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Resistance to High-Temperature Gas Corrosion of Chromaluminizing Coatings with a (Ti, Zr)N Layer on Nickel

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The paper presents the results of a study on the thermal insulation properties of polymer composites based on fly ash from thermal power plants. Aqueous dispersions of styrene-butadiene (Latex 2012) and acrylic (Policril 590) polymers were used as the matrix. A complex multifactorial dependence of the thermal conductivity of the composite materials on the type and properties of the ash, its content, the type and concentration of the polymer dispersion, density, porosity, and temperature was established. The decisive role of the pore structure in forming effective thermal insulation was demonstrated. It was found that a more developed specific surface area, higher wettability, and lower filler density contribute to the reduction of thermal conductivity. A tendency for thermal conductivity to increase with rising composite density was identified, which is explained by a reduction in the volume of air pores. lowest thermal conductivity was shown by composites containing fly ash with a filler concentration of 65 wt.% in combination with an acrylic matrix. This particular ash consist of: 46.1 wt.% silicon dioxide, 18.0 wt.% aluminum oxide, 22.2 wt.% iron oxide, with the total content of alkali and alkaline earth oxides reaching 7.6 wt.%. The acrylic matrix demonstrated thermally stable behavior within the investigated temperature range. The developed materials are characterized by a low thermal conductivity, acceptable density, and high potential for use as lightweight thermal insulation coatings in energy-efficient technologies, construction, and other industries. The obtained results provide a basis for determining the optimal conditions for the production of efficient thermal insulation materials utilizing industrial waste.

Keywords: Barrier layer, Heat resistance, Microhardness.

1. INTRODUCTION

Analysis of contemporary events in the world and Ukraine, in particular, increases interest and relevance of the issue of proper use of mineral resources, specifically metals and their alloys. Approximately 25 countries conduct audits and assess the crucial term «criticality of raw materials». As noted in [1], the classification is based on two key factors-supply disruption risks and the economic significance of raw materials.

Currently, there are specific risks for countries with a high level of technological development. Their economies' dependence on critical raw materials is increasing yearly. At the same time, this issue is also relevant for less technologically developed countries in terms of responsible extraction and processing. The issue of responsible consumption is crucial for the development of all countries worldwide.

Several studies critically analyze this issue. It is known that the list of critical and strategically important chemical elements is periodically updated. For example, lithium has changed its status from a non-

critical to a critical raw material [2]. However, it is necessary to consider not only the availability and accessibility of raw materials but also their economic importance. For instance, according to [2], nickel is not classified as a critical raw material, although its moderate economic importance suggests that trends should be monitored.

However, as stated in [1, 3], demand for nickel is expected to increase in the coming years, mainly due to its leading role in energy production and the transition to digital technologies, especially in battery production for electric vehicles and low-carbon energy generation technologies.

Currently, the industries using nickel, ranked from highest to lowest consumption, are as follows: manufacture of machinery and equipment, Manufacture of basic metals, Manufacture of other transport equipment, Manufacture of electrical equipment, Manufacture of fabricated metal products, except machinery and equipment [2].

The aerospace industry should be highlighted separately, as nickel and its alloys are widely used in this

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field [4].

Additionally, nickel is widely used as a coating material [5]. Electrolytic nickel coatings are considered both decorative and promising for enhancing wear resistance, heat resistance, and corrosion resistance.

As stated in [3], there are several approaches to addressing the existing and potential future shortage of critical rare materials: partial or complete replacement of these materials with alternative non-critical ones, additive manufacturing of metal alloys [6], and the use of secondary raw materials (recycled materials).

