STUDIES AND RESEARCHES REGARDING THE OBTAINING AND CHARACTERIZATION OF COMPOSITE NICKEL COATINGS-Ni/Al_2O_3, Ni/KAOLIN ELECTROCHEMICALLY PRODUCED

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ABSTRACT

This study presents the composite nickel coatings electrochemically obtained using as disperse phases Al_2O_3 particles and kaolin. There were used Watts electrolytes, with a pH = 4, current density of 4 A/dm^2, a concentration of the disperse phase of 20 g/l, a speed of agitation of 300 rpm, deposit strokes of 60, 90, 120 minutes. The characterization of those has been done microstructurally and also from the point of view of the film thickness and microhardness. The existing of the disperse phase particles has led to the shrinking of the nickel dimensions of crystalites, which has determined a decrease in roughness and an increase in hardness.

KEYWORDS: Ni composite coatings, electrodeposition, Al_2O_3, kaolin, microhardness

1. Introduction

Composite coatings are considered revolutionary materials, with real improvement perspectives of their properties and multiple utilizations in the aerospatial industry, marine industry, automotive industry, electric industry and electronic industry.

The major advantage, essential for composites is the possibility of properties modulation and by that obtaining a wide variety of materials. In most cases, the composite material comprises a core matrix in which is dispersed a material complementary to the form of fibers or particles.

Composite materials in the nickel matrix electrochemically obtained are characterized by high hardness, good wear behavior and high resistance to corrosion. As complementary materials, can be used the following types of disperse phases: oxides such as TiO_2, SiO_2, Al_2O_3, CeO_2, ZrO_2, SnO_2 and Cr_2O_3, carbides such as WC, TiC and SiC, nitrides such as Si_3N_4 and BN, carbon nanotubes [1].

Composite coatings Ni - Al_2O_3 are used in applications which require a high resistance to wear, corrosion, temperature, so they can be used as an alternative to chrome coatings. They can be used in the case of high pressure cylinder engines, molds, components of musical instruments, accessories machinery, electrical components, electronic microdevice [2].

Composite materials Ni – kaolin are used in the aerospatial industry, automotive industry, in applications which require a good behavior to wear, abrasion, fatigue, corrosion [3].

The properties of the composite materials depend on the type, structure, shape, the size, morphology and the content of the additional phase particles and their distribution in the metal matrix.

The functional properties of composite coatings are strongly influenced by their structure, tensions developed in the layer and also by the orientation and complementary phases which may cause the anisotropy of the properties.

Nickel has been chosen as a covering material because it is one of the most common industrial coverings used for decorative and functional applications [4].

Technical alumine used in experimental researches has almost 98.5% γ - Al_2O_3 and a maximum of 10% α - Al_2O_3.

α - Al_2O_3 crystallizes into a hexagonal lattice, has a density of 3.66 to 3.99 g/cm^3, a good resistance to high temperatures (T_t = 2054 °C), with higher mechanical properties (modulus of elasticity E = 440 GPa hardness H = 30 GPa). It has the form of colorless crystals or white and it is chemically inert.

γ - Al_2O_3 crystallizes in a face-centered cubic lattice, has a density of 3.65 to 3.67 g/cm^3, a modulus of elasticity of approx. 275 GPa, and a stability limit of 1200 °C.

Kaolin - Al_2O_3 * $2SiO_2$ * $2H_2O$ – is a clay (mineral) very fine-grained, white, with a density of 2.65 g/cm^3, low plasticity, with high resistance to high temperatures and by burning it becomes solid and very compact. A mineral layer has a triclinic crystal symmetry made of octahedral configurations composed of Al_2O_3 and SiO_2 tetrahedral parties that share a common plan-made of oxygen atoms. The layers thus formed are connected to each other by the links of hydrogen atoms [3].

Electro-co-deposition of the dispersed particles which is obtained in parallel with the electro crystallization of pure metal is presented in five steps [4]:

- the forming of the double electric layer around each of the particles after their introduction into the electrolyte;
- the transport of the particles to the hydrodynamic limit;
- the diffusion of the positively charged particles to the cathode;
- the reduction of free electroactive species or adsorbed by the particles;
- the embedding of the particles in the metal matrix.

The researches presented in this study have as a general objective the development of a technology of an electrochemical coatings of nickel composite matrix technique using Al_2O_3 as a dispersed phase with a size of approx. 5 μm and kaolin with a particle size of approx. 2 μm and their characterization in terms of microstructure of the layer thickness and hardness.

2. Experimental condition

In order to obtain nickel coatings and composite nickel coatings in nickel matrix it has been used a direct current source, a device provided with a magnetic stirrer and a temperature regulation system and a cell for the electrolyte bath. It has been used a solution volume of 300 ml, and the experiments have been made at 50 °C temperatures.

There were used electrolyte Watts-type consisting of $NiSO_4 \cdot 6H_2O$ - 300 g/l, $NiCl_2 \cdot 6H_2O$ - 50 g/l and H_3BO_3 - 40 g/l, pH = 4, with a current density of 4 A/dm^2, with the concentration of the dispersed phase 20 g/l, a stirring speed of 300 rpm, with a deposition time of 60, 90, 120 minutes.

The electrodepositions were conducted by the vertical arrangement of the electrodes at a distance of 14 mm from each other. As anode was used nickel in high purity (99%), the cathode being of copper strip, representing the support material for depositions at the size of 76 x 20 x 1 mm.

