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Nanocomposite Ni/diamond layers produced by the electrocrystallization method

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ABSTRACT

Purpose: The aim of this paper concerns the manufacturing of nanocomposite Ni/diamond surface layers deposited on S235JR carbon steel using electrocrystallization method and examinations of their structures and properties.

Design/methodology/approach: The performed research covers Ni/diamond nanocomposite layers and for comparison purposes also nickel layers produced by the electrocrystallization method. The disperse phase structure and whole produced layers were characterized by: X-ray diffraction (XRD), scanning electron microscope (SEM), optical microscopy. The realized research was performed to select the features of the produced layers such as microhardness, tribological properties and corrosion resistance.

Findings: The results of completed studies indicate on a compact structure and a good adhesion of produced layers with the steel substrate. The Ni/diamond nanocomposite layers have a higher hardness and wear resistance, as well as greater corrosion resistance tested in a corrosive environment as compared to Ni nanocrystalline layers.

Research limitations/implications: It is reasonable to continue further research on influences of different amount of nanodiament in nickel matrix and its impact on the tribological, corrosion, and thermal properties so produced nanocomposite layers.

Keywords: Ni/diamond; Electrodeposition; Nanodiamond; Composite layers

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MANUFACTURING AND PROCESSING

1. Introduction

The research on new materials is one of the main research directions related to the selection of appropriate materials for construction of products being particularly resistant to abrasive wear. These problems may concern the whole of the product's material or only a modification of the properties of surface layers of the given product. Due to the high potential of adjusting the performance of this type of composite materials, a large number of researchers from different countries have turned the attention to such type of materials [1-5]. The electrocrystallization based on electrochemical reduction processes plays an important role in the production of composite surface materials. Composite layers are basically formed of matrices with embedded fine particles of one or more other suitable materials. Depending on the desired properties, different matrices and dispersed phases are chosen. The research on composite layers with diamond disperse phases points to the beneficial effect of diamond particles on the tribological properties and hardness of composite materials [6-9].

The subject of research carried out within the framework of this paper are nanocomposite Ni/diamond surface layers produced by the electrocrystallization method and a comparison of their structures and selected properties with nanocrystalline Ni layers.

2. Experimental procedures

2.1. Materials and preparation methods

Nickel and Ni/diamond composite layers were deposited using electrocrystallization in Watts baths containing nickel(II) sulfate(VI), nickel(II) chloride, boric acid and organic compounds additives with diamond powder of nanometric dimensions as the dispersed phase (2 g/dm^3). For a good dispersion of the particles in the bath, ultrasonic bath mixing was applied to it before the process, whereas during the process the bath was mixed using a mechanical stirrer working at a speed of 200 rpm. The electrocrystallization process was carried out at 45°C within 45 minutes at a current density of 3 A/dm^2.

Carbon steel S235JR was used as the substrate material. The preparation of the substrate included sanding, degreasing with Vienna lime and pickling in sulfuric acid.

2.2. Analytical methods

The characteristics of the diamond dispersed phase was determined with X-ray diffraction using a diffractometer and a transmission electron microscope (Libra 120, ZEISS). The structure of the nickel and composite layers was analysed using X-ray diffraction (MiniFlex II, Rigaku), scanning electron microscopy (LEO 435VP, Zeiss), and optical microscopy (VHH 5000, KEYENCE).
To determine the distribution of the elements on the surface of the composite layers an EDS electron microprobe coupled with a scanning electron microscope were used. A surface roughness tester (Surftest SJ-210P, MITUTOYO) was used to examine the surface roughness of the substrate and the produced layers. The testing of the tribological properties of the materials was performed using a Calotest (apparatus of the IMP) [10,11]. The testing apparatus consisted of a sample placed in the holder and a ball (the counter-sample) propelled by a rotating roll and a motor. The rolling ball with a diameter of 30 mm made of bearing steel, produced a bowl-shaped trace on the surface of the sample. The depth of the wear was adopted as a criterion for the wear resistance of the examined material. The corrosion resistance of the layers was examined using a computer-controlled Atlas rig. Study was carried out in a three-electrode setting, with silver chloride electrode as the reference electrode, and a platinum electrode as the counter-electrode. Measurements were performed in 0.5 M sodium chloride solution at a temperature of approx. 28°C. The examination was carried out in the potential range from -400 to 400 mV. Scanning speed was set at 0.2 mV/s in the corrosion potential area and 0.4 mV/s in the anode area. Microhardness measurements were made on metallographic lateral microsections perpendicular to the layer's surface with the Vickers method at a load of 10 G using an INNOVATEST microhardness tester.

