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Study of electric spark coatings of nanocrystal structure

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Study of electric spark coatings of nanocrystal structure

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Abstract. The article presents the results of studies of new electrospark coatings having an amorphous and nanocrystalline structure. The results of metallographic and X-ray diffraction studies are analyzed. The microhardness and thickness of the coatings were studied. For the formation of a nanostructure, a plasma spraying method is used, in which materials are formed upon separate solidification of particles, most often disk-shaped. When the thickness of the sprayed particles is up to 10 μm, they quench from the liquid state at a cooling rate of 10^8 K / s. Nonequilibrium structures in sprayed particles with a nanocrystalline and amorphous structure have high mechanical properties. However, the use of plasma spraying in some cases is not advisable, due to the relatively high cost of equipment and materials. Therefore, this article will present the results of studies on the creation of amorphous and nanocrystalline coatings using electrospark processing (ESP), which is a cheaper way to obtain them.

1. Introduction

One of the most important directions in the development of modern metallurgy is the formation of materials in the nanocrystalline and amorphous states [1-3]. This scientific direction is implemented by powder metallurgy methods.

At present, significant difficulties have been identified associated with the formation of nanostructures in large volumes of a wide range of materials, for example, steels, nickel superalloys, and refractory metals. At the same time, the opinion is becoming more and more rooted that in many cases there is no need to form nanostructures in the entire volume of a workpiece or part from a structural material. Sometimes it can even worsen some of its properties. It is enough to form nanostructures with the required set of properties in the surface layer of a certain thickness, firmly bound to the base metal. One of these methods is plasma spraying, in which materials are formed during separate solidification of particles, often disk-shaped. With a thickness of the sprayed particles up to 10 μm, they quench from the liquid state at a cooling rate of 10^8 K/s. Nonequilibrium structures in sprayed particles with a nanocrystalline and amorphous structure have high mechanical properties [1].

However, the use of plasma spraying in some cases is not advisable, due to the relatively high cost of equipment and materials. Therefore, this article will present the results of studies on the creation of amorphous and nanocrystalline coatings using electrospark processing (ESP), which is a cheaper way to obtain them.

High wear resistance, microhardness and ductility, good performance properties of amorphous and nanocrystalline coatings are associated with the relaxation nature of their physical and mechanical properties. The complex of unique mechanical properties is largely determined by the structure of the coatings. Therefore, at the initial stage of research, metallographic and X-ray structural studies were carried out, the results of which will determine the method of measuring microhardness.

2. Research methodology

In the framework of this work, special electrodes were made from quickly hardened tapes. For the manufacture, alloys of grades 84КХСР, 2НСР, 82Н7ХСР, with an amorphous structure and a nanocrystalline alloy of the grade 5БДСР were used. Coatings were applied to samples of steel У9 GOST 1435-99, some of the samples (substrates) were preliminarily quenched by high-frequency currents (HFC). The surface roughness on which the coatings were applied was in the range of Ra 0.8–0.63. The coatings were applied by the installation for electric spark treatment of the brand UR-121 in the second and first energy modes of operation of the installation.

After that, transverse sections of coated samples were made. Sections were etched with a four percent solution of nitric acid in ethanol until the microstructure was clearly identified. Before etching, as well as after it, the thin sections were washed with water, dried with alcohol and filter paper.

Metallographic studies were performed using a Hitachi TM-1000 scanning electron microscope. The quality, thickness and structure of the resulting coatings were studied in the low-vacuum Standart Mode (standard conditions for industrial material samples) with a gap of 1.0 mm between the upper part of the test sample and the upper part of the shaft under the table with the sample. Sixteen samples were studied, of which eight were subjected to hardening of the high-frequency current. Each sample was coated with one of the experimental electrodes.

The microstructure of the coatings was investigated by taking diffraction patterns on a UNISANTIS XMD-300 diffractometer according to the Debye-Scherrer X-ray optical scheme. Beam size 0.2x6 mm. X-ray diffraction patterns were recorded in copper monochromatic radiation (1.54 A). The survey was carried out in the range of angles $2\theta=20°–100°$. The indicated part of the spectrum makes it possible to observe a halo characterizing the amorphous state of the studied coating.

