41% at 300 °C, and 29% at 600 °C as compared to the reference sample.

The addition of a small amount (1 wt%) of BF gave good results in the wear rate reduction: by 27% at 20 °C, 45% at 300 °C, and 32% at 600 °C as compared to the reference sample. Raising the concentration of BF to 5 and 11 wt% led to an up to 76-fold increase in the wear rate. A homogeneous distribution of fibres in the matrix of R750 + 5 wt% BF was reached with the selected procedure of sample preparation (Fig. 5).

Generally, the addition of fine crystalline silica flour (M4) to SiO₂-based cast ceramic resulted in an increased wear rate for all concentrations and at all testing temperature regimes, except for the sample with 11 wt% at 300 °C (exhibiting a 46% decrease in the wear rate). This could be explained with reduced possibility of brittle fracturing due to filled gaps, pores, and other structural defects by fine silica particles.

The addition of ABF resulted in a similar behaviour as in the case of BF addition. A lower wear rate was achieved with a small amount (2 wt%) of added ABF. Although no effect occurred at 20 °C, the wear rate decreased by 54% at 300 °C and by 34% at 600 °C. This could be explained by the presence of flakes in and above the pores and other structural defects as can be seen in Fig. 6. These flakes may reduce the possibility of brittle fracturing. A significant rise was observed in the wear rates due to a higher concentration of ABF (12 wt%). In case of a lower ABF concentration (6 wt%) the wear rate were similar to those with R750 at all temperatures.
3.3. Liquid glass-based ceramic composites

The lowest wear rate at 20°C was reached by using the liquid glass + BP composition, exhibiting a 73% decrease in the wear rate as compared to the reference sample (Fig. 7). However, at 600°C a significant increase in the relative wear rate – 27 times was observed for this material.

The composition of liquid glass + crystalline silica flour (M4) showed up to about 14 times higher wear rates compared to the reference sample. The composition of liquid glass + sand at 20°C exhibited better performance (by 29% lower wear rate) as compared to the reference sample, but an increase in the temperature led to the failure of the samples during the wear test. Optimal results were obtained with the composition of liquid glass + ABF, exhibiting up to 37% lower wear rates as compared to the reference samples at 20 and 600°C. However, the wear rate was significantly higher at 300°C (by 50%). The SEM images (Fig. 8) of the tested surfaces.
show pores due to the above-mentioned reaction (see (Eq. 1)). The presence of ABF was observed on the surface of liquid glass.

All samples based on liquid glass survived the wear test at all testing temperatures, except the liquid glass + sand composition, which failed during the 300°C test and it was impossible to measure the loss of weight properly.

The phase change in the liquid glass during heat treatment can have significant influence on the wear behaviour of a liquid glass ceramic. The formation of β-Na2Si2O5 at about 400°C and crystallization of SiO2 into the modification of cristobalite at about 600°C, which coexists with β-Na2Si2O5 [21], can make the matrix structure porous and more brittle than the initial amorphous phase. Relatively large ABF, bonded in the liquid glass structure, covered opened pores, creating a shield-like effect on erosive particles, while the grains of the powder-type materials (BP, M4, and sand) increased the brittleness of the structure and ensured a weaker bonding by the increased size of grains.

As there was no expected phase change of the basalt materials up to 600°C, in further research basalt firing at 950°C before adding it to liquid glass could be done. This can help to model a more comparable condition to the high temperature cast ceramics based on SiO2, Al2O3, and ZrO2.

4. CONCLUSIONS

Based on the results of the present study, the following conclusions can be made:

(1) Regarding commercially available cast ceramic materials without reinforcement:

- The wear rate decreased by increasing the temperature from 20 to 600°C at chosen conditions.
- The Al2O3-based ceramic (R780) exhibited the lowest wear rate at all temperatures, which is similar to the behaviour of the SiO2-based ceramic (R750).
- The ZrO2-based ceramic (R760) exhibited the highest wear rate as compared to the other studied commercial materials.
- The use of SiO2-based ceramics is beneficial from two points of view: (i) its structural similarity to SiO2-based reinforcements; (ii) SiO2 and basalt as reinforcements are less expensive than other alternatives.

(2) Regarding SiO2-based commercially available ceramic materials with the reinforcement additives (as compared to cast ceramic R750 without additives):

- An addition of BP up to 19 wt% led to an up to 41% decrease in the wear rate at all temperatures.
- A small amount (1 wt%) of BF decreased the wear rate by up to 45% (at 300°C).
- An addition of fine crystalline silica flour (M4) generally increased the wear rate at all concentrations, except 11 wt% at 300°C (exhibiting a wear rate decreased by 46%) due to the reduced possibility of brittle fracturing.
- A small amount (2 wt%) of ABF provided the best performance, an up to 54% lower wear rate at 300°C, but showed a significant increase in the wear rate when the concentration of ABF was increased up to 6 wt% or 12 wt%.

(3) Regarding the compositions with liquid glass as compared to SiO2 cast ceramic (R750):

- The best results at 20°C were achieved by using a composition of liquid glass + basalt powder, which exhibited a 73% lower wear rate as compared to R750.
- Compositions of liquid glass + ABF exhibited an up to 37% lower wear rate at 20°C and 600°C as compared to R750.
- A relatively large amount of ABF bonded in the liquid glass structure covered an extensive area of opened pores, creating a shield-like effect on erosive particles.

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Basaldi- ja ränidioksiidilisandite mõju valatud keraamiliste materjalide erosioonkulumisele

Janis Baronins, Maksim Antonov, Roman Ivanov, Volodymyr Shuliak ja Irina Hussainova

Artiklis on keskendutud valatud keraamilistele materjalidele, mis on valmistatud basaldi- (kiud, helbed ja pulber) või ränidioksiidi- (peen või jäme) lisanditega, ning nende erosioonkulumisele. Eesmärgiks oli saada keraamilise kompositsiooni parandatud erosioon vastupi davusega, mida saaks kasutada rakendustes, kus nendest materjale valmistatud toetus- ja/või kaitseelemendid töötaksid erosioonitingimustes toa- ning kõrgetel temperatuuridel. Valmistati ja katsetati kolme tööstuslikult valatava keraamikat (SiO₂, Al₂O₃ ning ZrO₂ baasil) koos ja ilma lisanditeta, samuti vedelast klaasil põhinevat kompositsioonide kulumise mõju kuju.

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