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Hardening of Ceramic Heat-protective Layers on Metallic Substances

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Abstract

We developed a technology of hardening heat-protective ZrO_2-Y_2O_3 ceramic coatings. The method includes reinforcement of ceramics by ceramic fibers and reinforcement of metal substrates using nichrome spirals. The microstructure of the coating, strength characteristics and heat resistance were studied.

Keywords: composite coatings, zirconium dioxide, yttrium oxide, ceramic fiber, metal substrates, heat resistance.

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1. INTRODUCTION

The efficiency of ZrO_2-Y_2O_3 heat-protective coatings is sharply reduced when their thickness exceeds 1 mm, due to substantial thermal stress at the "base-coating" boundary during heating and cooling [1-3]. The lack of plasticity in the ceramic layers and their insufficient adhesion to the metal substrate lead to the reduction of stress via formation and spreading of cracks at the "base-coating" boundary, which results in the detachment of the coating [4]. To create thick-layer ceramics and to preserve the necessary requirements for heat resistance, multilayer structures are used, e.g. gradient layers with a variable content of the sublayer and ceramic components between them [5]. The authors of [6] developed a special intermediate layer as a low-modulus compliant deformation compensator (BRUNSBOND gasket) for a more reliable attachment regarding thick layers of ceramics onto the surface of a metal part. Such a gasket made of low modulus sintered metal fiber is brazed to the metal surface. It acts as an elastic stress absorber during thermal cycling. We have developed a technology to obtain more durable thick-layer oxide coatings with increased thermal stability.

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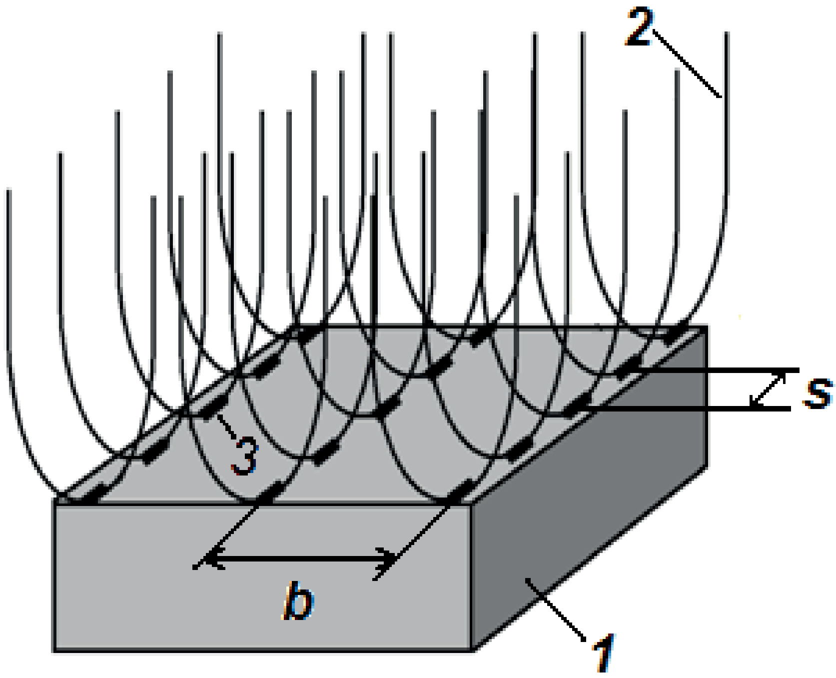

Figure 1: Scheme of reinforcing elements attached to the metal substrate: 1 - substrate, 2 - reinforcing elements, 3 - places of soldering.

2. EXPERIMENTAL

2.1. Substrate preparation

At the initial stage, the 12C18H10T stainless steel metal substrate was prepared with an arrangement of metal wire spirals (nichrome). The helices were attached to the surface of the substrate with VPr-11-40N high-temperature solder [7]. After this, the upper crests of the spirals were cut and the created "whiskers" were orientated perpendicular to the surface, as shown in Fig. 1. The bending diameter of the nichrome spirals (d) depends on the thickness of the coating (h) and should be

$$\frac{h}{2} < d < \frac{h}{1,25}$$

The optimal pitch of the spiral turns (s) is 2-4 mm. For s < 2 mm, the defectiveness of the ceramic layer was significantly increased, and its deposition was technologically challenging. At s ≥ 4 mm, the reinforcement efficiency of the metal surface was reduced. Recommended spacing between spirals is $b \leq 2d$.