3. Results

Diamond powder was used to produce composite layers. A TEM image of the diamond nanoparticles is shown in Figure 1. The diameters of the diamond particles were varied and ranged from 5 to 20 nm. The X-ray diffraction of diamond powders shown in Figure 2 confirms the nanocrystalline size of the powder particles.

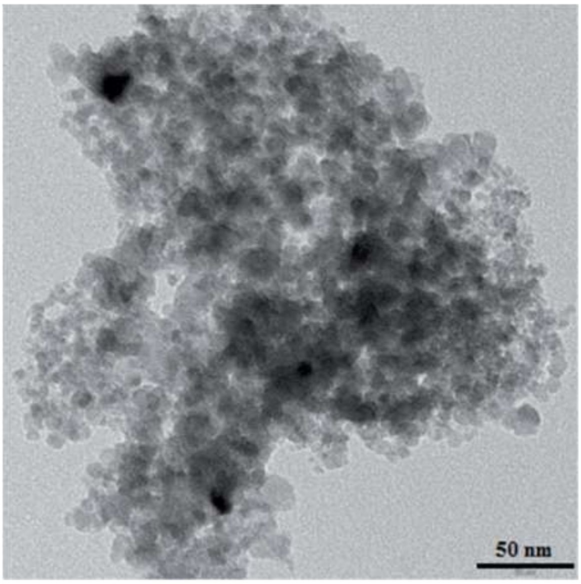

Fig. 1. TEM image of the diamond nanoparticles

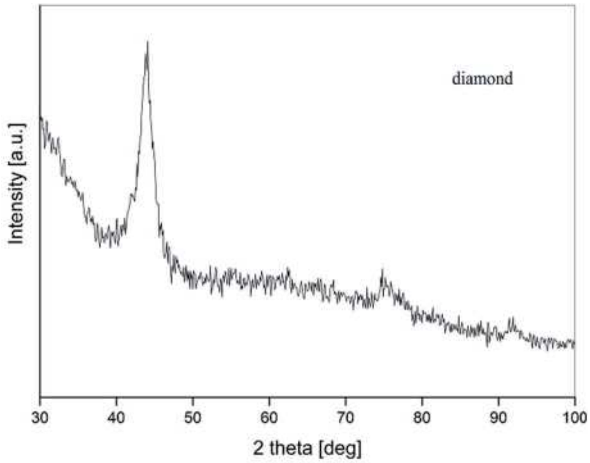

Fig. 2. X-ray diffraction pattern of used diamond nano-particles

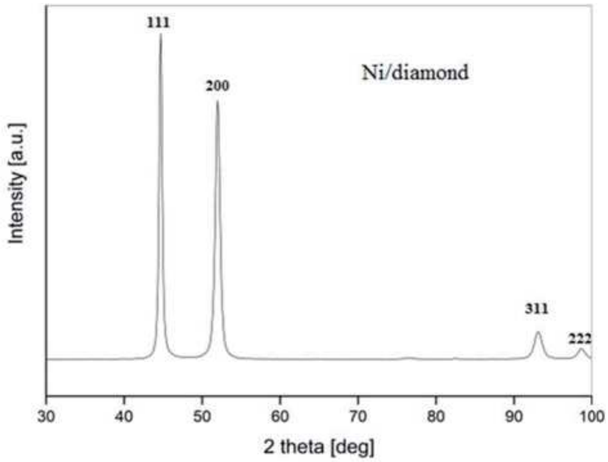

Fig. 3. X-ray diffractograms of Ni and Ni/diamond layers

The tests presented in this paper included Ni/diamond composite layers and nickel layers produced by means of the electrocrystallization method for comparative purposes. The results of the X-ray diffraction analysis of the produced composite layer are shown in Figure 3. The analysis of the graph demonstrates that the surface layer has a nano-crystalline structure as evidenced by the wider peaks in the diffraction pattern. Reflections originating from the diamond are not present in the diffraction pattern due to the nanometric size of the particles and too low their content in the material layer.

