Research of plasma impact on migration of silicon in ceramic substrates prepared on the basis of MgO

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Abstract. To solve a number of problems in metallurgy and construction, it seems promising to obtain physical and chemical methods of coatings on ceramic materials. In particular, the plasma-arc method makes it possible to obtain coatings with a thickness of 100–200 μm from oxides and carbides. To solve this problem, it is necessary to investigate the interaction processes of the ceramic substrate and the coating that occur during plasma spraying. In this work, ceramics with different Si contents with a deposited Al₂O₃ coating were selected as the object of study.

1. Introduction
At the first stage of the work, it was found [1] that when using casting molds consisting of oxides in the casting process of titanium alloys: MgO (92.3 wt.%), CaO (3.2 wt.%), SiO₂ (2.2 wt.%) and liquid glass Na₂O (SiO₂) n in an amount of 12 wt.% With respect to the basic composition of the form, the formation of an alpha layer on the surface castings. One of the main reasons for the oxidation of Ti is the migration to the surface of Si and its compounds due to the resulting temperature gradient when the melt contacts the form.

2. Experiment
One way to reduce the alpha layer is by spraying plasma coatings on casting molds. The temperature of the plasma jet of the arc plasmatron at distances of 100–150 mm from the anode nozzle varies from ~ 4000 K to ~ 2000 K, therefore, when the coating is sprayed, penetration of the Si and its compounds onto the coating surface is also possible.

Comparison of the elemental composition and atomic masses of the elements allows us to conclude that the ceramic material consists of metal oxides and SiO₂ (the main material).

An Al₂O₃ coating was sprayed onto the surface in a mode that ensures the absence of significant thermal loads on the surface part of ceramics. A powder of Al₂O₃ particles with a size of ≤ 100 μm was used. The thickness of the deposited layer was measured on transverse sections of the sample using an electron microscope. The spraying modes were selected, providing a coating thickness of ~ 100 μm (± 10 μm), the distance between the anode cut and the sample surface is 150 mm.

The study was performed using a Philips XL 30 ESEM-TMP scanning electron microscope with an energy dispersive spectrometer EDAX C-UTW. The purpose of electron microscopic analysis was to study the samples using topographic contrast methods, visualize the surface structure and conduct an elemental composition study using energy dispersive spectroscopy. An increase of 100 - 2500 times was used. From each sample a 3mm by 3mm segment split off. Further, using carbon tape, these
segments were fixed on specialized holders. Then the samples were placed in an electron microscope chamber for the purpose of studying the elemental composition in the low vacuum mode. The mode of low vacuum was chosen in order to exclude the procedure of sputtering onto the surface of a conductive carbon film at the stage of spectral studies. At the second stage of the study, a 50 nm thick carbon film was applied to the samples. This procedure was performed in order to organize the charge drain from the surface, which in turn makes it possible to obtain images of the surface morphology of the sample with high resolution.

Table 1. The composition of the material on the basis of silicon dioxide

| Element | Wt, % | At, % |
|---------|-------|-------|
| O       | 50.88 | 65.64 |
| Na      | 1.01  | 0.91  |
| Mg      | 1.32  | 1.12  |
| Al      | 7.39  | 5.66  |
| Si      | 32.10 | 23.59 |
| K       | 1.61  | 0.85  |
| Ca      | 0.71  | 0.36  |
| Ti      | 0.44  | 0.19  |
| Fe      | 4.53  | 1.68  |

3. Results

Figures 1, 2 show the results of studying the surface of the coating using an electron microscope.

![Figure 1](image_url)  
**Figure 1.** The spectra collection areas are 50 µm by 50 µm. Spectral analysis was performed in 150 µm increments.
Figure 2. Surface morphology of the sample with plasma Al$_2$O$_3$ coating

Table 2 The composition of the transition layer.

| Element | Spectr 19 | Spectr 20 | Spectr 21 | Spectr 22 |
|---------|-----------|-----------|-----------|-----------|
|         | Wt, %     | At, %     | Wt, %     | At, %     | Wt, %     | At, %     |
| O       | 49.28     | 62.13     | 49.68     | 62.67     | 50.97     | 63.68     | 51.24     | 65.34     |
| Al      | 49.01     | 36.64     | 49.39     | 36.94     | 49.03     | 36.32     | 42.07     | 31.81     |
| Ti      | -         | -         | 0.93      | 0.39      | -         | -         | 6.69      | 2.85      |
| Si      | 1.71      | 1.23      | -         | -         | -         | -         | -         | -         |

Figure 3. The end part of the sample with a cross section of the coating.
Repeated measurements on several samples prepared by the same method show that at ≥ 90% of scanning areas the Si content (At%) does not exceed 2, in 50% of measurements it is less than 1. The distribution of silicon over the coating cross-section has been studied. The regions of the set of spectra are 10 µm per 10 µm. Table 3 presents the spectral analysis, which was carried out in increments of 15 µm from the surface to the ceramic base.

Table 3 Spectral analysis of the surface with a ceramic base.

| Spectr 27 | Spectr 28 | Spectr 29 | Spectr 30 |
|----------|----------|----------|----------|
| Element  | Wt, %    | At, %    | Wt, %    | At, %    | Wt, %    | At, %    |
| O        | 53.08    | 65.66    | 53.95    | 66.44    | 50.11    | 63.17    | 50.34    | 63.19    |
| Al       | 44.32    | 32.51    | 43.61    | 31.85    | 44.48    | 33.25    | 44.80    | 33.34    |
| Si       | 2.60     | 1.83     | 2.43     | 1.71     | 4.53     | 3.25     | 4.85     | 3.47     |
| Fe       | -        | -        | -        | -        | 0.88     | 0.32     | -        | -        |

Table 4 provides a comparison of the elemental composition of the base material at the border with the coating (left column) with the main ceramic material (right column). A small change in the ratio of aluminum and silicon indicates the penetration of the sprayed material into the pores on the surface.

Table 4 Comparison of the elemental composition of the coated base material.

| Element | Wt, % | At, % | Wt, % | At, % |
|---------|-------|-------|-------|-------|
| O       | 52.14 | 66.79 | 50.88 | 65.64 |
| Na      | 0.89  | 0.79  | 1.01  | 0.91  |
| Mg      | 1.34  | 1.13  | 1.32  | 1.12  |
| Al      | 11.02 | 8.37  | 7.39  | 5.66  |
| Si      | 27.04 | 19.73 | 32.10 | 23.59 |
| K       | 1.60  | 0.84  | 1.6   | 0.85  |
| Ca      | 0.94  | 0.48  | 0.71  | 0.36  |
| Ti      | 0.35  | 0.15  | 0.44  | 0.19  |
| Fe      | 4.67  | 1.71  | 4.53  | 1.68  |

During the deposition of plasma coatings on a ceramic material with a high silicon content, migration to the surface of the active metals and their compounds due to the thermal effect of the plasma jet is insignificant. The concentration of Si and its compounds on the surface can be reduced by increasing the thickness of the plasma coating.

References
[1] Kashapov N F, Dautov I G, Tkachenko L A, Fadeev S A, Shaidullin L R 2018 Calculation of the distribution of temperature in the form based on magnesium oxide for the casting of titanium products Journal of Physics: Conference Series 1058(1) № 012062