The application of protective coatings extends the service life of finished products, enhances surface properties, and achieves similar results when combined with the substitution of base materials. These methods are believed to significantly reduce the consumption of base materials. In particular, the application of protective coatings on nickel-based alloys is discussed in [7]. Three enrichment methods were analyzed, and it was determined that the best protective properties are provided by a complex treatment combining High-Velocity Oxygen Fuel (HVOF) spraying of AMPERIT 405.001 powder and vapor phase aluminizing (VPA) using CrAl 70/30 with AlF_3 activator. This treatment forms a two-layer protective coating consisting of an inner adhesion layer (NiCoCrAlY) and an outer diffusion aluminide layer (NiAl). The inner layer protects the turbine blade material from high-temperature corrosion, while the outer layer shields it from combustion product flow at elevated temperatures. The authors of [8] evaluated the heat resistance of protective coatings applied to nickel-based alloys. The inner layer of the two-layer coating ensures thermal-physical compatibility between the coating and the base material, while the outer layer serves as thermal protection. The inner layer was applied using plasma spraying, while the outer layer was aluminized using diffusion techniques, including non-contact gas-phase aluminizing, suspension-based methods, and plasma-sprayed ceramic coatings. It was found that a protective coating with an MCrAlY-type inner layer applied by plasma spraying and an outer layer aluminized using a non-contact gas-phase diffusion method effectively protects the turbine blade material from oxidation and ensures thermal insulation.

The study [3] examines the potential use of thermal barrier coatings (TBCs), which can be effective in extending the service life of nickel-based alloys. In particular, such coatings ensure the safe operation of components under high/low temperatures, severe thermal shocks, high pressures, mechanical loads, radiation, or corrosion. One of the key mechanisms by which these coatings enhance performance is reducing surface temperature. A simple two-layer TBCs system for protecting nickel-based superalloys has been proposed. The inner layer is designed to shield the base material from oxidation and high-temperature corrosion while also improving adhesion with the outer ceramic coating, mitigating the thermal expansion mismatch between the top layer and the substrate. The recommended phase composition for this layer is β-NiAl or MCrAlY. The outer layer consists of Yttria-Stabilized Zirconia (YSZ). During high-temperature operation, a thermally grown oxide (TGO)-primarily aluminum oxide – is formed due to oxidation, acting as an interface product

between the inner and outer coating layers [9].

Another promising method for enhancing the high-temperature gas corrosion resistance of nickel-based alloys has been proposed in [10]. The process of simultaneous saturation of the alloy surface with titanium, aluminum, and chromium was carried out in a mixture of metal powders, aluminum oxide (Al_2O_3), and ammonium chloride (NH_4Cl). It has been demonstrated that, as a result of titanium-aluminochromization, a multiphase, multilayer coating is formed on the surface of the alloys.

Metal materials actively corrode at high temperatures in an oxidizing environment. Resistance to high-temperature oxidation is based on the ability of metals to form a uniform, dense, high-adhesion oxide film on the surface. For high-temperature product applications, such as aircraft turbines, alloys based on refractory metals with high strength at temperatures of 1000-2500°C cannot be used in air or other oxidizing environment without a heat-resistant coating. The main alloying elements that increase the heat resistance of alloys are aluminium, chromium, and silicon.

The analysis of literary sources [11-14] showed a high ability of aluminides to resist aggressive environments at elevated temperatures, as they contain a sufficient amount of aluminium to form an Al_2O_3 film. Aluminium oxide Al_2O_3 has many useful properties, such as low cost, low density, good wear resistance, corrosion resistance. Unfortunately, the Al_2O_3 compound has limited plasticity at room temperatures, low high-temperature strength, and low crack resistance. Such significant improvement of these properties can be achieved by doping the coating. In particular, the formation of an aluminium coating on steels leads to the formation of iron-aluminium coatings, titanium-aluminium coatings on titanium alloys, and nickel-aluminium coatings on nickel [14-17]. In work [16], it was shown that the presence of active elements in a small amount in the material improved the stability of oxidation coatings.