Images of the morphology and topography of the produced layers shown in Figure 4 indicate on the fact that the produced layers have smooth surfaces. A cross-section of the layers perpendicular to their surfaces is shown in Figure 5.

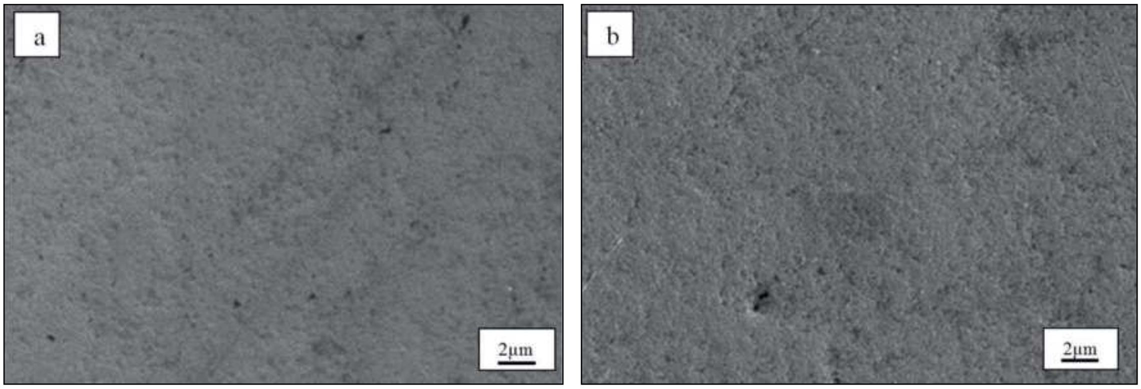

Fig. 4. Morphology and topography of the surface Ni (a) and Ni/diamond (b) layers

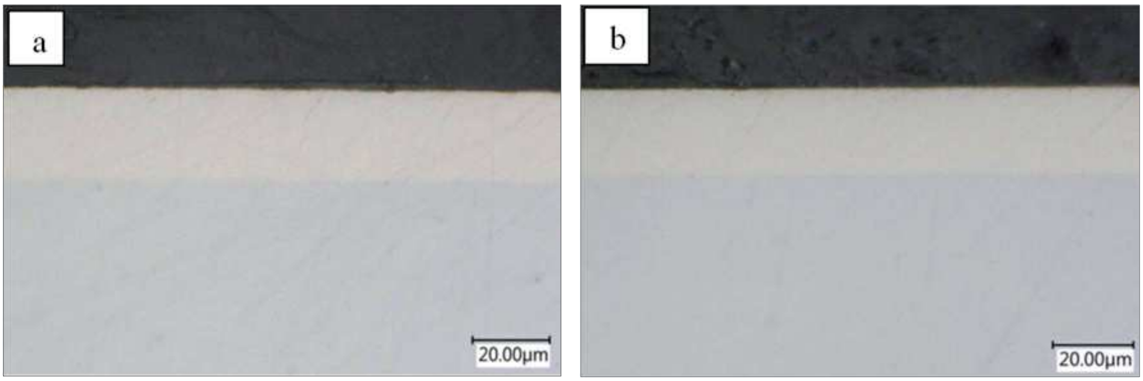

Fig. 5. Cross-sections of produced layers: a) Ni, b) Ni/diamond

The produced layers are characterized by compact structure, uniform thickness (approx. 20 µm), and a good adhesion to the substrate material. Due to the nanometric dimensions of the diamond particles, their identification in the layer was difficult. An energy-dispersive detector used to determine the distribution of carbon in the surface layer exhibited results shown in Figure 6.