The copper strip used was prepared by degreasing (with organic solvents - trichlorethylene), etching (HNO_3 + HCl at the room temperature for 1 to 2 minutes) followed by washing with distilled water.

The metallographic analysis of samples was performed on a Neophot 2 microscope with the data acquisitions on a computer and it highlighted the appearance of the surface of the nickel depositions compared to composite coatings, their adherence, how they grew the electrodeposited crystals and the presence of flaws such as pores, cracks, exfoliations. To determine hardness was used a microdurimeter PMT-3, with a load of 50 g.

3. Experimental results

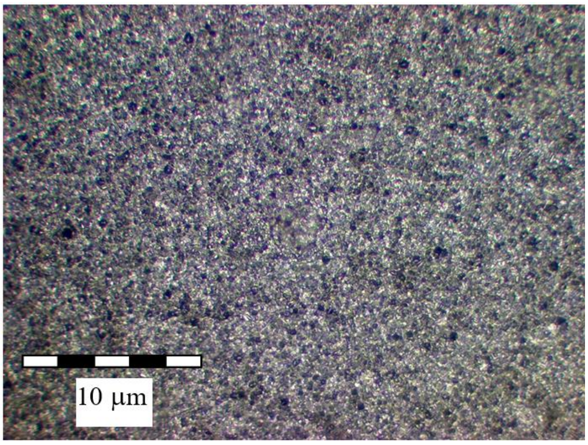
The characterization of ACE composite coatings was performed compared to pure nickel coatings and consisted of macro and microstructural analysis, determinations of the layer thickness, microhardnesses.

3.1. The characterization of the nickel matrix composite coatings with a dispersed Al_2O_3 phase electrochemically obtained

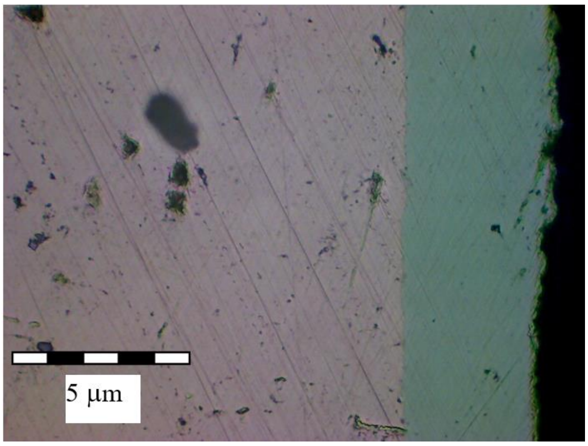
Fig. 1 shows the appearance of pure nickel coatings obtained at different electrodeposition times of coating. It can be seen that the deposited layers are homogeneous, no crackings and good adhesion to substrates of copper. It can be seen that by increasing the time of electrodeposition, the coating thickness increases, the current density and stirring speed being maintained constant.

Fig. 2 shows the microstructural appearance of the composite coatings, electrochemically obtained nickel matrix as particles of Al_2O_3 by embedding the dispersed phase into Ni matrix.

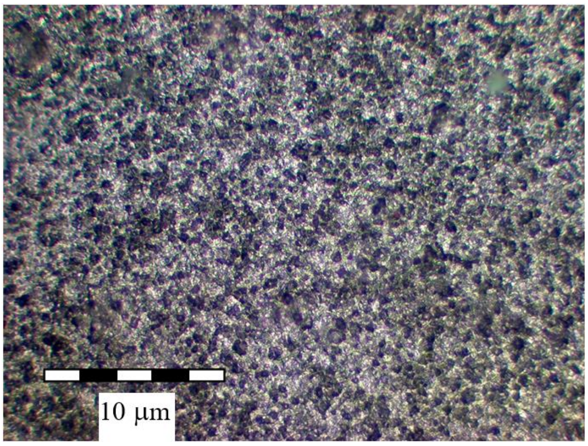
By looking at Fig. 2 it can be seen that the obtained composite electrochemical coatings in nickel matrix using as disperse phases Al_2O_3 particles exhibit good adhesion to the substrate, homogeneity and compactness. It can be seen that the presence of disperse phase particles, the best inclusion being at a time of deposition of 90 minutes. Al_2O_3 particles led to decreased crystallite size (compared to pure nickel deposits), which decreased the roughness deposits and increased hardness (it simultaneously occurs the blocking of the dislocations movements in the nickel matrix) (Fig 4).



