Microstructure of the coating obtained by magnetron sputtering of a Ni-Cr-B4C composite target

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Abstract. This paper presents data on obtaining a composite coating by radio frequency (RF) magnetron sputtering of a Ni-Cr-B4C composite target in an inert gas (argon) environment. To make the target, Ni-Cr-B4C composite powder was applied to the copper base of the target by detonation gas-thermal spraying. The obtained targets served as a source of coating material during high-frequency magnetron sputtering. This method of coating production ensures the reproducibility of their properties, as well as the uniformity of coating thickness and good adhesion to various target backings.

The data of the study of the structure and morphology of the composite coating are presented. The resulting composite coating Ni-B/Cr3C with a thickness of 2 microns has a dense homogeneous structure with expressed textured polycrystallinity. The surface of the resulting coating is represented by nanoscale and homogeneous grains. There is no columnar crystal growth in the coating, which has a positive effect, as the columnar structure reduces the mechanical characteristics of the coatings due to faster oxygen diffusion along the grain boundaries. It is established that the combined use of the Ni-B and Cr7C3 binary phases in composite coatings leads to an increase in operational properties.

1. Introduction

Simple coatings have been widely used as protective coatings for many years. Taking into account the ever-increasing industrial requirements, such as high characteristics of corrosion resistance, hardness and wear resistance necessitates the development of new coatings. World recent experience has shown that multicomponent coatings obtained by synthesis from a combination of materials having different element composition have unique functional and protective properties [1, 2].

Nickel boride (NiB) has excellent mechanical properties and chemical resistance. The hardness of NiB coatings can reach 1100 HV; in addition, after heat treatment the hardness increases by several times [3]. The binary Cr-C carbide system is of particular interest because of its high hardness, toughness and chemical stability. The Cr-C system contains several crystalline phases with a complex structure, such as Cr5C2, Cr7C3 and Cr23C6. Crystalline Cr5C2 of these phases exhibits the highest hardness and therefore is the most preferred as a protective coating [4]. The coatings under consideration have been studied in terms of their composition, structure and mechanical properties [5-9]. Thus, it can be expected that the combined use of NiB and Cr5C2 binary phases in composite coatings will lead to improved performance properties.

There are various methods of surface coating, such as physical vapor deposition (PVD), chemical vapor deposition (CVD), thermal spraying, electroplating, and chemical coating [10-14]. Among these methods, physical vapor deposition by magnetron sputtering of coatings has become important
because of its specific characteristics such as uniformity, durability, high wear resistance and low cost. In addition, magnetron sputtering is one of the most suitable methods for industrial use \[15, 16\].

The present work is aimed at studying the microstructure and chemical composition of the coating obtained from the Ni-Cr-B4C composite target by magnetron sputtering.

2. Materials and methods

The Ni-B/Cr3C2 coating was applied by RF magnetron sputtering at 13.56 MHz on silicon and 65G steel target backings, the chemical composition of which is shown in Table 1. A 100-mm-diameter, 4-mm-thick, metal-ceramic-coated copper disk was used as the target. A composite Ni-Cr-B4C powder was applied on the copper target by detonation gas-thermal spraying. The metal-ceramic-coated disk was mounted on a water-cooled magnetron connected to an RF generator (13.56 MHz). In the RF mode, the target with the ceramic-metal coating was sprayed at a discharge power of 150 W for 15 min. The discharge voltage was set at 350 V and the discharge current was 1 A.

The vacuum chamber was evacuated to a pressure of no more than $2 \times 10^{-3}$ Pa, which was achieved using a turbomolecular pump. To remove residual contaminants, the surface of the target backings was cleaned with argon ions for 10 min at a pressure of 0.12 Pa and a voltage of 2.2 kV on the ion source.

The operating pressure in the chamber was 0.2±0.001 Pa. Pure argon (99.999% purity) was used as the atomizing gas, and the flow rate was 34 SCCM. The target was pre-sprayed for 1 minute to remove any surface contaminants. The distance from the target backings to the magnetron and ion source was the same, 60 mm. While coating, the target backing was not additionally heated.

The phase analysis was performed using X-ray diffraction (XRD) on an ARL X'TRA diffractometer (ThermoTechno) with Cu-Kα radiation ($\lambda = 0.1541744$ nm). The $\theta$-2$\theta$ scans were obtained in the range of 5-110° with a step of 0.05°. Phase identification and peak indexing were performed using the JCPDF database. The morphology of the surfaces and chips of the coatings were studied using a scanning electron microscope (TESCAN MIRA 3 LMU). SEM images were acquired at 5 kV acceleration voltages.

### Table 1. Chemical composition in % of material steel 65G.

|   | C   | Si   | Mn   | Ni   | S   | P   | Cr   | Cu   |
|---|-----|------|------|------|-----|-----|------|------|
|   | 0.62–0.7 | 0.17–0.37 | 0.9–1.2 | up to 0.25 | up to 0.035 | up to 0.035 | up to 0.25 | up to 0.2 |

3. Results and discussions

The diffractogram of the coating applied by magnetron sputtering from the Ni-Cr-B4C composite target is shown in Figure 1. The analysis of the coating diffraction pattern shows the presence of three main phases $\gamma$-Ni, Ni-B and Cr$_7$C$_3$, which corresponds to the results on the production of nickel boride and chromium carbide coatings in works \[17-19\].

The microstructure of the coating applied by magnetron sputtering from the Ni-Cr-B4C target composite is presented in figure 2. From the analysis of the cross-sectional image (figure 2a) we can see that the coatings have a dense homogeneous structure without columnar growth with expressed textured polycrystallinity. The coating thickness was about 2 μm.
The surface of the synthesized coating consists of nanosized and homogeneous grains, as shown in figure 2b. It should be noted that the columnar structure reduces the mechanical characteristics and oxidation resistance of the coatings due to faster oxygen diffusion along the grain boundaries [20].

The elemental composition of the coatings was determined by analyzing the spectra of characteristic X-rays using a spectrometer built into the scanning electron microscope. The spectra of the characteristic X-ray radiation and the elemental composition of the coatings are shown in Fig. 3.
Figure 3. EDS analysis of composite coating Ni-B/Cr$_7$C$_3$.

Analysis of the EDS spectra shows that Ni, Cr, B and C elements are present in the coating. This demonstrated that Cr$_7$O$_3$ particles were embedded in the Ni-B coating. It can be found that Ni content is the highest and B is the lowest. According to the authors of [21], element B is a light element, it can overlap the peak of Ni element, which leads to lower content of element B.

4. Summary
In this work the Ni-B/Cr$_7$C$_3$ composite coating was applied by HF magnetron sputtering of the metal-ceramic composite target Ni-Cr-B$_4$C in Ar gas environment. According to the X-ray diffraction data the presence of three main phases γ-Ni, Ni-B and Cr$_7$C$_3$ was found out. Electron microscopy analysis
showed that the deposited composite coating has a dense homogeneous structure with expressed textured polycrystallinity. From the EDS analysis of spectra it was found that Ni, Cr, B and C elements are present in the composite coating.

Thus, the obtained experimental data of the structure and morphology of Ni-B/Cr₃C₃ composite coating synthesized from the metal-ceramic composite target Ni-Cr-B-C.

Further research will focus on the tribological properties of the Ni-B/Cr₃C₃ composite coating.

5. References

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