An essential component of modern heat-resistant coatings is the barrier layer, which affects the phase composition, distribution of elements, structure of coatings during chemical and thermal treatment, and also reduces the rate of change in the phase composition of coatings during heat resistance tests [17, 18]. In study [19], the barrier function of a titanium nitride (TiN) layer for operation under extreme conditions was analyzed. The study determined that multicomponent coatings based on Ti, Al, and Si applied to nickel and its alloys – in terms of composition, structure, and properties – can be promising for use under high temperatures, aggressive environments, and severe wear conditions.

Ni-based alloys are well-known materials with high mechanical properties at elevated temperatures. But nickel and its alloys do not have sufficient heat resistance. To use nickel alloys at high temperatures, it is necessary to modify the surface to increase its corrosion resistance. The formation of compounds on the surface, for example, nickel intermetallics, is one of the ways to increase heat resistance [11, 18, 20]. Studies on obtaining, researching the structure and properties of chrome-alloyed coatings with a barrier layer on nickel and its alloys are limited.

Thus, the work aims to obtain multicomponent chromaluminizing coatings of nickel with a barrier layer of (Ti, Zr)N. In addition, the goal is to establish the phase and chemical compositions, analyze the structure, and determine the microhardness and heat resistance of coated nickel at a temperature of 1000°C. Based on the data obtained, practical recommendations for further implementation of the proposed technology of chromaluminizing of nickel in production will be developed.

2. MATERIALS AND METHODS

As the base material for coating application, carbonyl nickel was used. The carbonyl nickel samples contained an iron impurity of 0.03-0.04 % wt.

The diffusion coatings were applied by the powder method in containers with a fused seal using a mixture of the following composition: Cr (40 % wt); Al (15 % wt); Al_2O_3 (40 % wt); NH_4Cl (5 % wt). The process temperature was 1050°C, and the time was 4 hours.

The coatings, investigated in this research, were applied using two main methods:

1) chromaluminizing (1050 °C; during 4 hours);

2) (Ti, Zr)N coatings applied by physical vacuum deposition (PVD) from the gas phase followed by chromaluminizing (1050 °C; during 4 hours).

The coated samples were examined using modern physical materials science methods: X-ray phase, micro-X-ray spectral, metallographic, and durometric analysis.

The phase composition of the coatings studied in this work was determined using a Rigaku Ultima IV diffractometer in Cu Kα-radiation ($\lambda = 0.1541841$ nm). The Bragg-Brentano focusing scheme was used, which allows recording lines with reflection angles from 20 to 90° with step $\Delta 2\theta = 0.04$ deg. The accelerating voltage in the tube was 40 kV, the current was 40 mA, and the exposure time in point 2 s. The X-ray diffraction patterns were interpreted, and the lattice periods of the obtained phases were determined using the PowderCell 2.4 software package according to the Rietveld method (complete profile analysis). The obtained diffractograms were compared with the reference diffractograms in the ICDD PDF-2 diffractogram database.

Samples for electron microscopic studies were prepared according to the standard method in the form of flat cross-sections pressed on Struers UniForce and PrestoPress machines into conductive Struers PolyFast resin, followed by polishing on Struers Abramin grinding and polishing machines using 1 μm diamond paste and suspension.

The microstructure of the samples was studied on a Carl Zeiss SIGMA 300 SEM in phase contrast mode (backscattered electrons) with an accelerating voltage of 5 kV. A Carl Zeiss SIGMA 300 scanning electron microscope (SEM) equipped with an Oxford Instruments X-MaxN energy dispersive EDX spectrometer and INCA 5.05 software was used to determine the elemental composition of the coatings. Operating parameters of the SEM for EDX: accelerating voltage 5 kV; absorbed current 0.8×10^{-9} A. The content of elements in the coating and the base metal was measured consecutively by step scanning of the sample in the direction perpendicular to the side surface of the sample cross-section. SEM was performed at positions along the coating growth with a step of 1-2 μm and in zones located at different distances from the surface. Additionally, metallographic studies of cross-sections were carried out using an optical microscopy. A 50% nitric acid solution in ethanol was used as an etchant to reveal the substrate's microstructure.