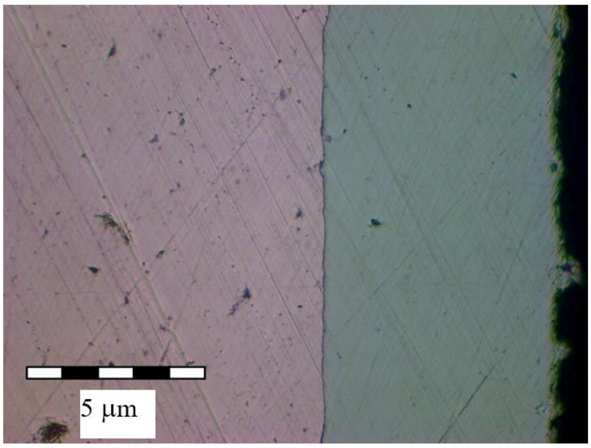
P1 – 60 minutes



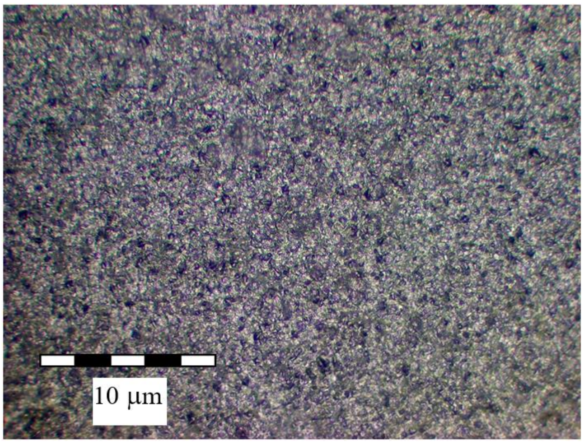
P1 – 60 minutes



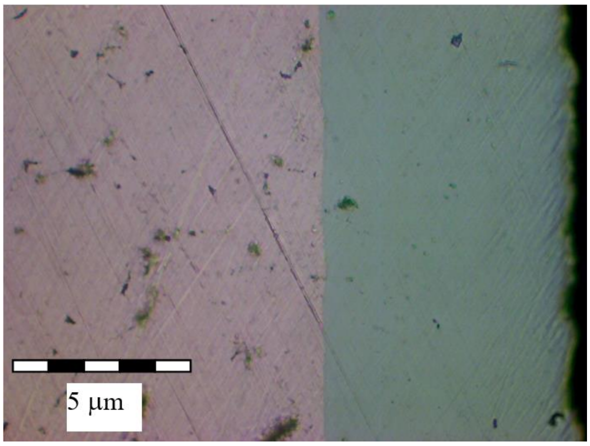
P2 – 90 minutes



P2 – 90 minutes

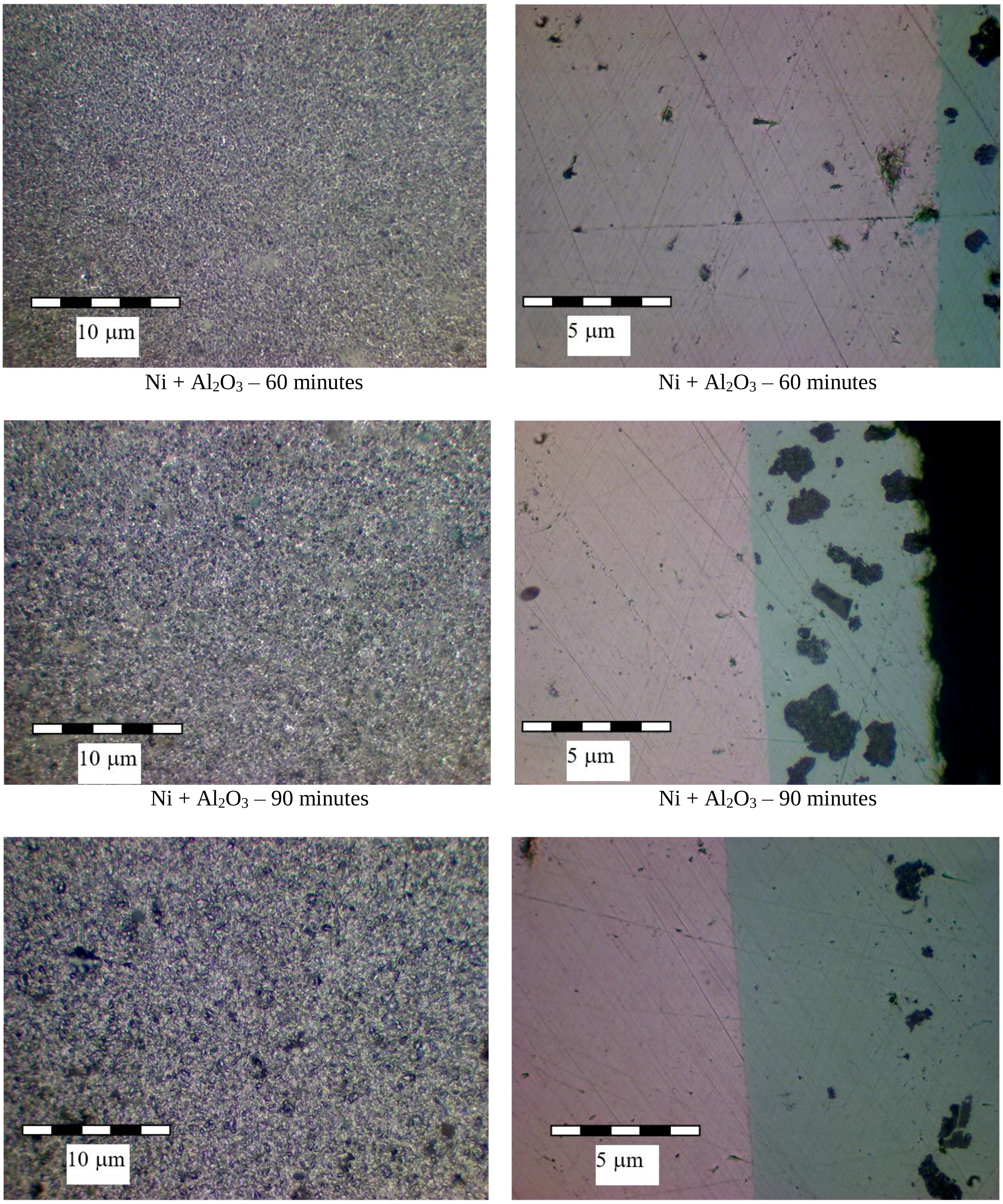


P3 – 120 minutes



P3 – 120 minutes

Fig. 1. *The microstructure of the pure nickel coatings*



Ni + Al_2O_3 – 60 minutes | Ni + Al_2O_3 – 60 minutes

Ni + Al_2O_3 – 90 minutes | Ni + Al_2O_3 – 90 minutes

Ni + Al_2O_3 – 120 minutes | Ni + Al_2O_3 – 120 minutes

Fig. 2. *The microstructure of the electrochemically composite coatings obtained in nickel matrix by using Al_2O_3 particles as dispersed phases*

