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SURFACE ROUGHNESS AND TOPOGRAPHY OF Ni / MICRO-SiC LAYERS: INFLUENCE OF CURRENT DENSITY ON ELECTRODEPOSITION PROCESS

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ABSTRACT

The surface properties are directly responsible for the performance of engineering materials because most of failures such as friction, wear, corrosion and fatigue often take place on the material surface. Electrodeposition technique is of great interest for industrial usage because it produces functional and protective coatings with low cost and easy control. The present work has the purpose to evaluate roughness and to analyze surfaces of Ni / micro-SiC composite layers obtained at different parameters for electrodeposition. The layers were electroplated from a Watts bath with a suspension of SiC particles (mean diameter size of particles 30 µm) by adding 5g/L of particles. Electrodeposition took place at 40 °C and a current density of 2 A/dm^2 and 4 A/dm^2 for 1h. The suspension bath was stirred by a mechanical stirrer at a constant rotational speed of about 500 rpm. The roughness and topography of the layers were evaluated by atomic force microscopy (AFM) method.

KEYWORDS: electrodeposition, composite layers, atomic force microscopy, topography, roughness

1. Introduction

In the last years, composite layers have been developed quickly because they have many advantages in technology with a good resistance to aggressive working environments and fast processing systems. Embedding different particles into a metal matrix improve the material properties such as surface hardness, corrosion resistance, wear resistance, erosion – corrosion resistance etc. [1-3].

Layers of Ni / SiC composites have been studied because of their high wear resistance and good corrosion properties provided by the ceramic particles embedded in the nickel matrix [4-6].

Electrodeposition is one of the most used techniques for obtaining metallic and non-metallic composite layers. Between 1950-1960, the development of electrodeposited composite coatings expended progressively [7]. During the 1970s and 1980s, researches were focused on the need to produce coatings with enhanced mechanical, corrosion and tribological properties.

The advanced physical properties of composite layers quickly became clear and during the 1990s, new areas such as electrocatalysts and photoelectrocatalysts were considered [8].

To obtain composite layers a metal can be used as a matrix that could embed a dispersed phase with a good adhesion property on a substrate. As dispersed phases to obtain composite layers pure metals particles, ceramic particles or organic particles could be used.

The surface roughness has an important role on the mechanical as well as tribological properties of the composite layers.

The different properties such as wear, friction, corrosion, creep, fatigue failure, heat transmission, electrical conductivity, light reflection and lubricity etc. are affected by the surface roughness that has an important role for composite layers because affects their porosity. Due to higher porosity the corrosion resistance of the composite coating may be reduced. Hence, it is very essential to carry out investigations on the surface roughness and to optimize the bath process parameters to reduce the surface roughness at the preferred level. Atomic force microscopy (AFM) is a method to see a surface in its full, extremely versatile tool to investigate the three- dimensional morphology of surfaces. The method applies to hard and soft synthetic materials as well as biological structures (tissues, cells, biomolecules), irrespective of opaqueness or conductivity. The 3D object is not perceived in the usual way, that is, by line-of-sight, reflections or shadows. Given that the image is constructed from height numbers, one also can measure peak-to-valley distances, compute standard deviations of height, compile histograms of heights or slopes of hills. These metrics of topography can be directly relevant to the technological performance or biological function, whether in microelectronics (roughness of layers in multilayer deposition processes), tribology (friction and wear on hard disk read heads), polymer-drug coatings (surface contour area impacting drug release), intrabody medical devices (shape of surface in contact with cells, tissues), cellular membranes and surface components (phospholipid bilayer, protein receptors) and much more [10-12].

The paper intends to study the surface topography and roughness of Ni/micro-SiC composite layers compared with pure nickel layers. The samples were obtained at two values of the electrodeposition current densities because the the electrodeposition parameters influence the topography of the surfaces. The topography of the layers was evaluated by the atomic force microscopy method and the values of surface roughness were evaluated by histograms of the tested layers.

2. Experimental

Nickel and Ni / micro-SiC composite layers were electrochemically deposited from a Watts bath with the following composition: 0.90M $NiSO_4x6H_2O$; 0.21M $NiCl_2x6H_2O$; 0.48M H_3BO_3 and additive: SDS (sodium dodecyl sulphate) 0.4 g/L. The pH of the solution was maintained at 4. The suspension was prepared by adding SiC particles with a mean diameter size of 30 µm to the solution to give a concentration of 5 g/L in the nickel plating electrolyte. The electrodeposition took place in the bath at a temperature of 40 ºC and a current density of 2 A/dm^2 and 4 A/dm^2 for 60 minutes. The suspension bath was stirred by a mechanical stirrer at a constant rotational speed of about 500 rpm.

