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The Possibility of Using of Aluminium–Titanium Intermetallics to Obtain Heat-Resistant Metal Coatings on ZrO_2- and Al_2O_3-Ceramics

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The intermetallic compound Al_3Ti is tested for metallization of the ZrO_2 and Al_2O_3 ceramics. For ZrO_2, the contact angle of Al_3Ti melt is of 35°, and for Al_2O_3, it is of 47°. Metallization is carried out by both infiltration of titanium powder layer applied on the ceramic surface with molten aluminium and further heating up to melting of formed Al_3Ti. The coating with high adhesion to ceramics is obtained for ZrO_2 ceramics, which can withstand heating in air up to 1000°C for 60 min. For Al_2O_3 ceramics, results are much worse, probably, due to high deoxidation of the interface.

Key words: metallization, zirconia, alumina, Al_3Ti, wetting, infiltration, heat resistance.

Інтерметалід Al_3Ti був випробуваний для металізації ZrO_2- й Al_2O_3-керамік. Для ZrO_2-кераміки контактний кут змочування розтопом Al_3Ti становив 35°, а для Al_2O_3-кераміки — 47°. Металізацію проводили шляхом просочування шару порошку титану, нанесеного на поверхню кераміки, розтопленим алюмінієм з подальшим нагріванням до розтоплення утвореного Al_3Ti. Для ZrO_2-кераміки одержано покриття з високою адгезією до кераміки, яке витримує нагрівання на повітрі до 1000°С впродовж 60 хв. Для Al_2O_3-кераміки результати були набагато гіршими, ймовірно,

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через високий ступінь розкиснення поверхні поділу.
Ключові слова: металізація, діоксид Цирконію, оксид Алюмінію, змочування, термостійкість.

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1. INTRODUCTION

Titanium is often used in joining (brazing, metallization) ceramic materials, primarily oxides, as a dopant to filler alloys based on tin, copper, silver, nickel, *etc*. The adhesion properties of metal melts containing titanium have been studied in many works [1–3]. Fillers compositions, brazing and soldering technologies have been developed that provide sufficiently high strength characteristics of joints.

The disadvantage of such technologies is the low heat resistance of fillers and metallization coatings due to the low melting point, as well as the oxidation of titanium. Since ceramic joints are required in devices operating at high temperatures (turbine parts, internal combustion engines, electron-beam equipment, fuel cells, *etc*.), it is necessary to develop high-temperature fillers with increased heat resistance and technologies for their application [4–12]. Until now, oxygen-free types of ceramic materials have been mainly studied [5–12]. As for oxide materials, aluminium–titanium alloys with high aluminium content can be used for their metallization and soldering. When metallic materials with high aluminium content are oxidized, an oxide layer consisting of Al_2O_3 is formed on their surface, which ensures high heat resistance of the alloys [13, 14]. At the same time, aluminium and titanium have high affinity for oxygen, which should ensure good wetting of oxide materials by these alloys [15].

In the aluminium–titanium system [16], there is an intermetallic compound Al_3Ti, which is a suitable candidate, since its melting point is 1377°C and the aluminium content is high enough to provide oxidation resistance. Al_3Ti also has fairly good mechanical properties, including high-temperature ones, for example, the strength is 700–900 MPa [17]. In this work, Al_3Ti is tested for metallization of oxide ceramics based on Al_2O_3 and ZrO_2.

2. MATERIALS AND METHODS

Single crystals and ceramics made of ZrO_2 stabilized with 3.5 mol.% Y_2O_3, Al_2O_3 ceramics and sapphire were used. The surface of the ceramic and single crystal samples was polished with 0.7–0.3 μm diamond powder. High-purity aluminium and titanium, and technical-grade titanium powder were used to prepare the metallization composition.

Wetting was studied on the single crystals using the sessile drop method in a vacuum of $5\cdot10^{-5}$ Pa. The melt was formed directly during the experiment: titanium and aluminium weights corresponding to the Al–25Ti composition were prepared, the titanium part was placed on the surface of the single crystal, the aluminium part was placed on it and at a temperature of about 800°C aluminium and titanium reacted to form Al_3Ti. Then, the samples were heated to melting of the Al_3Ti and contact angles were measured.

Metallization was carried out by the infiltration method: a layer of titanium powder was applied to the ceramic surface in the form of a suspension in an organic solvent and then part of aluminium was placed on the surface covered with titanium and heated in a vacuum. When melted, aluminium infiltrates in titanium, reacts with it and a loose porous layer of Al_3Ti is formed. With further heating to 1377°C, Al_3Ti melts and covers the ceramic surface with an even layer.

3. RESULTS AND DISCUSSION

In the wetting experiments, the equilibrium contact angle after melting Al_3Ti and reaching a temperature of 1400°C was set during several seconds. For ZrO_2, the contact angle was 35°, and for Al_2O_3, 47° (Fig. 1). However, after cooling, the Al_3Ti drop did not stay on the Al_2O_3 surface and broke off easily. There were not any visible signs of interaction on the sapphire under the drop; the base of the drop was the same colour as its top surface.

Samples of Al_2O_3 and ZrO_2 ceramics were metallised. The coating thickness was of 0.5–0.7 mm; photographs of the samples are shown in Fig. 2. For Al_2O_3, the joint strength of the coating to the ceramics was very low, the metallization layer peeled off spontaneously (Fig. 2, *a*), but, for ZrO_2 ceramics, a uniform coating with high adhesion to the surface of the oxide material was obtained (Fig. 2, *b*).