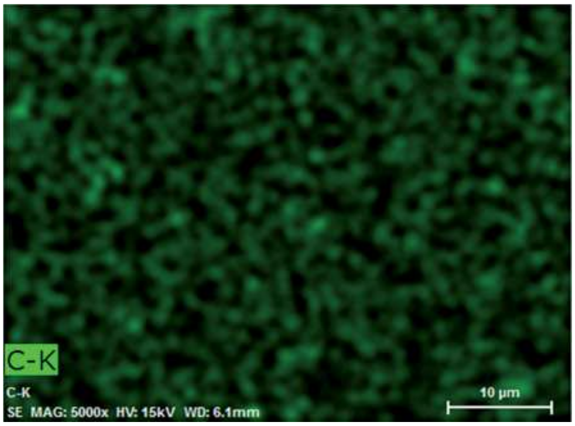

Fig. 6. EDS analysis of chemical distribution in Ni/diamond composite layer

The measurements of the surface roughness of the steel substrate with deposited Ni and Ni/diamond layers indicate that the surface roughness after the deposition of a Ni layer practically does not change. In contrast, the incorporation of diamond particles in the nickel layer increases the degree of surface development (Table 1). Results of the microhardness measurements presented in the table show that the hardness of nanocomposite Ni/diamond layers is greater than those of Ni layers.

Table 1.
Roughness and microhardness of substrate and produced layers

Materials	Ra [µm]	HV0.01
steel	0.10	170
Ni	0.11	416
Ni/diamond	0.17	453

The results of electrochemical corrosion tests performed on the substrate material and the tested Ni/diamond and Ni layers as the potentiodynamic curves are presented in Figure 7. Both nickel and composite layers produced by electrocrystallization processes provide good corrosion protection of the steel substrate.

The analysis of the polarization curves allowed to determining the corrosion current densities and corrosion potentials that are listed in Table 2. The composite layer showed a higher corrosion resistance as compared to the nickel layer in the same testing environment. Scanning electron microscopy was used to assess the damage of the layer material after the corrosion tests. Figures 8 and 9 shows the corrosive destruction of the nickel and composite layers. Uneven pickling of the surface layers and local pits reaching the substrate are visible.

Table 2.
Corrosion parameters of nickel and Ni/diamond layers and steel substrate in 0.5 M solutions of NaCl

Materials	E_{cor} [mV]	I_{cor} [µA/cm^2]
steel	-606	102
Ni	-170	4.3
Ni/diamond	-100	1.6

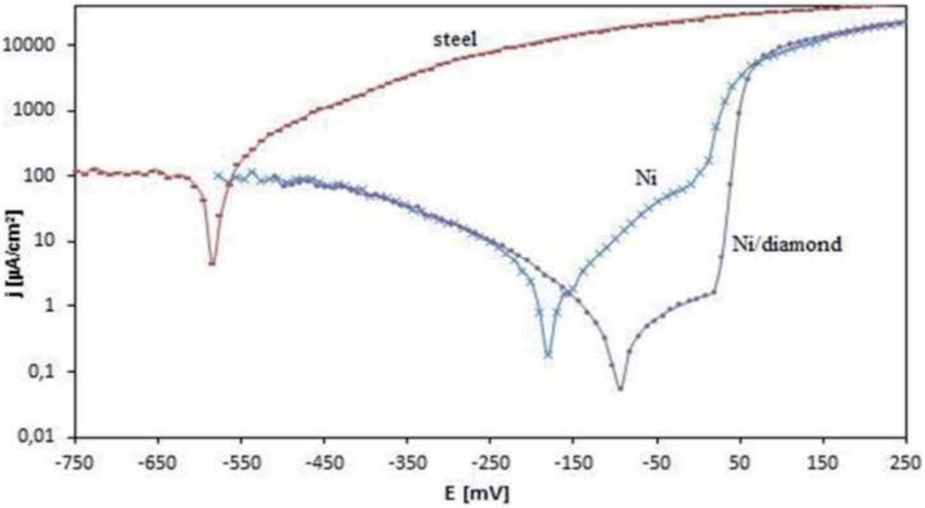

Fig. 7. The potentiodynamic polarization curves of Ni and Ni/diamond layers and steel substrate