Microhardness was measured on the LHVS-1000Z (Touch Screen) in at least 20 fields of view. The load on the indenter was N$\mu = 0.25$ N, the measurement error was 0.1 GPa.

The heat resistance was estimated by the change in the mass of the samples after their oxidation at 1000 °C for 100 hours under natural aeration using an analytical balance of the ML204/01 (Mettler-Toledo GmbH, Nänikon, Switzerland).

3. RESULTS AND DISCUSSION

The chemical compound (Ti, Zr)N applied to nickel before chromaluminizing (method 2) had a lattice period of 0.4379 nm. According to Wegard's rule, the concentration of zirconium in the (Ti, Zr)N compound with a lattice period of 0.4379 should be about 24.0 % wt Micro-X-ray spectral analysis showed a zirconium content of 24.4 % wt in the nitride. Thus, the compound on the surface of nickel with a thickness of 5.5-6.0 μm can be identified as $(Ti_{0.7}, Zr_{0.3})N$. The microhardness of the resulting layer is 20.6 GPa. The (Ti, Zr)N layer is light yellow in the optical microscope.

The microstructure and distribution of chemical elements in the thickness of the coatings obtained by the two methods in the initial state and after oxidation are shown in Figs. 1-3 The phase composition and distribution of chemical elements in the diffusion zone of the coatings can be distinguished into a compound zone and a transition zone.

The zone of compounds formed on the surface of nickel after chromaluminizing under the accepted conditions of chemical and thermal treatment consists of a layer of the β-NiAl phase with a thickness of 50.0-52.0 μm and a layer of Ni$_3$Al compound with a thickness of 3.0-5.0 μm. The β-NiAl layer can be divided into two zones in an optical microscope. The upper zone contains 60.0-70.0 % wt of nickel and is light blue. The lower zone contains about 80.0 % wt of nickel and has a light yellow color. The thickness of both zones is almost identical and ranges from 24.0 to 26.0 microns. After a light etching with Murakami reagent, the boundaries of the individual components of the coating are visible in an optical microscope (Fig. 1, b).

In this research, the presence of chromium in the light blue zone had been established, the concentration of which was 5.5-5.8 % wt on the outside of the coating and monotonically decreased to almost zero at the border with the light yellow layer (Fig. 1). A similar coating of two differently coloured zones based on the β-NiAl phase had been investigated in [18] after aluminizing of nickel at 950 °C in a mixture of ferroaluminium and ammonium chloride. Both zones' lattice periods were the same – 0.2866 nm [18]. The lattice period of the outer layer of the NiAl compound obtained after nickel chromoalloying was 0.2869 nm, which turned out

to be larger than the lattice period of the compound on alloyed nickel [18]. The latter is due to a chromium layer in the blue zone of NiAl with atomic dimensions larger than nickel's. This explanation will be valid for solid chromium solutions in the nickel sublattice.

It is known that when the nickel content in the β-NiAl phase is more than 50 % aluminum atoms are replaced by nickel atoms. The period of the crystal lat-