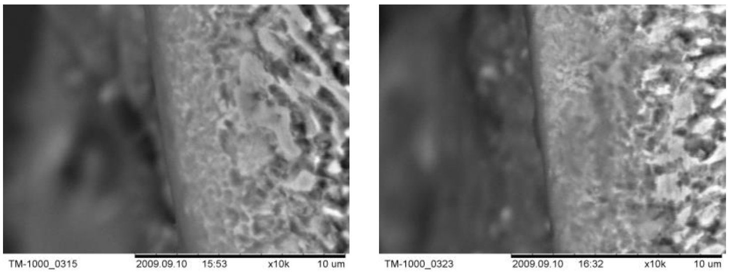
The microhardness of the coatings was measured on a PMT-3M instrument at a load of 0.49 N (50 g), taking into account the requirements of GOST 9450-76, at a length of at least 15 mm.

3. Research results and discussion

Metallographic studies of the zone of connection of the samples showed the absence of defects, pores, and cracks in the zone of connection of the coating with the base metal. All applied coatings have a clearly defined interface between the coating and the base metal. At the entrance of the studies, it was not possible to identify the layered structure characteristic of most electrospark coatings, which is obtained by treating steel substrates with steel electrodes. The substructure of the coatings under study is similar to the substructure of the plasma coatings described in the source [1], but the coatings obtained do not have a pronounced layered structure characteristic of plasma coatings. Based on the results of metallographic studies, we can conclude that the coating retained its original extremely thermally unstable structure characteristic of electrode materials [3]. However, in the transition zone of the coating and substrate there are crystalline inclusions formed by mixing the electrode material and the substrate. Studies of the coating showed that after processing the same area in two passes (first in rough and then in soft modes), microcracks and traces of chipping are observed on its surface. Therefore, an increase in the specific processing time will lead to the destruction of the coating [4, 5]. Figure 1 shows snapshots of the coatings. The results of measurements of the thickness of the coatings are presented in table 1.

Table 1. The thickness of the coatings obtained by electrospark processing

The coating thickness of the alloy 84КХСР, μm	The coating thickness of the alloy 5БДСР, μm	The coating thickness of the alloy 2НСР, μm	The coating thickness of the alloy 82Н7ХСР, μm
22	23	20	25



a b

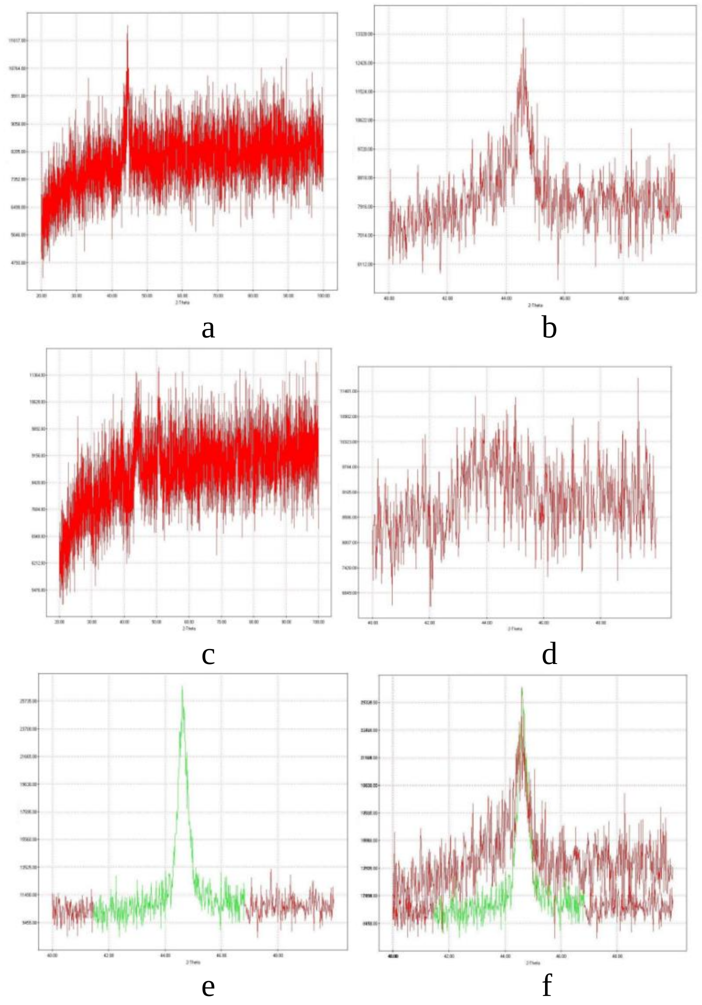
Figure 1. The microstructure of the electrospark coating, × 10000: a) a coating formed by an electrode made of 84КХСР alloy; b) a coating formed by an electrode of alloy 5БДСР.