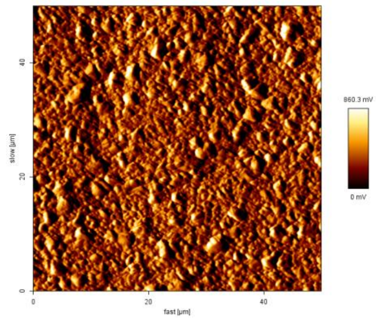
A NanoWizardII atomic force microscope (JPK Instruments, Germany) was used for surface analysis and for evaluating the roughness of the surfaces.

For AFM images, a silicon cantilever CSC37 A (Mikromasch, Estonia) with the following features was used: typical length, 250 µm; width, 35 µm; thickness, 2 µm; resonance frequency, 41 kHz; and nominal force/spring constant, 0.65 N/m. Each AFM image consists of 512 by 512 pixels. AFM images were performed by contact mode in air.

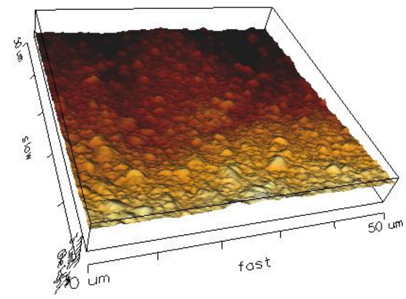
3. Results and discussion

The surface topography of pure nickel layers electrodeposited at a current density of 2A/dm^2 and 4A/dm^2 under atomic force microscope are presented in Figures 1 and 2.

From the AFM images of the tested surfaces for nickel obtained at a low current density, the electrodeposited nickel was observed to consist of pyramidal crystals with pronounced crystallographic polyhedral form with equigrannular structure. A bimodal grain structure of truncated pyramidal type can be observed. A cluster of fine grains is surrounded by relatively large grains. For nickel layers obtained at 4 A/dm^2 the surface topography was changed and it a homogeneous metallic structure with regular pyramidal shape was observed, characteristically for nickel crystallites [13-15].

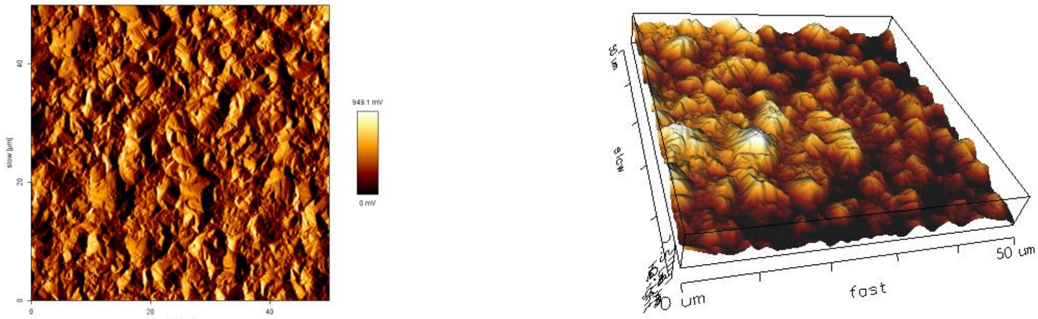


(a)



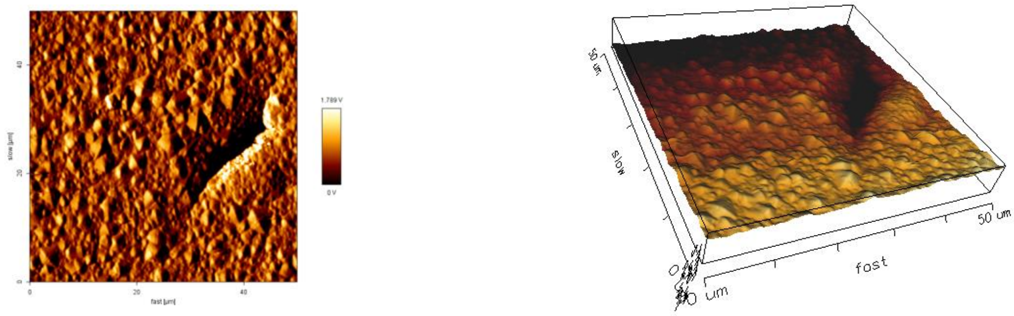
(b)

Fig. 1. *AFM images of pure nickel surface electrodeposited at 2A/dm^2: 2D (a) and 3D (b)*