tice decreases because the atomic dimensions of nickel are smaller than those of aluminum. The lattice period also decreases when the nickel content is below 50 %. This is because the positions of nickel atoms remain vacant and are not filled with aluminum atoms. A completely different picture occurs when the NiAl phase is alloyed with chromium. The latter replaces the vacant nickel positions in the crystal lattice.
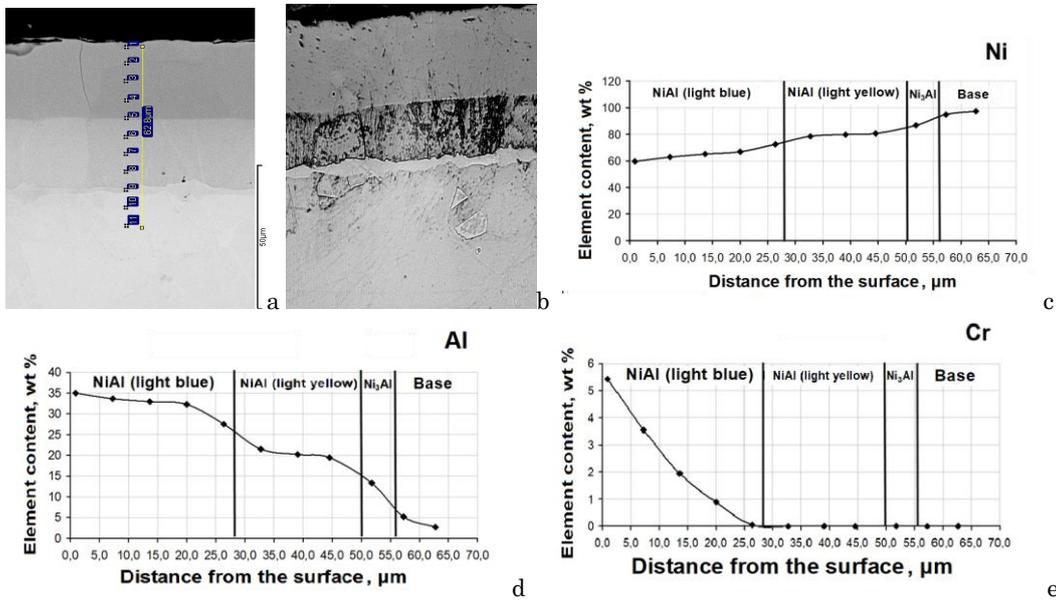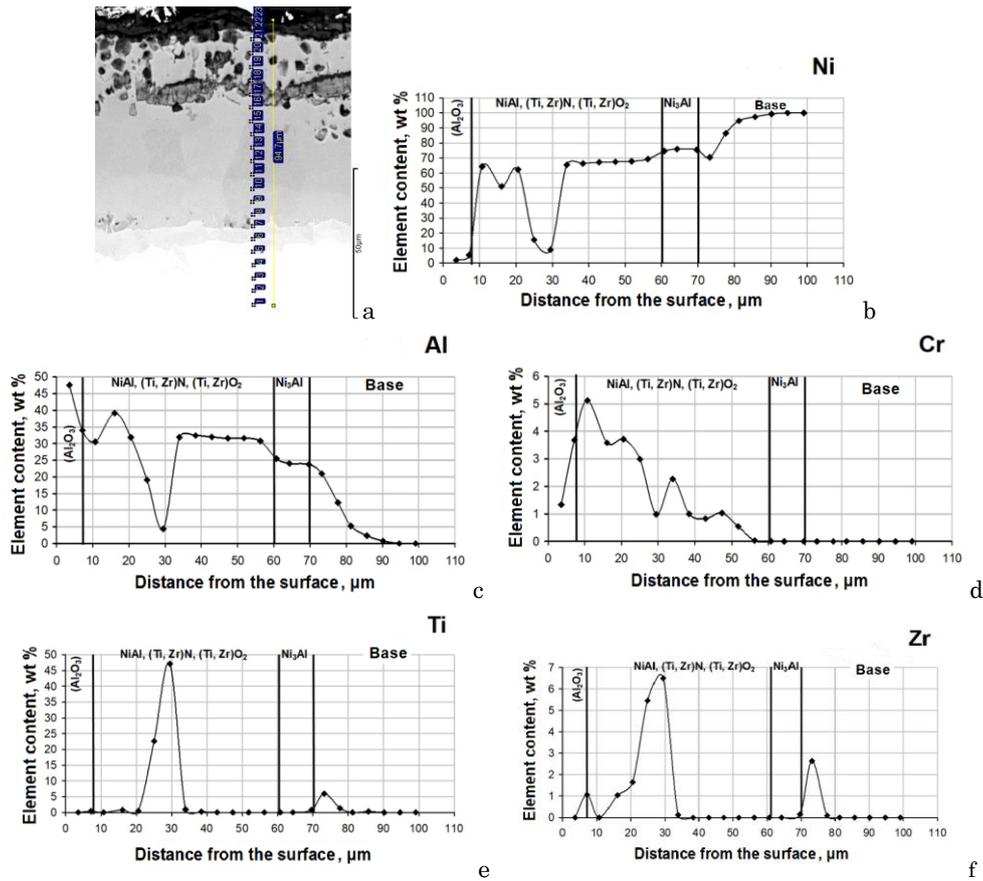