Fig. 3 shows the deposition time influence on the thickness of pure nickel coatings and on composite coatings electrochemically obtained in nickel matrix using as disperse phases Al_2O_3 particles. It can be seen that by increasing the time of electrodeposition the coating thickness increases, the current density and stirring speed is maintained constant.

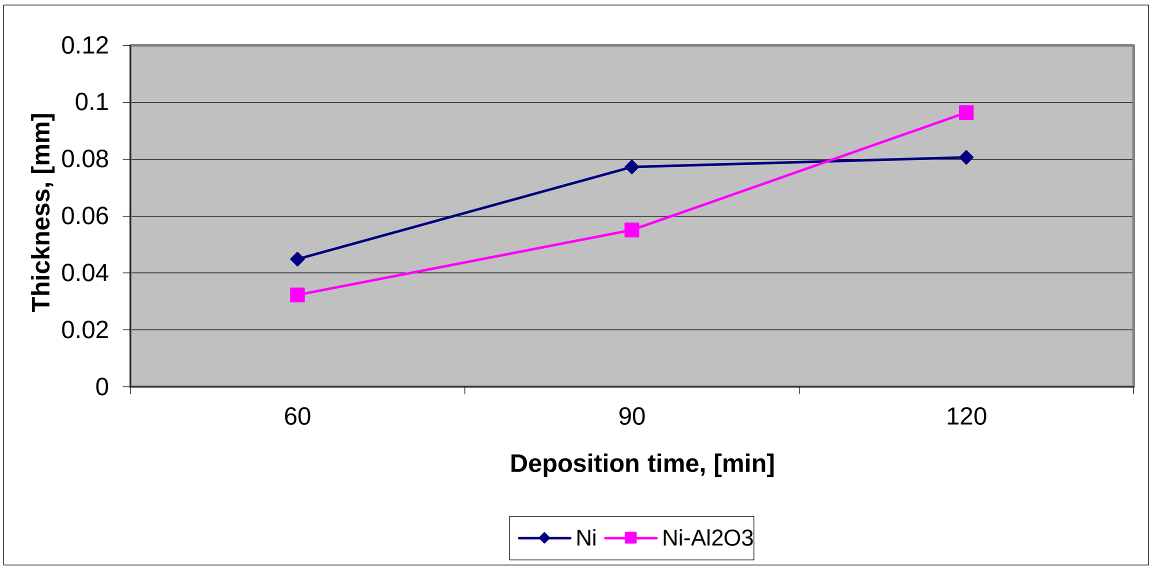


Fig. 3. *The influence of deposition time on the thickness of pure nickel coatings and on composite coatings electrochemically obtained in nickel matrix using as disperse phases Al_2O_3 particles*

Fig. 4 shows the deposition time influence on the microhardness of pure nickel coatings and on composite coatings electrochemically obtained in nickel matrix using as disperse phases Al_2O_3 particles. It may be noticed that in the case of pure nickel coatings and also in composite coatings electrochemically produced, in nickel matrix using as dispersed phases Al_2O_3 particles, it can be seen an increase of microhardness compared with cooper substrate which has a microhardness of 126.7 daN/mm^2. Increased hardness of pure nickel coatings with increased time deposition growth might be attributed to internal stresses in the deposited coating.

However, it appears that the presence of the Al_2O_3 dispersed phase leads to a further increase in the microhardness of obtained coatings compared to pure nickel coatings.

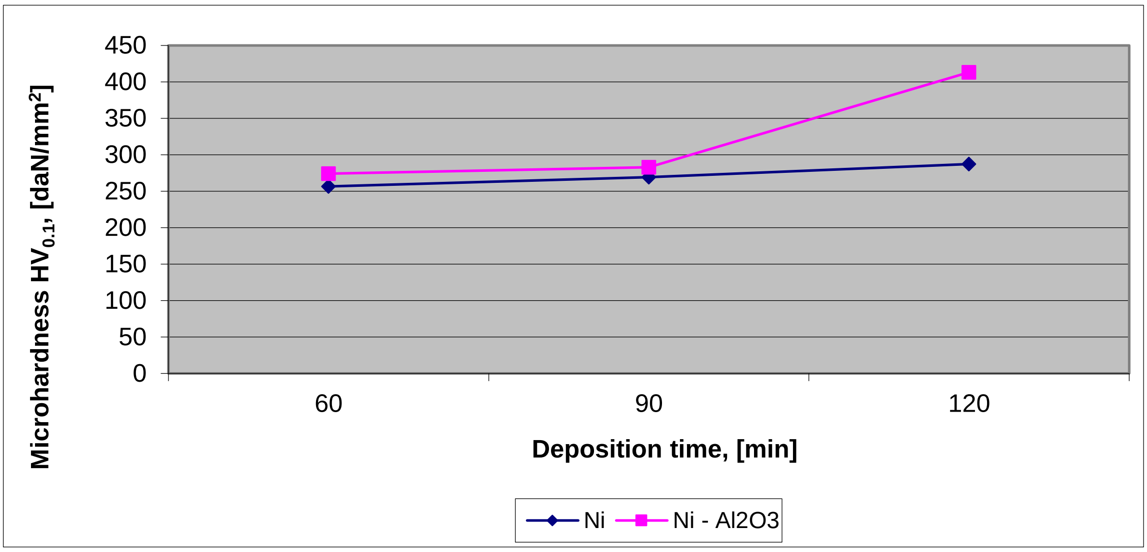
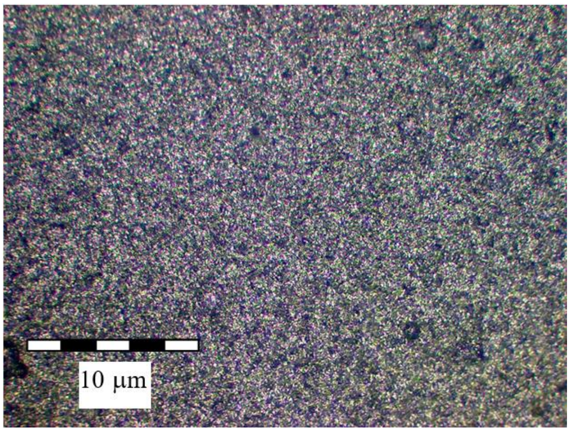