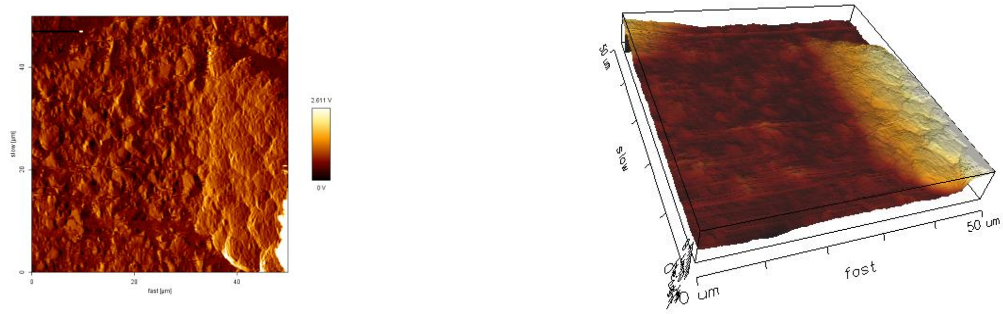
(a) *(b)*

Fig. 2. *AFM images of pure nickel surface electrodeposited at 4A/dm^2: 2D (a) and 3D (b)*



(a) *(b)*

Fig. 3. *AFM images of Ni / micro-SiC surface layers obtained at 2A/dm^2: 2D (a) and 3D (b)*



(a) *(b)*

Fig. 4. *AFM images of Ni / micro-SiC surface layers obtained at 4A/dm^2: 2D (a) and 3D (b)*

Figures 3 and 4 represent the 2D and 3D AFM images of Ni / micro-SiC composite layers obtained at 2A/dm^2 (Fig. 3) and 4 A/dm^2 (Fig. 4).

From the AFM images for Ni / micro-SiC composite layers it could be observed that the composite layers are smooth surfaces and it could be observed particles of SiC embedded into Ni matrix.

The particle size of SiC has an important role in the entrapment of SiC in the electrodeposited nickel coatings layers. The micro SiC particles could adsorbe more nickel ions and the Coulombic force is stronger than in case of nano SiC particles. On the other hand, the codeposition of pure nickel compared with SiC particles codeposited into nickel matrix is complex because a lot of factors act simultaneously. These factors include particle-particle interactions and the mobility of particles as a function of size and charge [16].

Comparing the topography of pure nickel layers with composite layers the pure nickel layers can be observed to have a rather regular surface and the composite coatings to have a nodular disturbed

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surface structure. Growing nickel crystals was perturbed by the current density used for electrodeposition and the incorporation of SiC macro particles. Fig. 3 and 4 show that the micron-sized particles are codeposited at the borders and the edges of the nickel crystallites and the embedding mechanism can be characterized as inter-crystalline [17, 18]. Fig. 5-8 show the histograms of the scanned surfaces of pure nickel layers and Ni / micro-SiC composite layers.

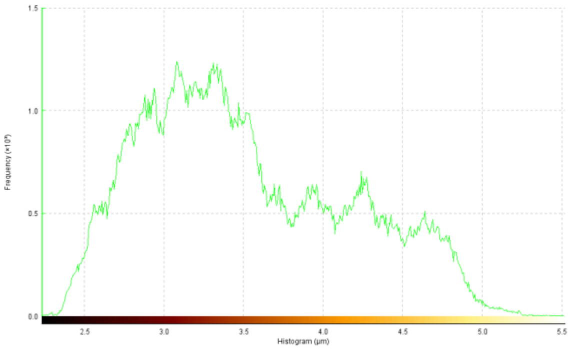

Fig. 5. *Histogram of the scanned surfaces for pure nickel layers obtained at 2A/dm²*

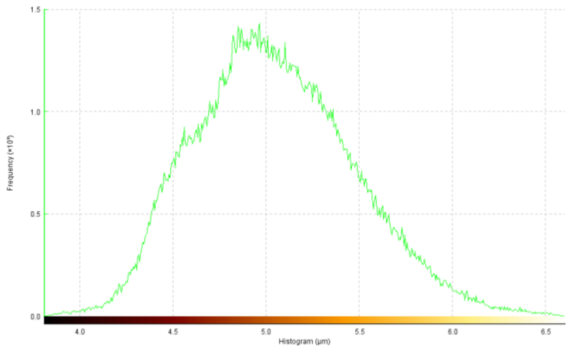

Fig. 6. *Histogram of the scanned surfaces for pure nickel layers obtained at 4A/dm²*

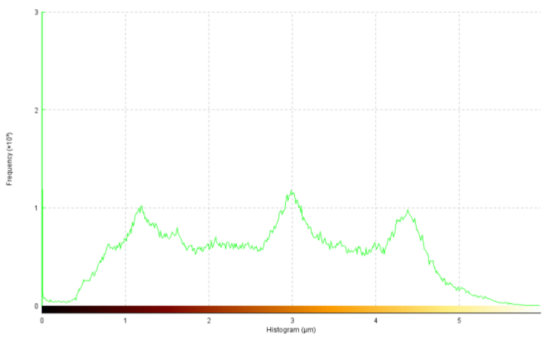

Fig. 7. *Histogram of the scanned surfaces for Ni / micro-SiC composite layers obtained at 2A/dm²*