Fig. 1 – Microstructure of Nickel after Chromaluminizing; electron microscope (a); optical microscope, × 400 (b); chromaluminizing (1050 °C, 4 hours (method 1)); distribution of elements by coating thickness: Ni (c), Al (d), Cr (e)
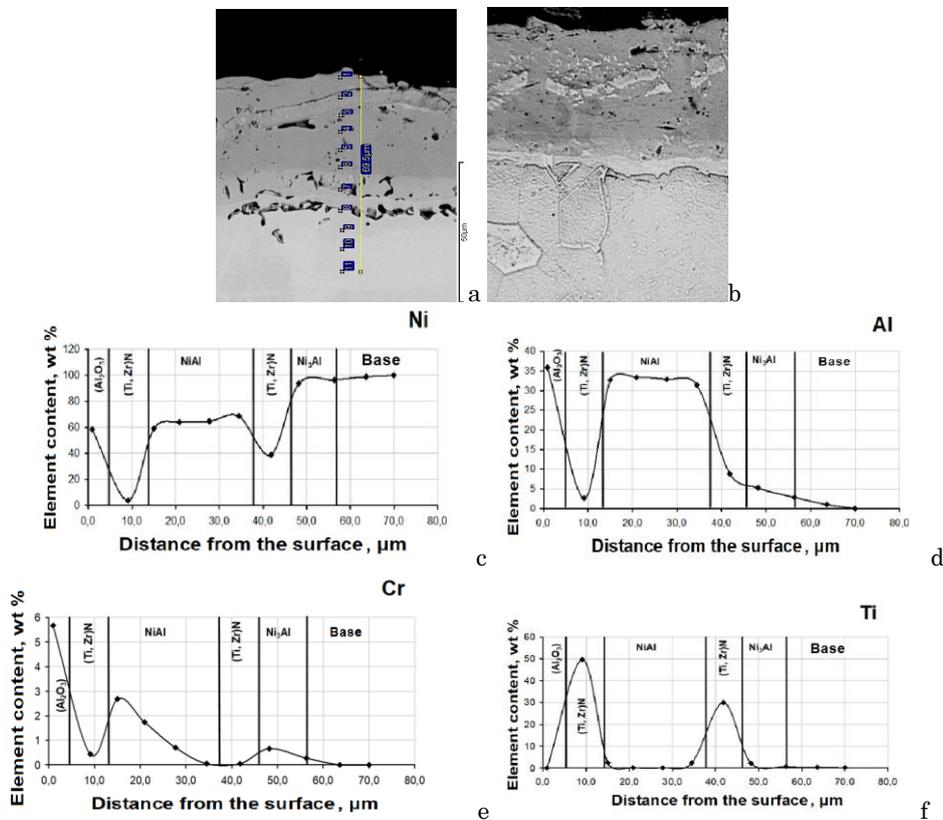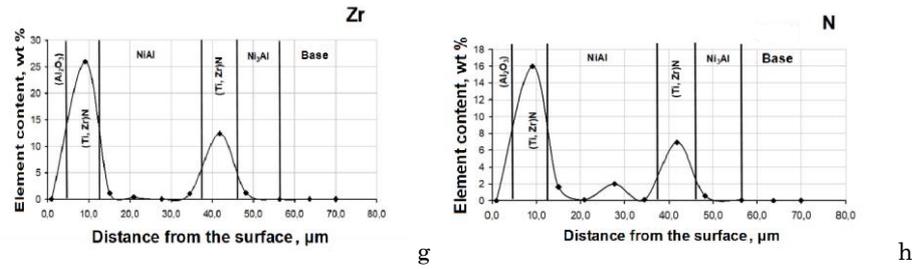
Fig. 2 – Microstructure of nickel after chromaluminizing with a layer of (Ti, Zr)N; electron microscope (a); optical microscope, × 400 (b); chromaluminizing (1050 °C, 4 hours (method 2)); distribution of elements by coating thickness: Ni (c), Al (d), Cr (e), Ti (f), Zr (g), N (h)