The heat resistance of the metallised zirconia-ceramics samples was estimated by annealing in air at 1000°C for 60 min. The metallised surface turned white, but the thickness of the oxide film was no more than 5 µm. There was no peeling of the metallised layer from the ceramic

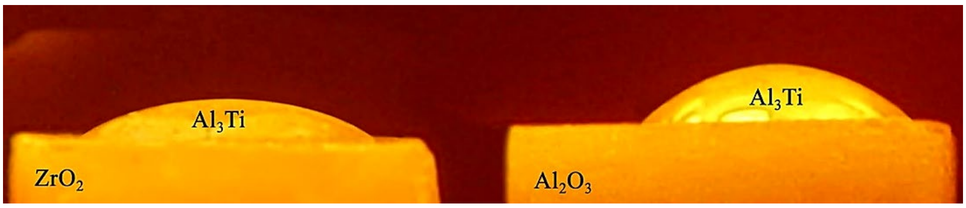

Fig. 1. Drops of the Al_3Ti melt on the ZrO_2 single crystal (left sample) and sapphire (right sample).

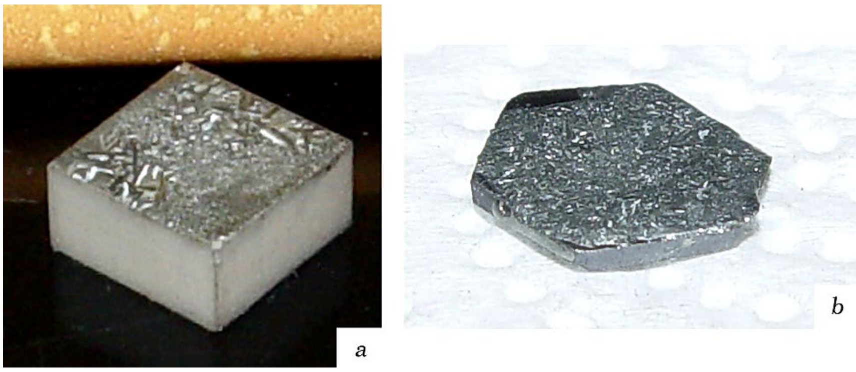

Fig. 2. Al_3Ti metallised Al_2O_3-ceramics (*a*) and ZrO_2-ceramics (*b*) samples.

Fig. 3. ZrO_2-ceramics metallised with Al_3Ti after annealing in air at 1000°C during 60 min.

surface. It should also be noted that the ZrO_2 ceramics, which, after metallization in a vacuum, turned black due to the loss of oxygen, turned white again after annealing in air (Fig. 3).

With drops of the Al_3Ti melt on the ZrO_2 single crystal (left sample) and sapphire (right sample), a cross-section of the metallised sample was made. Its micrograph is shown in Fig. 4.

The formation of a transition layer at the oxide–metal boundary is not visible on the cross-section, and there are also no signs of oxidation of the metallization coating.

The reason of absence of strong joint between Al_3Ti and Al_2O_3 ceramics after cooling despite good wetting is not clear and require a special consideration. Next explanation may be proposed: the strong joint of aluminium and its alloys to solid oxides is provided by formation of thin Al_2O_3 interlayer on the interface, which has high adhesion to both

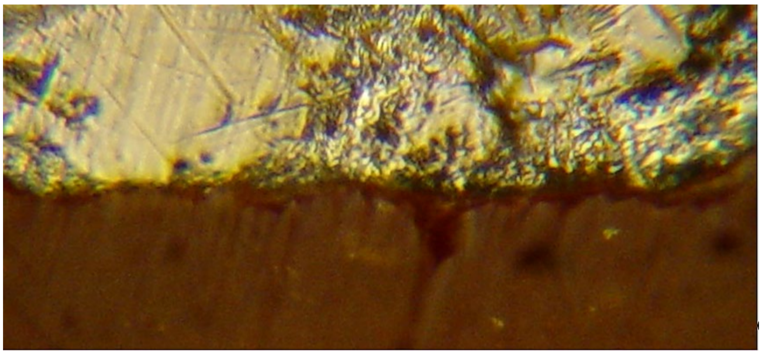

Fig. 4. Microstructure of Al_3Ti–ZrO_2-ceramics transition zone for sample annealed in air (×1000).

substrate and metal [18, 19], but presence of titanium in the melt increases the solubility of oxygen within it [20], thus, deoxidizing the melt and preventing the formation of a transition Al_2O_3 layer. At the same time, zirconia ceramics has high mobility of anions, so, supplies enough oxygen to form a transition layer despite titanium oxidation.

4. CONCLUSIONS

The use of Al_3Ti as a composition for metallization of zirconia ceramics allows obtaining metallised samples with increased heat resistance. This opens up the possibility of obtaining heat-resistant brazed joints of ceramics with metal, if a heat-resistant metal is used as the metal part of the joint.

The Al_2O_3 does not form strong joint with Al_3Ti despite of good enough wetting. This phenomenon needs additional study. The absence of strong joint between Al_3Ti and Al_2O_3 can be used in refractory technology, where adhesion of melt to crucible or mould is undesirable.

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