Fig. 4. *The influence of deposition time on the microhardness pure nickel coatings and also on the composite coatings electrochemically obtained in nickel matrix using as dispersed phases particles of Al_2O_3*

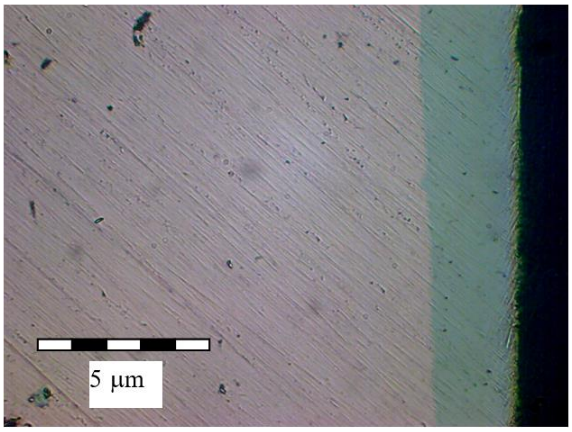
3.2. The characterization of composite coatings in nickel matrix electrochemically obtained at the disperse phase of kaolin $Al_2O_3 * 2SiO_2 * 2H_2O$

Fig. 5 shows the appearance of microstructural the coatings composition obtained electrochemically in the matrix of nickel using as the dispersed phase particles of kaolin.

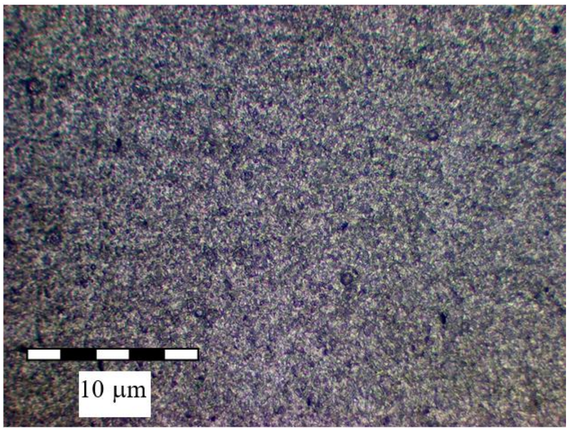
It can be seen that the deposited layers are homogeneous, no crackings and good adhesion to substrates of copper. The presence of particles of kaolin modifies the growth of crystals of nickel affording coatings smoother (as compared to coatings of the pure nickel), compact and continuous.