Fig. 3 – Microstructure of nickel after chromaluminizing with a layer of (Ti, Zr)N after oxidation; 1000 °C, 100 hours; electron microscope (a); distribution of elements in the coating thickness: Ni (b), Al (c), Cr (d), Ti (e), Zr (f)

The presence of chromium in the light yellow part of the β-NiAl phase layer was not detected.

A chromaluminizing coating on nickel with a (Ti, Zr)N layer consists of layers of the β-NiAl phase, the Ni$_3$Al phase, Al$_2$O$_3$ oxide, and the (Ti, Zr)N compound itself. A part of the (Ti, Zr)N compound layer looks like either a fractured strip curved from the surface to the base or individual particles in the NiAl phase layer (Fig. 3a).

Two zones can be distinguished in the structure of the nickel coating after chromaluminizing with a layer of (Ti, Zr)N oxidized at 1000 °C for 100 hours: the zone of Al$_2$O$_3$ oxides on the outside of the coating and the zone of internal oxidation. The zone of internal oxidation consists of oxidized (Ti, Zr)O$_2$ inclusions, a (Ti, Zr)N layer, and layers of NiAl and Ni$_3$Al compounds (Fig. 3a).

It should be noted that the (Ti, Zr)N layer, destroyed in many places, limited oxygen penetration. It was found that before the heat resistance tests, the particles from the destroyed layer of (Ti, Zr)N nitride were light grey. These particles darkened after oxidation, indicating the presence of oxygen. The lattice period of the (Ti, Zr)N compound was 0.4379 nm and, as seen from the results, was significantly different. An almost black band with a 1.5-2.0 μm thickness was formed on the outside. This band can be identified as a mixture of titanium and zirconium oxides.

It was shown that after oxidation of nickel after chromaluminizing, the thickness of the Ni$_3$Al layer had been increased from 20 to 25.0-30.0 μm, and to 10.0-15.0 μm on samples after chromaluminizing with a layer of (Ti, Zr)N.

The increase in the weight of the samples after the

heat resistance tests is shown in Fig. 4. Analysis of the data showed that at 50 hours of testing, chromaluminizing coatings have higher protective properties.
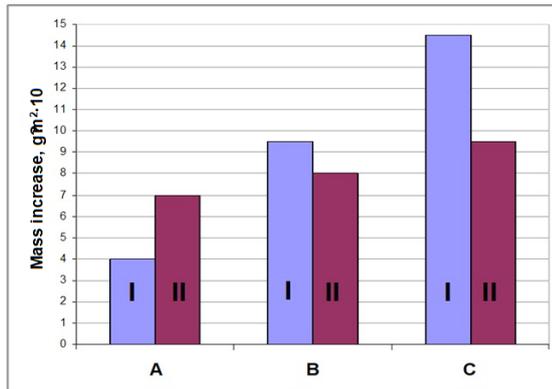
Fig. 4 – Mass increase of chrome-alloyed nickel during oxidation (I), chrome-alloyed nickel with a (Ti, Zr)N layer (II); oxidation time – 50 hours (A); 75 hours (B); 100 hours (C); temperature – 1000 °C

Having increased the oxidation time, coatings with (Ti, Zr)N oxidized at a lower rate. There was a high probability that in the first 50 hours, not only the surface of the coating actively interacted with oxygen, but also the inclusion of (Ti, Zr)N. At subsequent times, the penetration of oxygen into the coating was inhibited by the compound (Ti, Zr)N.