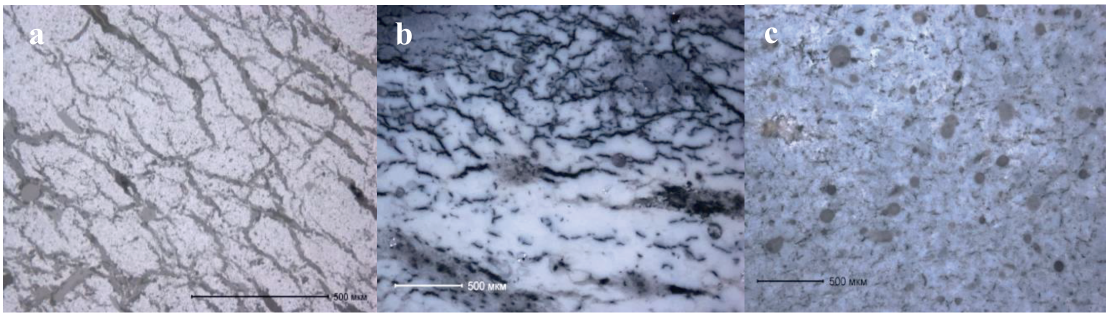

Figure 2: Microstructure of the coating material: a - co-precipitation; b - thermohydrolysis; c – hydrothermal treatment.

2.2. Synthesis of powders

For the production of ceramic heat-shielding coatings, we previously carried out studies with ZrO_2-Y_2O_3 powders synthesized in different ways.

- co-precipitation of zirconium and yttrium hydroxides.
- synthesis under thermal hydrolysis at 60 ° C with a solution of zirconium and yttrium nitrates in the presence of sulfuric acid.
- hydrothermal treatment of the solution at pH 8.

Fig. 2 is a comparison between the microstructures of coating materials synthesized by various methods. Studies have shown that the best results were achieved for coating material obtained from a powder synthesized with hydrothermal treatment. The structure of the material was more uniform throughout the surface with the lowest number of defects. These powders were subsequently used to produce thick-layer coatings.

2.3. Preparation of coating material

To reinforcing the coating material, we used discrete ceramic fibers obtained from a fibrous material with the following composition: ZrO_2 - 14-17%; Al_2O_3 - 50-56%; SiO_2 - 27-36% by grinding them in an aqueous medium with mixer. It was determined that 15-25 sec was the optimum grinding time to obtain fibers of medium length 200 μ ± 60 μ (standard deviation) (Fig. 3). Composite "ZrO_2 - 7%Y_2O_3 - ceramic fiber" was produced by slurry technology. For this, ZrO_2-7% Y_2O_3 powders were post-milled in a ball mill, mixed with the resulting discrete fiber and dried to the constant weight. Then a thick slip with paraffin binder was prepared from the mixture, and this was subsequently used for the application of the thick-layer coatings.

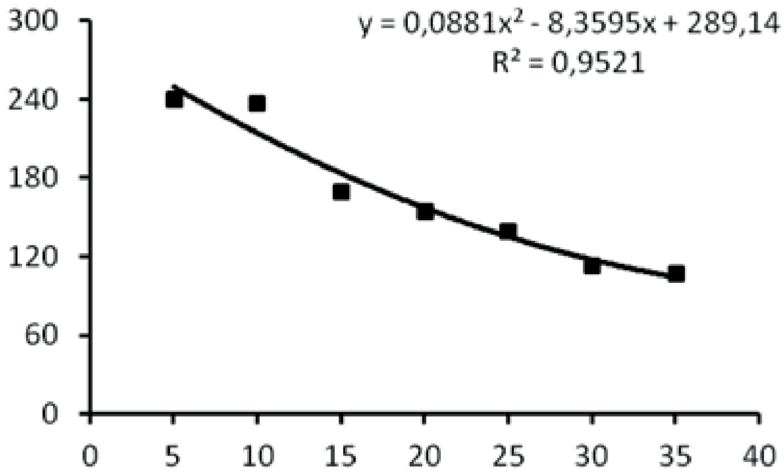

Figure 3: Dependence of average length of fibers on grinding time.