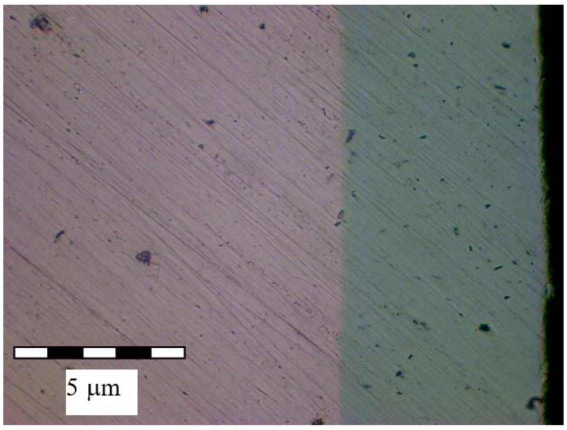
Ni – kaolin - 60 minutes



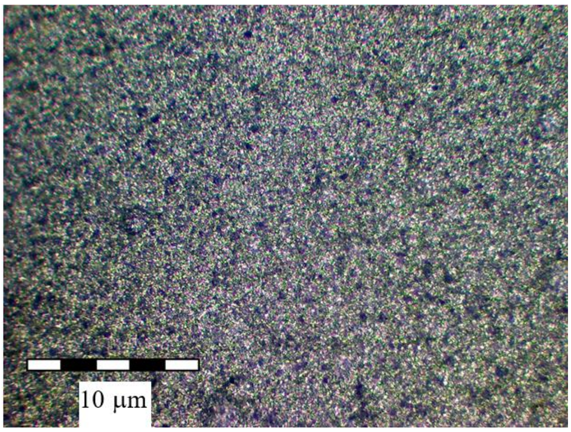
Ni – kaolin – 60 minutes



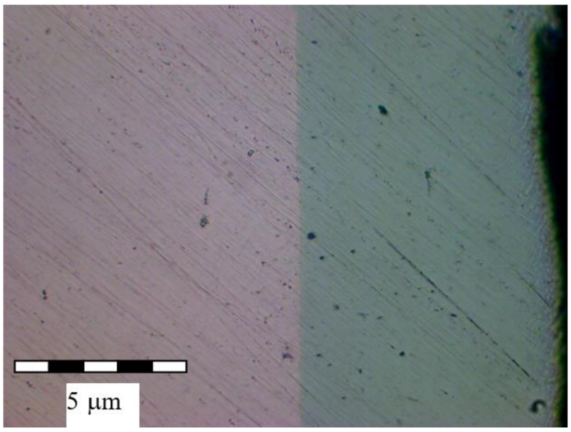
Ni – kaolin - 90 minutes



Ni – kaolin – 90 minutes



Ni – kaolin – 120 minutes



Ni – kaolin – 120 minutes

Fig. 5. *The microstructure of the electrochemically composite coatings obtained in nickel matrix by using the kaolin particles as dispersed phases*

Fig. 6 shows the influence of the time of deposition on the thickness of coatings composites produced electrochemically in the matrix of nickel using the phase dispersed kaolin clay particles. It may be noted that the increase in time deposit increases the thickness of the deposited layer.

Comparing the layer thickness of the deposit obtained at different times for the two types of dispersed phase particles it can be stated that the use of kaolin which has submicroscopic dimensions, resulting in a layer thickness higher than the case of using alumina particles, Fig. 7. So, kaolin particles show a better ability than embedding the alumina particles.

Regarding the microhardness of the obtained composite, electrochemical coatings in nickel matrix using as kaolin clay particles dispersed phases, we find that the increase in time deposit increases their hardness (Fig. 8). This increase is due to the increase in tension on the one hand during the deposition of the layer, and on the other hand a good embedding of the particles of the dispersed phase in the coating.

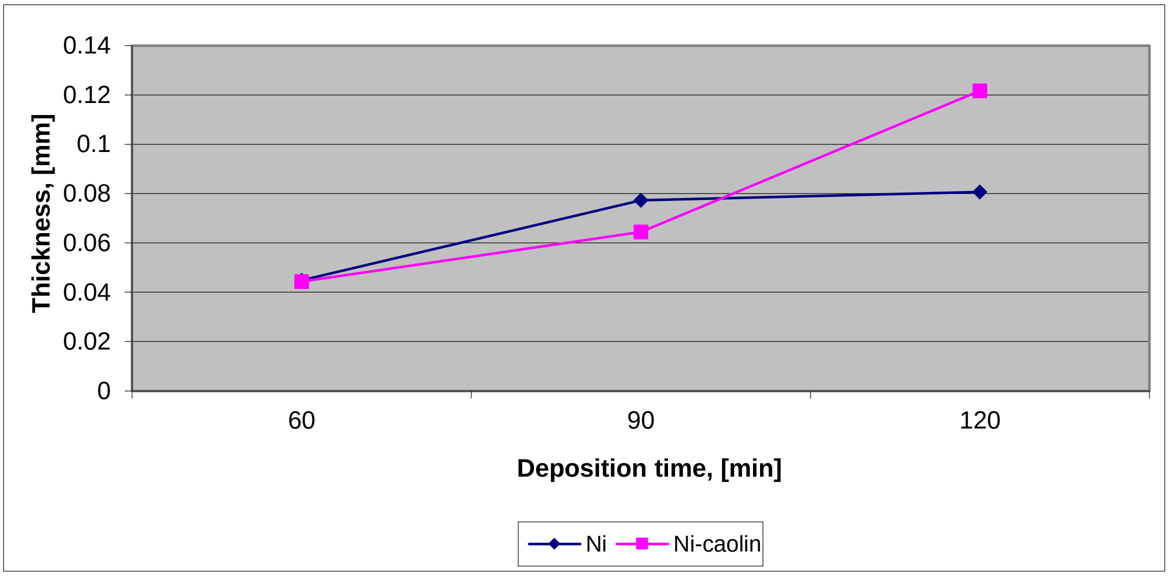


Fig. 6. *The influence of deposition time on the thickness of pure nickel layer coatings and composite coatings on electrochemically produced in nickel matrix using as dispersed phases kaolin particles*

