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**ELECTRODEPOSITION AND PROPERTIES
OF NICKEL COATINGS AND FOIL REINFORCED
WITH ALUMINA**

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The results of the electrochemical formation of composite coatings and foil based on nickel reinforced with nanoscale alumina are reported. Nickel based foil reinforced with the variable dispersion alumina phase content and adhesive composite coatings were deposited from nickel sulphamate electrolytes on polished steel substrates. The chemical top-down principle method is used for commercial Al_2O_3 dispergation. The hydrosol of alumina was received by dispersion of the high-temperature $\gamma\text{-Al}_2\text{O}_3$ in water solution at $\text{pH} > 13$ with the following élutriation of unreal solution. The influence of the alumina concentration in the electrolyte on the physic-mechanical properties of reinforced foil is established. Inclusion of reinforcing phase particles into the Ni matrix leads to a decrease in the grain size. The topography of the surface and cross-sectional profile of composites are studied by scanning probe AFM microscope and their influence on the properties of materials is established. The surface of synthesized Ni- Al_2O_3 composites is well developed, but the composite electrolytic coatings are more roughness, crystalline, characterized by a wide range of grain sizes within the same scan compared with a foil. The content of alumina 1,5 at.% (in terms of metal) in foil and composite coatings enhances microhardness, tensile strength and yield point of the material twice compared to monometallic systems.

In recent years, new technology development and their facility creation require materials with unique functional characteristics. One of the most advanced research vectors is the synthesis of composite coatings and foils based on metal matrix with the second phase inclusions [1]. As second phase dopants there are used oxides, carbides, silicides, borides, nitrides and other. The achieved composites combine both the properties of the metal and the dispersed phase. They have higher corrosion resistance, micro-hardness, improved durability and heat resistance. In practical terms the important properties of the composite coatings and foils are defined with the nature of the dispersed phase. As long as the improvement of the composite materials performance index is tighten with the combination of two different-nature

materials, it is necessary to use those materials which have the enough strong interaction with the metal matrix [2]. That is why it is an increasing interest in using the metal oxides (Aluminum, Zirconium etc.) as second phase materials [3]. The choice of alumina is based on its essential physico-mechanical properties, as follows, the improved hardness, crushing stress and high resistance to corrosion and cupping. More than that-the alumina is a low-cost material, which does not lead to critical rise of the self-cost [4].

Improved physico-mechanical properties without changing the metal crystal lattice are possible when adding less amount of strengthening phase. The new class of composite materials, meeting the same requirements, came up recently, and it is based on usage of sintered alumina, zircon dioxide and others, which size is about 1-100 nm [5]. The wide application of the nickel coatings is based on valuable properties, such as atmosphere corrosion resistance in alkali media and some of the acids. That's why they are used for improvement of steel chemical resistance in operating conditions, including under pressure and aggressive media influence. At the same time the specified coating are strained and fragile enough making it difficult to obtain a nickel foil and restricts electroforming [6]. These points are the base for major interest in composite electrolytic coatings (CEC) and foils development basing on nickel reinforced with nano-sized alumina.

Research Methodology

Nickel based foil reinforced with the variable dispersion alumina phase content was deposited on polished stainless steel AISI 304 substrates. Adhesive composite coatings with the same composition were settled on steel 20 [7]. Both types of deposits were deposited from nickel sulphamate electrolytes [8] of the following composition, g dm⁻³: nickel sulphamate 80-320; nickel chloride 7-20; boron hydroxide 25-40 with variable concentration of dispersion phase. The content of the second phase was within 1 to 2.5 g dm⁻³ in electrolyte solution.

Electrolytes were prepared of the certified reactive «pure grade» mark on the distilled water. The regulated power supply B5-47 was used for electrolysis, performed with current density 2-3 A dm⁻² at the temperature of 20-25°C during 30-40 min. The thickness of the received composites was 30-50 μm.

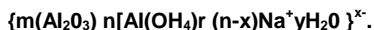
Physic-mechanical tests of the Ni-Al₂O₃ foils - micro-thickness, yield point σ_T , tensile strength σ_{np} , were held at room-temperature using mechanical testing machine TIRAtest-2300 with the scanning speed of 0.36 mm·min⁻¹.

The chemical composition of the coatings was analyzed by X-ray photoelectron spectroscopy with an INCA Energy 350 energy-dispersive

spectrometer; the X-rays were excited by exposure of the samples to a beam of 15 keV electrons. The surface morphology of the coatings was studied with a Zeiss EVO 40XVP scanning electron microscope (SEM). The surface roughness was evaluated by the contact method on 10x10x2 mm samples with an NT-206 scanning probe AFM microscope (lateral and vertical resolutions 2 and 0.2 nm, respectively; 1024x1024 scanning matrix, CSC cantilever B as probe, probe tip radius 10 nm).