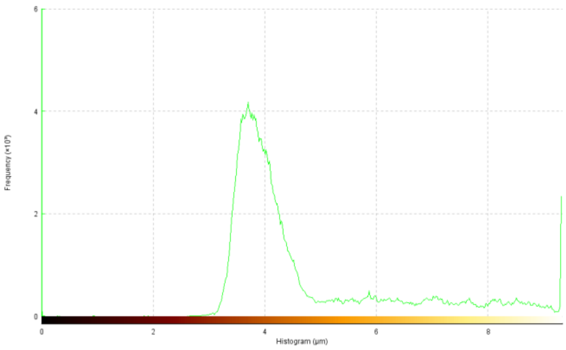

Fig. 8. *Histogram of the scanned surfaces for Ni / micro-SiC composite layers obtained at 4A/dm²*

The histograms presented in Figures 5-8 were used to estimate the surface roughness of pure nickel and Ni / micro-SiC composite layers.

The surface roughness has an influence on how a real system interacts with the environment and is quantified by the vertical deviations of a real surface from its ideal form. If these deviations are large, the surface is rough; if they are small the surface is smooth.

The roughness can be characterized by several parameters and functions, but the average value is by far the most commonly used. The average roughness is the area between the roughness profile and its mean line, or the integral of the absolute value of the roughness profile height over the evaluation length and is described in equation 1.

$$R_a = \frac{1}{L}\int_L^0 |Z(x)|\,dx \tag{1}$$

where Z(x) is the function that describes the surface profile analyzed in terms of height (Z) and position (x) of the sample over the evaluation length "L". Thus, the Ra is the arithmetic mean of the absolute values of the height of the surface profile Z(x) [19, 20].

Variations of the surface roughness for pure nickel and Ni / micro-SiC composite layers are shown in Fig. 9.

From the values of average roughness of the surfaces presented in Fig. 9 it could be observed that the roughness decreases for Ni / micro-SiC composite layers. The roughness is higher for pure nickel and composite layers obtained at a value of $4A/dm^2$ for electrodeposition current density as compared with the values of roughness obtained at a value of electrodeposition current density of $2A/dm^2$.

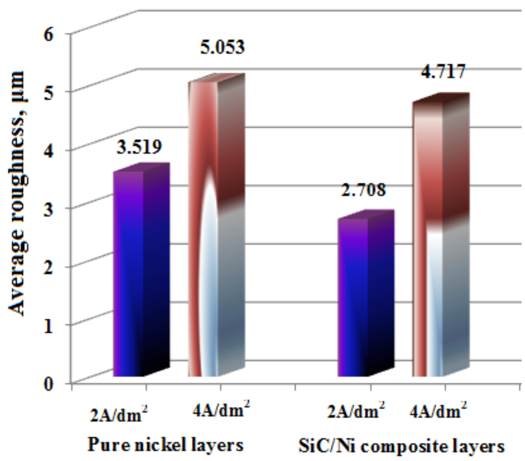

Fig. 9. *Variation of roughness surfaces of pure nickel and Ni / micro-SiC composite layers obtained at different electrodeposition current density*

The smaller values of the composite layers average roughness compared with the values of average roughness for pure nickel layers could be determined by the inhibition of nickel crystals growth induced by SiC micro particles.

The dispersed phase was uniformly distributed in the nickel matrix and the Ni / micro-SiC composite layers are more smoothed, uniform and compact surface than the pure nickel surface, that is indicated by the values of average surface roughness.

Conclusions

From the AFM images of the tested surfaces for nickel obtained at 2A/dm^2 the electrodeposited nickel was observed to consist of pyramidal crystals with pronounced crystallographic polyhedral form with equigrannular structure and for nickel layers obtained at 4 A/dm^2 a homogeneous metallic structure with regular pyramidal shape characteristical for nickel crystallites was observed.

From the AFM images of Ni / micro-SiC composite layers it could be observed that the surfaces are smooth and it could be observed the micro SiC particles embedded into Ni matrix. The particle size of SiC has an important role in the entrapment of SiC in the electrodeposited nickel coatings layers. The micro SiC particles could adsorb more nickel ions and the Coulombic force is high. Comparing the topography of the pure nickel layers with the composite layers it could be observed that the pure nickel layers have a rather regular surface and the composite coatings have a nodular disturbed surface structure. The growing of the nickel crystals was perturbed by the current density used for electrodeposition and the incorporation of SiC macro particles.

From the surface average roughness values roughness is observed to decrease for composite layers. The roughness is higher for the pure nickel and composite layers obtained at a value of 4A/dm^2 for electrodeposition current density compared with the values of roughness obtained at a value of electrodeposition current density of 2A/dm^2.

The smaller values of average roughness of the composite layers compared with the values of average roughness for pure nickel layers could be explained by the inhibition of nickel crystals growth induced by micro SiC particles.

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