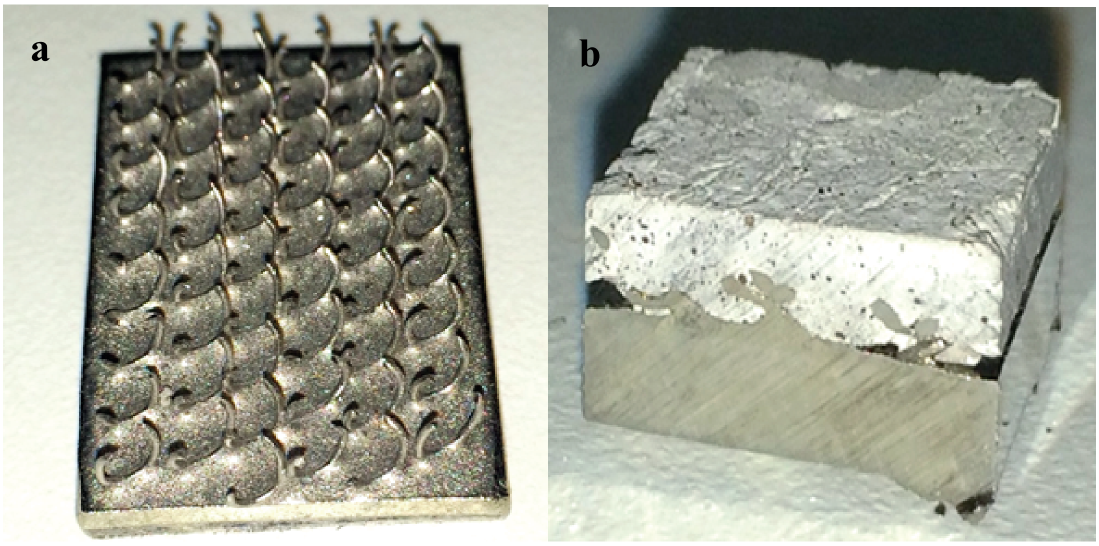

Figure 4: Reinforced substrate of 12C18H10T stainless steel before coating (a) and after coating (b).

2.4. Application of coating

On a preliminary stage, a sublayer of a mixture including nickel and aluminum powders was applied to a substrate of steel 12C18N10T with a reinforced surface (Fig. 3). After that, a layer of thick slip with paraffin were applied. The resulting composition was compacted by pressing, drying and calcining at 1200 °C under vacuum. The microstructure of the "base-coating" boundary is shown in Fig. 4.

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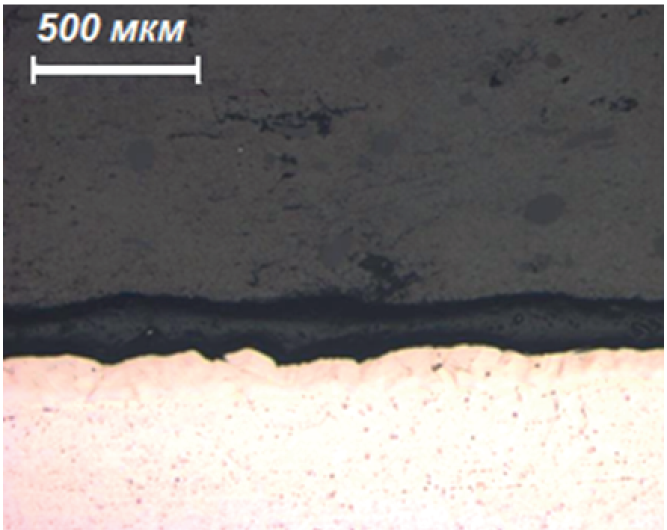

Figure 5: Microstructure of the composite coating ZrO_2 - 7% Y_2O_3 - ceramic fiber on 12C18H10T stainless steel.

3. RESULTS AND DISCUSSION

Nickel aluminide Ni_3Al forms due to the exothermic reaction between nickel and aluminum in the deposited sublayer during the vacuum annealing with a temperature of 1200 °C, and partly at the boundary with the iron aluminide substrate. This contributes to a further hardening of the ceramic layer on the metal substrate. It was found that the content of aluminum in the mixture is in average 10-15 wt. %. With a lower aluminum content, hardening is not sufficient. At more than 15 wt. % aluminum, the sublayer becomes brittle, which increases the number of microcracks in the interaction zone.

Studies of thick-layer coatings adhesion strength were carried out according to the adhesive technique. For this, the plates were cut into 10 × 10 mm pieces and a special grip was attached to the substrate. On the side of the ceramic layer, a second grip was fastened with epoxy glue [8]. Measurement of the adhesion strength showed that a cohesive separation occurs over the coating body at a pressure/pull with up to 10 MPa (Fig. 6).

The main characteristics of coatings are determined: density, open porosity, thermal stability. The thermal stability of the coatings was evaluated based on the reduction in the strength properties of the coating material under thermal cycling conditions: heating to 1100 °C and cooling in water (Table 1). It was found that the introduction of ceramic fiber with up to 10 wt.%. leads to a decrease in strength of the ceramic composite. Probably, the reason is a significant increase in the porosity of the ceramic layer reaching 40%. However, increasing of fiber content in the composite contributes to the production of more stable materials, which can be used under thermal cycling

Figure 6: Destroyed specimen after the adhesion test.

conditions. Consequently, composites with a fiber content of more than 13 mass % practically did not sinter at 1200 °C and were destroyed when unloaded from the furnace.

Table 1: Influence of fiber content in composites on their properties.

Content of fiber, wt. %	Density, g/cm^3	Porosity, %	Flexing strength, MPa	
			After sintering	After 20 thermal cycles
1	4.80	14	49	2
2	4.19	24	39	4
3	4.09	26	27	5
5	3.84	29	22	7
7.5	3.25	35	18	8
10	2.87	40	15	10

4. CONCLUSIONS

The technology of thick-layer heat-resistant composite coatings "ZrO_2-7% Y_2O_3-ceramic fiber" has been developed, which increases the heat resistance with thermocycling. The optimum compositions for thick-layer heat-resistant coatings are determined. The basic characteristics of coatings are estimated: density and porosity, bending strength, heat resistance and adhesion properties.

The resulting composite coatings can be recommended for use as thermal protection materials.

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