The maximum microhardness in the obtained coatings was established for (Ti, Zr)N layers – 22.5-23.5 GPa; for a layer based on the NiAl phase (light blue zone) – 5.5-7.5 GPa; NiAl (light yellow zone) – 4.5-5.0 GPa. The microhardness of the light blue zone turned out to be higher than the microhardness given in [18]. This may be related to the presence of chromium in the obtained coatings. Heat resistance tests did not affect the microhardness of the (Ti, Zr)N layer and reduced the microhardness of NiAl and was light blue – 5.0-7.0; light yellow – 4.0-4.5 GPa.

The multilayer chromaluminizing coatings with a (Ti, Zr)N layer can be recommended for protecting products made of nickel alloys that operate under conditions of high temperatures, aggressive environments, and contact loads.

4. CONCLUSIONS

1. The possibility of forming chromaluminizing coatings on nickel with the participation of a layer previously applied by the physical deposition method from the gas phase (Ti, Zr)N had been shown. The coating consisted of compounds Al_2O_3, NiAl, Ni_3Al; and the concentration of chromium on the surface reached 5 % wt.

2. It was found out that the heat resistance of nickel with chrome-alloyed coatings with the participation of (Ti, Zr)N was 1.5 times higher than the heat resistance of chrome-alloyed ones after tests at a temperature of 1000 °C for 100 hours.

3. The microhardness of individual layers in the coating was: (Ti, Zr)N – 22.5-23.5 GPa; NiAl (light blue zone) – 5.5-7.5 GPa; NiAl (light yellow zone) – 4.5-5.0 GPa.

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Стійкість до високотемпературної газової корозії хромоалітованих покриттів з шаром (Ti, Zr)N на нікелі

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У статті представлено результати дослідження теплоізоляційних властивостей полімерних композитів на основі золи виносу. В якості матриці використані водні дисперсії стирол-бутадієнового (Latex 2012) та акрилового (Policril 590) полімерів. Встановлено, складну багатофакторну залежність теплопровідності композиційних матеріалів від типу та властивостей золи, її вмісту, типу та концентрації полімерної дисперсії, густини, пористості та температури. Доведено визначальну роль порової структури у формуванні ефективного теплозахисту. Встановлено, що більш розвинена питома поверхня, висока змочуваність та менша густина наповнювача сприяють зниженню теплопровідності. Визначена тенденція до зростання теплопровідності зі збільшенням густини композитів, що пояснюється зменшенням об'єму повітряних пор. Композити з використанням золи при концентрації 65 % наповнювача та акриловій матриці демонструють найнижчі значення коефіцієнта теплопровідності. Дана зола містить: діоксид кремнію 46,1 %; оксид алюмінію 18,0 %; оксиду заліза 22,2 %; сумарна кількість лужних та лужноземельних оксидів становить 7,6 %. Акрилова матриця демонструє термічно стабільну поведінку в межах досліджуваного температурного діапазону. Розроблені матеріали характеризуються низьким коефіцієнтом теплопровідності, оптимальними показниками щільності, а також демонструють високий потенціал для застосування як легкі теплоізоляційні покриття в енергоефективних технологіях, в будівництві та інших галузях. Отримані результати дозволяють визначити оптимальні умови для створення ефективних теплоізоляційних матеріалів із використанням техногенних відходів.

Ключові слова: Бар'єрний шар, Термостійкість, Мікротвердість.