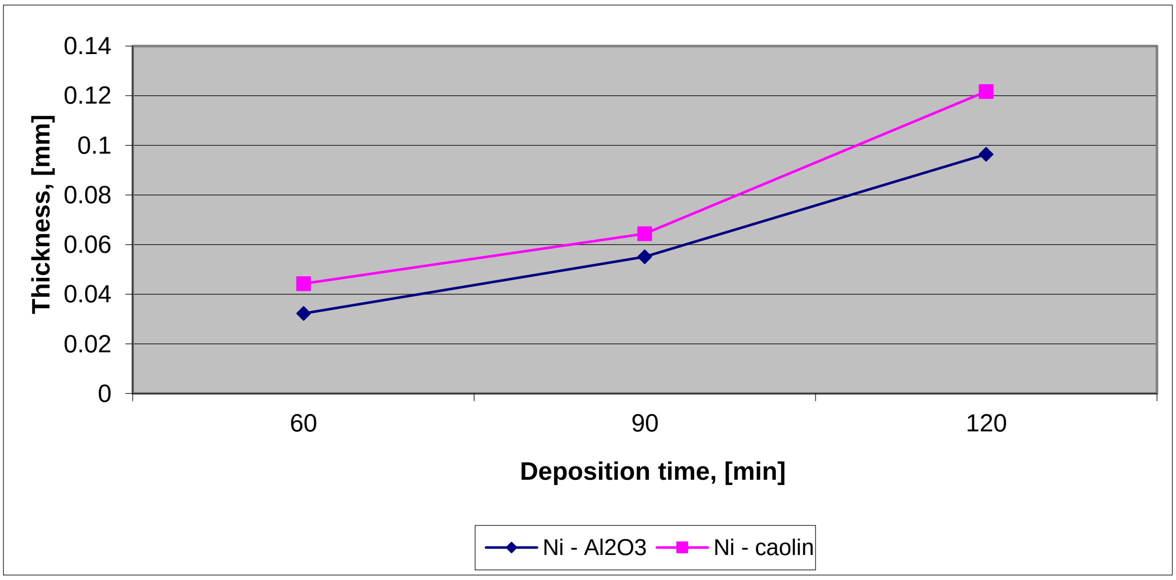


Fig. 7. *The influence of deposition time on the thickness of layer composite coatings electrochemically produced by using nickel matrix as dispersed phases* Al_2O_3 *particles and kaolin*

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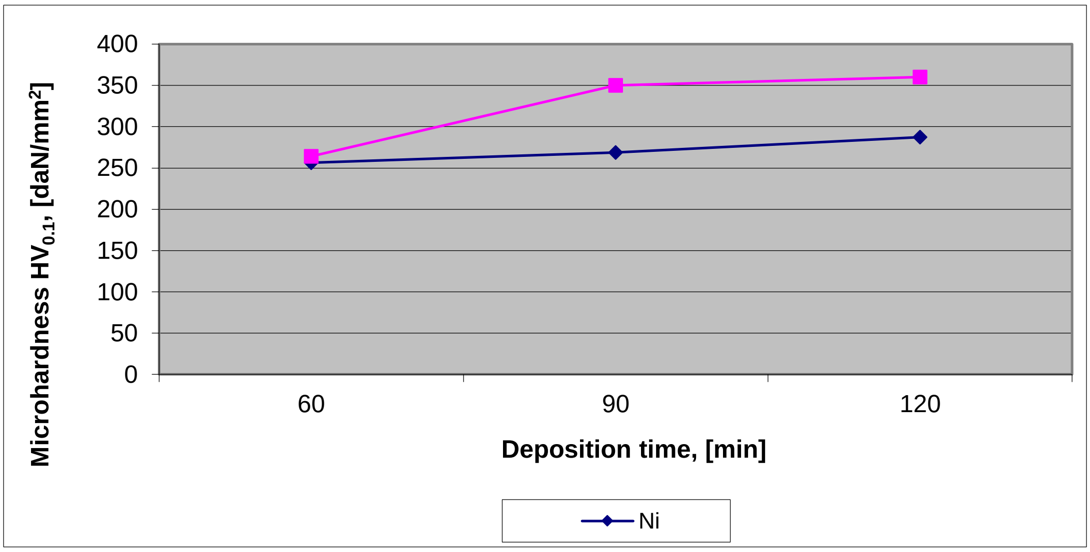

Fig. 8. *The influence of deposition time on microhardness of composite coatings electrochemically obtained in nickel matrix using as dispersed phases kaolin particles*

Fig. 9 shows the influence of deposition time on the microhardness of composite coatings electrochemically obtained in nickel matrix using as dispersed phases particles of kaolin and alumina compared with pure nickel coatings. It can be seen that the composite coatings obtained by means of using the electrochemical nickel matrix as dispersed phases particles of kaolin and alumina present higher microhardness compared to pure nickel coatings. However, due to the tendency to agglomerate larger particles of alumina compared to particles of kaolin, the higher the deposition times, the higher microhardness was obtained.

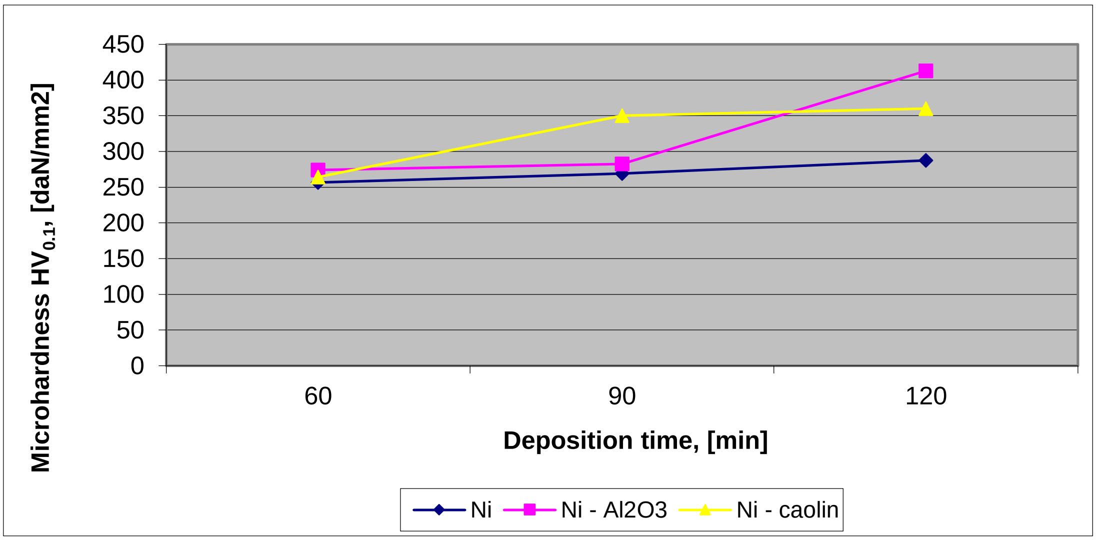

Fig. 9. *The influence of deposition time on microhardness of pure nickel coatings and on composites electrochemically produced in nickel matrix using as dispersed phases particles of kaolin and alumina*