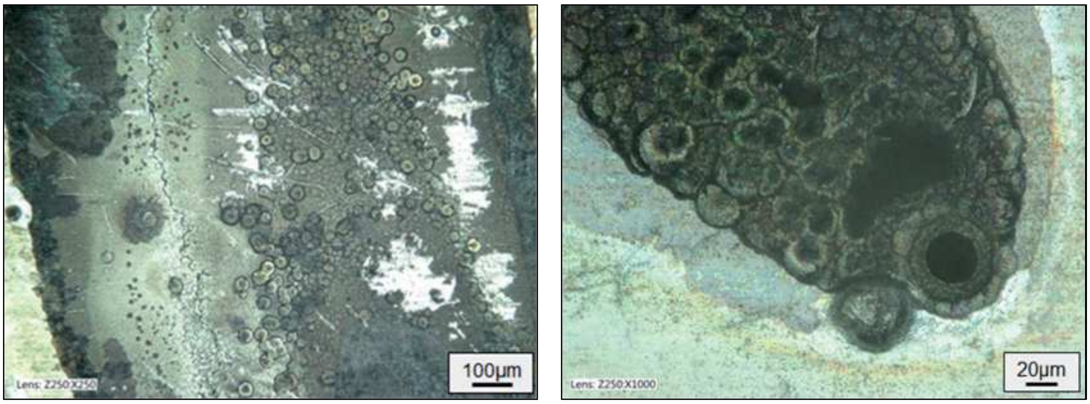

Fig. 8. Images of the surface of Ni layers after corrosion tests

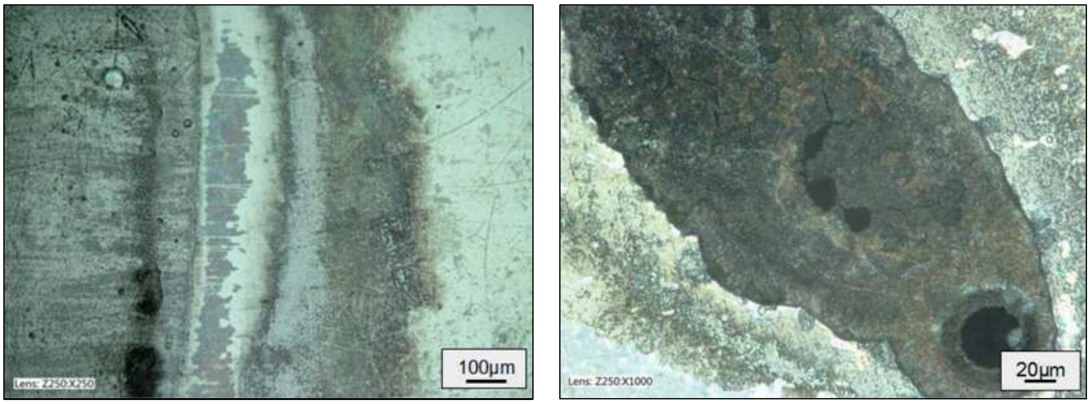

Fig. 9. Images of the surface of Ni/diamond layers after corrosion tests

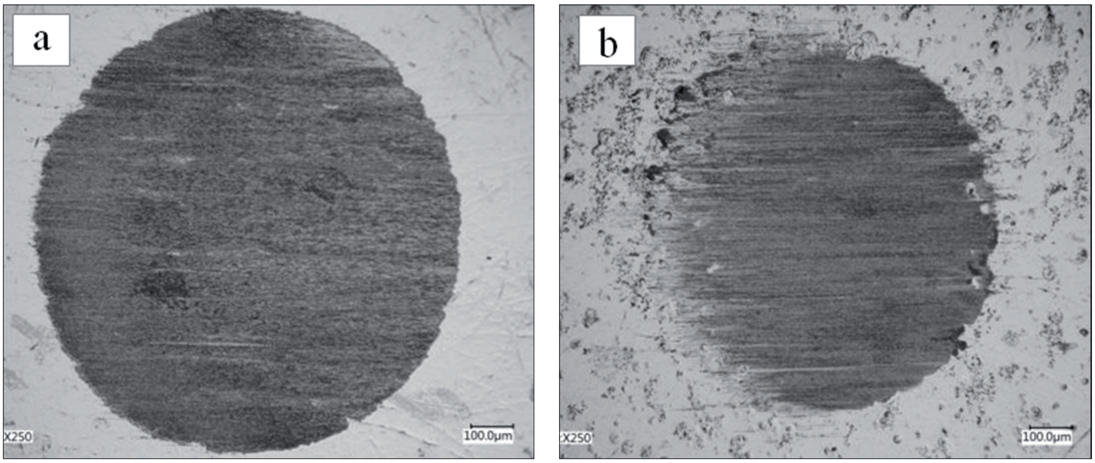

Fig. 10. Images of abrasion traces on layers surface after tribological tests on: a) Ni, b) Ni/diamond

Tests for abrasive wear resistance were carried out on the material in dry friction conditions at ambient temperature. As a criterion of wear resistance, the wear depth was calculated from the following formula:

$$h = R - \frac{1}{2}\sqrt{4R^2 - D^2} \tag{1}$$

where: h – depth of abrasion [µm], R – radius of steel ball [µm], D – diameter of abrasion [µm].

The parameters in the equation were determined based on geometric measurements of the images of wear traces made by the steel ball (Fig. 10). Abrasive wear of the layers materials is shown in Figures 11 and 12 shows the calculated wear depth of the material as a function of the duration of the test. The analysis of these curves shows that the nanocomposite Ni/diamond layer showed a greater