Analyzing the results of measuring the thickness of the coatings given above, we can conclude that it depends mainly on the energy treatment conditions. In the course of the studies, it was not possible to identify a significant effect of the substrate heat treatment on the thickness of the deposited amorphous and nanocrystalline coatings.

In order to identify the microstructure of the coatings, they were studied by X-ray radiation. It is known that the main parameters of X-ray reflection are the maximum value of the intensity, its position, integral width and integral intensity. The position of the halo observed upon diffraction from the amorphous phase is determined by the characteristic average distance between atoms in the alloy, and the width at half height characterizes how strictly the distance between atoms in the amorphous phase is maintained. New narrow reflections appearing in the spectrum of the analyte correspond to the resulting crystalline phases, if any. The position of the new reflections characterizes the type and periods of the crystal lattice. Reflection from the crystalline phase is distinguished by a substantially smaller width at half height [6]. Figure 2 shows the diffraction patterns of some studied coatings and substrates. For parts of the spectrum where narrow reflections were observed characterizing the crystalline structure of the substance, the diffraction patterns were recorded in the range of angles $2\theta=40°–50°$. When studying sections of diffractograms with narrow spectra, several assumptions were made explaining the reason for the appearance of these peaks in a coating with an amorphous structure. The first assumption is associated with metallurgical processes occurring during electric spark processing in a microvolume, as a result of which mixing of the electrode materials occurs, and crystals of the substrate material appear in the transition zone of the coating. The second assumption is associated with the possible partial crystallization of the coating, which occurs due to the inability to provide the cooling rate necessary for amorphization. The third assumption is that all coatings are always and everywhere amorphous, but the interference maximum of the substrate breaks through it, where it is worse and where it is better. To verify these assumptions, we took the diffraction patterns of untreated sections of the substrates.

Diffraction patterns of the substrates were superimposed on the diffraction patterns of the coatings and the coincidence of the angles of narrow reflections was obtained (Figure 2f). This fact refutes the second assumption about the possible partial crystallization of coatings due to the low cooling rate, because crystallization of the same coating based on 82Н7ХСР alloy would lead to the appearance of the main crystallizing phase Ni (Fe, Co) and additional $(Fe, Ni)_3B$, and crystallization of the coating based on the 2НСР alloy - Fe (Ni, Co) phases, which we did not observe in more than one diffraction pattern.

The coincidence of the angles of narrow reflections confirms the assumption that there is a substrate material in the coatings of crystals, the appearance of which is associated with mixing of the electrode materials. The third assumption that reflections from the crystalline phase is nothing but the interference maxima of the substrate, which break through the coatings, is also not unreasonable. A proof of this assumption is the fact that the intensity of narrow reflections decreases, and for the 82Н7ХСР alloy their complete disappearance, with an increase in the thickness of the coatings.



a b

c d

e f

Figure 2. Diffraction patterns of an electrospark coating: a) a diffraction pattern of a coating based on 2HCP alloy in the range of angles 2θ=20°–100°; b) a diffraction pattern of a coating based on 2HCP alloy in the range of angles 2θ=40°–50°; c) a diffraction pattern of a coating based on 82H7XCP alloy in the range of angles 2θ=20°–100°; d) a diffraction pattern of a coating based on 82H7XCP alloy in the range of angles 2θ=40°–50°; e) the diffraction pattern of the substrate in the range of angles 2θ=40°–50°; f) diffraction pattern of the substrate and coatings based on 2HCP alloy.

From the above it can be concluded that the coating as a whole has an amorphous microstructure. However, at some sites in the amorphous matrix there are nanosized crystalline inclusions.

The microhardness measurements of coatings formed using experimental electrodes are shown in Table 2.

Table 2. Microhardness of electrospark coatings

Grade of electrode material	Microhardness at a load of 50 g, MPa
84КХСР	8005
5БДСР	10147
2НСР	7279
82Н7ХСР	5390

4. Conclusions

1. The use of quickly hardened alloys of the grades: 5БДСР, 84КХСР, 2НСР, 82Н7ХСР as electrodes for electrospark processing allows one to obtain coatings with an amorphous and nanocrystalline structure that have high microhardness.

2. The number of crystalline inclusions in the microstructure of the resulting coatings depends on their thickness and on the ESP modes.

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