Fig. 10 shows the effect of deposition time on thickness of the composite coating layer obtained electrochemically using the nickel matrix as a dispersed phase particle of kaolin and alumina as compared to pure nickel coatings. It can be seen that by increasing the deposition time, it increases the thickness of deposited layers, the influence of complementary phase particles gets stronger at longer deposition times.

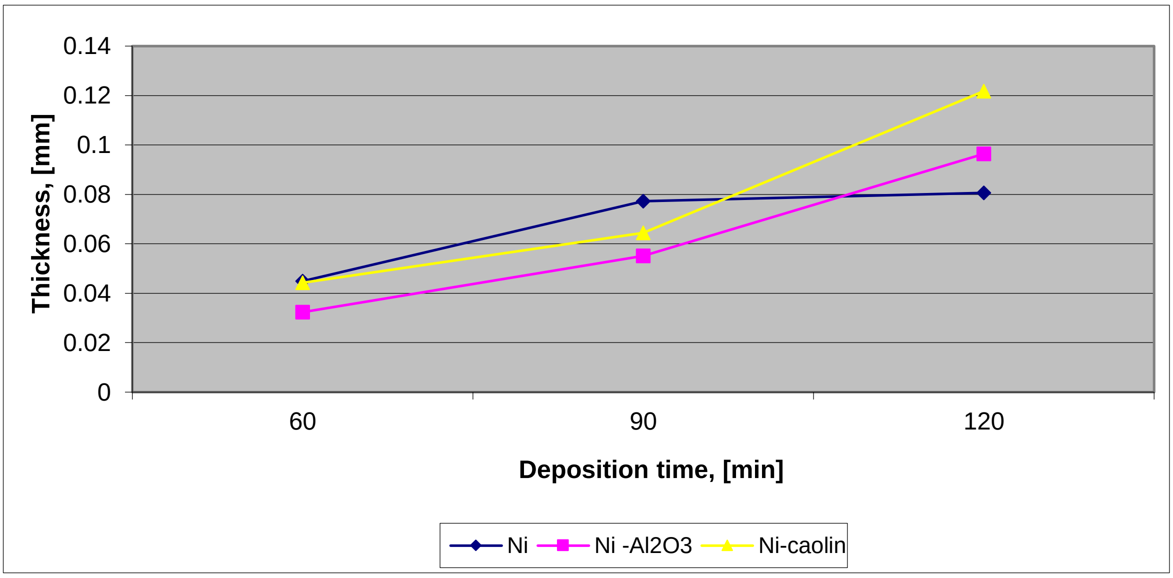

Fig. 10. *The influence of the deposition time on the thickness of the electrochemically obtained composite coating layer using the nickel matrix and as dispersed phases particles of kaolin and alumina as compared to pure nickel coatings*

4. Conclusions

By following the experimental researches, we can highlight the following conclusions:

❖ nickel coatings obtained are uniform, no crackings and a good adhesion to substrates of copper; the best coverage corresponded to the deposition time of 120 minutes;

❖ composite coatings electrochemically obtained in nickel matrix using as dispersed phases Al_2O_3 particles show good adhesion to the substrate, uniformity and compactness; the best inclusion particle dispersion was conducted at a deposition time of 90 minutes;

❖ composite coatings electrochemically obtained in nickel matrix using as dispersed phases kaolin clay particles are homogeneous, no crackings and a good adhesion to substrates of copper; the smaller size particles of kaolin has led to their inclusion in coating better than the Al_2O_3 dispersed phase coatings; the best matched sample has been obtained at a deposition time of 90 minutes;

❖ by increasing time of electrodeposition, the layer's coating thickness achieved increases from 0.0449 mm to 0.0806 mm to pure nickel coatings from 0.0323 mm to 0.0964 mm for ACE Ni - Al_2O_3, and from 0.443 mm to 0.1217 mm for ACE Ni - kaolin, current density and stirring rate being maintained constant;

❖ the layer thicknesses of the obtained coatings ACE Ni - kaolin are better than in the coatings of ACE results Ni - Al_2O_3; the smaller size of the kaolin particles has led to their improved inclusion in the deposited layer;

❖ composite coatings obtained by means of the electrochemical nickel matrix using as dispersed phases particles of kaolin and alumina present higher microhardnesses compared to pure nickel coatings, Such microhardness increases from 126.7 daN/mm^2 to copper support from 256.5 to 287.3 daN/mm^2 for pure nickel coatings, from 274.18 to 413 daN/mm^2 for ACE Ni - Al_2O_3 coatings, from 264 to 360 daN/mm^2 for ACE Ni-kaolin coatings, current density and stirring rate being maintained constant; this increase is due to the increasing tensions in the layer during deposition, and, on the other hand, good embedding of the dispersed particles in the deposited layer and its hardening.

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