resistance compared to the metal Ni layer, which is also confirmed by the wear traces shown in Figure 10. The difference in the size of the trace made during a tribological test is clearly visible.

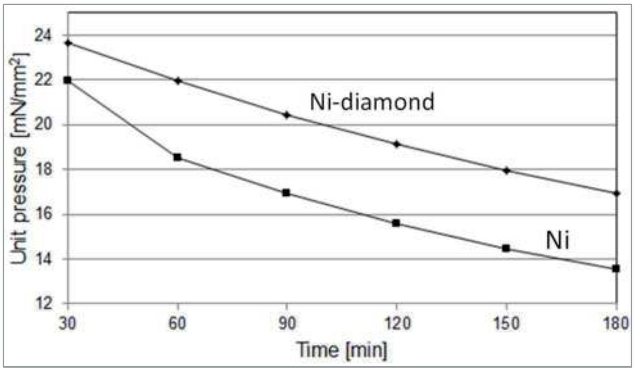

Fig. 11. Evolution of unit pressure during the tribological test of the investigated coatings

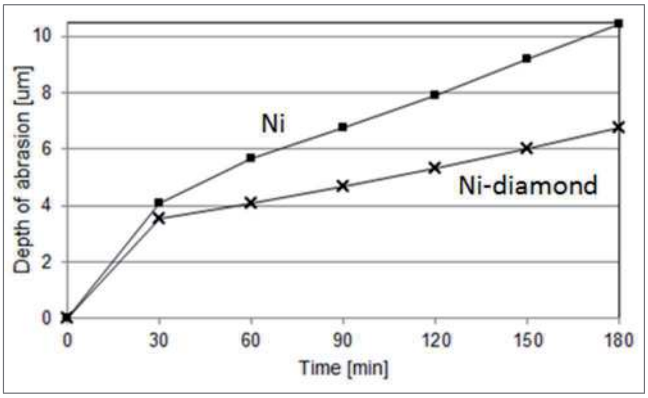

Fig. 12. Abrasive wear of investigated layers

4. Summary

The results of performed studies showed that the nickel layers and the Ni/diamond composite layers produced using the electrochemical reduction method are characterized by a good adhesion to the substrate material, compact structure and a smooth and uniform thickness over the entire coated surface. The deposition of nanodiamond in the nickel matrix resulted in an improvement of abrasive wear resistance and corrosion resistance in the testing environment when comparing to nickel layers. The test results indicate that it is expedient to develop research on the use of composite layers to improve the performance of various products.

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