Results and Discussion

The hydrosol of alumina was received by dispersion of the high-temperature γ - Al_2O_3 in water solution at $\text{pH}>13$ during 10-30 min with the following elutriation of unreal solution. Dispersion of the alumina particles achieves by partially dissolving amphoteric Al_2O_3 forming $[\text{Al}(\text{OH})_4]^-$. The last are adsorbed on Al_2O_3 surface defining the charge of colloid particle:



The stability of colloids is based on the presence of complexes $[\text{Al}(\text{OH})_4]^-$, which are more effective charge determine ions with the stability constant $K_s=3,2 \cdot 10^{32}$. The dispersion particles move to the cathode due to adsorption of Ni^{2+} on their surface. The particles holding on cathode initiate the nucleation in the points of contact with its surface stimulating the covering of these particles with the metal [9-11].

Analysis of the synthesized composite foils strength characteristic showed that while increasing the amount of alumina in electrolyte 0.25-1.5 $\text{g}\cdot\text{dm}^{-3}$ the microhardness increases in the range 1800-2900 MPa, yield point σ_T rises up to 150 - 980 MPa, tensile strength reaches 550-1200 MPa with the minor flexibility decrease. The reason of that is the incorporation into the Ni matrix adding into the main matrix the of Al_2O_3 nano-particles which are reliable restraint to dislocation moving. That is common to explain mechanism of reinforcement by Orován (rounding of the second phase particles by dislocations) [12].

The surface of synthesized Ni- Al_2O_3 composites is well developed, but the CEC (Fig. 1 a) are more roughness, crystalline, characterized by a wide range of grain sizes within the same scan compared with a foil (Fig. 1 b). It should also be noted that Al_2O_3 nano-particles tend to coalescence and formation of conglomerates. Profile intersection of crystallites between markers 1 and 2 shows that the grain size of CEC is 50-300 nm, while the foil - 180-200 nm. The difference between peaks and valleys of CEC relief distributes irregularly over the cross section and varies within 10-300 nm unlike evenly distributed difference of 100 nm to foil.

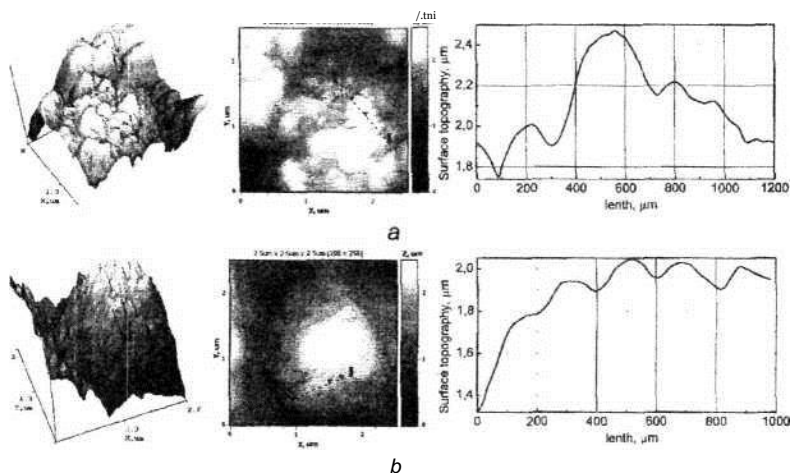


Figure 1. 3D and 2D surface maps and cross section profile between markers 1-2 for Ni-Al₂O₃ CEC (a) and foil (b) with Al₂O₃ content 1,5 at.% (in terms of metal). Scanning area 2,5x2,5 μm

The incorporation of the aluminum oxide nano-sized particles to the metal matrix leads to the metal crystallites size decreasing, which affects its mechanical properties. Decrease in crystallite size promotes grain boundaries increasing following by improving the mechanical characteristics of the material such as hardness [13, 14].

Conclusions

1. The compact fine-grained pore-free composite coatings, based on nickel matrix reinforced with the nano-sized alumina are electro deposited from sulphamate nickel bath containing chemically dispersed high-temperate oxide γ -Al₂O₃.

2. The incorporation of nanoscale reinforcing phase particles Al₂O₃ in Ni matrix reduces grain size and improving the mechanical properties of composites. The content of alumina 1,5 at.% (in terms of metal) in foil and CEC enhances microhardness, tensile strength and yield point of the material twice compared to monometallic systems.

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