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DEPOSITION OF TEXTURED CONDENSATES OF TUNGSTEN BY HYDROGEN REDUCTION OF ITS HEXAFLUORIDE

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In this work, a study of textured tungsten coatings deposited by the hydrogen reduction of tungsten hexafluoride was carried out. The formation of condensate is carried out by the natural selection of columnar grains growing perpendicular to the surface of deposition. Depending on the oversaturation of the gas phase above the growing solid one, some planes of the pyramids are subjected to a predominant growth rate, which leads to the formation of a texture. In the range of parameters studied, (100) and (111) tungsten textures are formed. The measured surface roughness is within the 5 class.

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INTRODUCTION

Emitter devices have become widely used in special-purpose electronic equipment, space communications, navigation, radar, and medicine. They are subject to increasingly stringent requirements regarding degree of efficiency, reliability, durability, and emission current density.

Recently, cathodes with field emission have been in demand as electron sources. Their advantage is the absence of a cathode incandescent source, high field emission current density, steepness of the current-voltage characteristic, non-inertia, and insensitivity to radiation. The use of field-emission cathodes opens up the possibility of improving the operational parameters of a new class of electro-vacuum devices with almost instant readiness. An important feature of such cathodes is the possibility of lowering the "cathode-anode" operating voltage. This is achieved by creating a rough cathode surface.

In this work, studies of the formation of the textured tungsten surface in the process of vapor-phase chemical deposition of tungsten by hydrogen reduction of its hexafluoride are carried out, and morphological features of the formation of deposited coatings are considered.

EXPERIMENTAL

On the vapor-phase deposition. The deposition of coatings from tungsten was carried out in a flow-type reactor with a horizontal arrangement. Hydrogen (H_2) and tungsten hexafluoride (WF_6) were launched into the reaction chamber and entered into a chemical reaction on the heated surface of a substrate placed inside the reaction chamber. The solid-phase reaction product, i.e., tungsten, forms a growing layer of condensate on the surface of the substrate, and the gaseous products formed as a result of the reaction are removed from the chamber, frozen out, and neutralized [1].

The deposition of tungsten was carried out in the diffusion region to control the rate of condensate deposition. The ratio of precursors in the reaction volume affects the saturation of the gas phase over the formed solid one and determines the type of pole figures, and the settling time determines the completeness of its formation due to natural selection [2, 3].

The substrates were used in the form of a cylinder with an outer diameter of 22 mm, an inner diameter of 16 mm, and a length of 100 mm, and were made of graphite and transition metals. The substrates were heated by the ВЧГ-25/044 high-frequency generator up to a temperature of 500 °C. The ratio of tungsten hexafluoride (WF_6) was 30 g per 30 l of hydrogen (H_2) and 60 g of WF_6 per 60 l of H_2. The settling time is one hour.

Research techniques. The coating morphology was studied using the PEMA-200 scanning electron microscope. When a narrow electron beam (probe) of a microscope interacts with the surface of a studied sample, the following types of signals arise: secondary electrons, reflected electrons, Auger electrons, characteristic X-rays, and photons of various energies.

The scanning electron microscope uses the signals created by secondary and reflected electrons as they change with the surface topography changes as the electron beam scans over the sample. Secondary electron emission occurs in the vicinity of the probe impact region, which makes it possible to obtain images with a relatively high resolution.

X-ray studies of the deposited tungsten coating were carried out on a ДРОН4-07 X-ray diffractometer. Full-profile diagrams were recorded using the usual Bragg-Brentano X-ray optical scheme in CuK_α radiation, using a selectively absorbing nickel filter and a pair of Soller slits. The diffracted radiation was recorded by a counter with a scintillation detector. According to the texture method of inverse pole figures (IPFs) [4], using the integral intensities of reflections from the coatings, the corresponding pole densities were calculated and the IPFs were constructed. The basic data of the IPF method were preliminarily prepared relating to studying the texture of tungsten.

The surface roughness of the tungsten coating was also studied. The TR200 indicator was used. The measurements were carried out on a flat sample 22 mm in diameter and 3 mm thick with a graphite substrate.

RESULTS AND DISCUSSION

On cylinders made of graphite, copper, molybdenum, and stainless steel, the tungsten coatings have been made by vapor phase deposition. Fig. 1 shows a tungsten-coated sample on a graphite substrate. The deposition process was carried out at a temperature of 500 °C. The consumption of WF_6 hexafluoride was 60 g/h, and hydrogen was 60 l/h.

Fig. 1. A sample of tungsten-coated cathode on the graphite substrate

Fig. 2 shows the morphology of the cathode surface on the graphite substrate (see Fig. 1). The figure shows pyramidal grain growth perpendicular to the deposition surface.

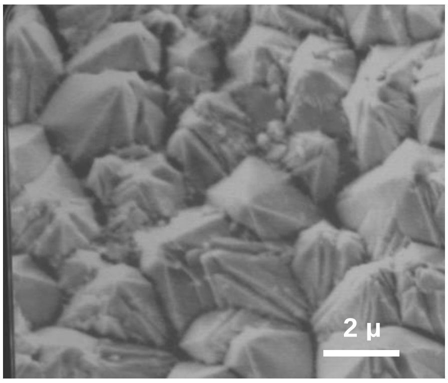

Fig. 2. Morphology of tungsten coating on graphite

The three-dimensionality of the image (see Fig. 2) was achieved due to the large depth of focus in the scanning electron microscope (2–3 times higher than in the optical microscope) as well as the effect of relief contrast shades in secondary electrons.

X-ray analysis of all samples showed that the coatings contain only crystalline tungsten with lattice periods $a = 0.3164…0.3168$ nm. Other phases are completely absent. Fig. 3 shows the angular X-ray diagram of the coating on the graphite substrate.

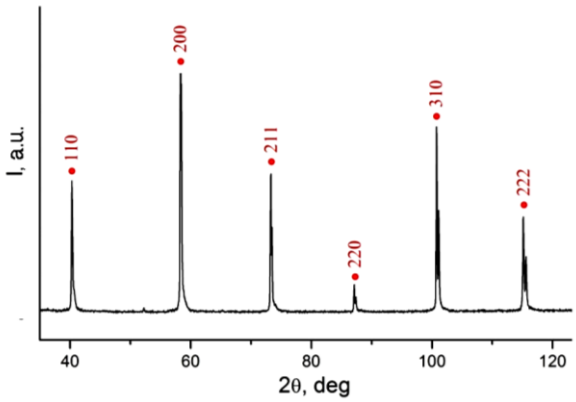

Fig. 3. X-ray CuK_α diagram of a tungsten coating on the graphite substrate

A clearly expressed crystallographic texture was revealed in the coatings. On graphite substrates, the tungsten coating was formed with (100) and (111) textures under different deposition conditions. Examples of pole figures are shown in Fig. 4.

The values of pole density in the texture maxima of all obtained coatings were found to be in the range of 7.2…9.7 (for a textureless sample, all the pole densities, by definition, are 1.0). Thus, the coatings have a high degree of texture.

Table shows the surface roughness values of the coating, measured on a sample with a flat graphite substrate. The roughness was measured in four orthogonal directions, with relatively small steps on the basic length.

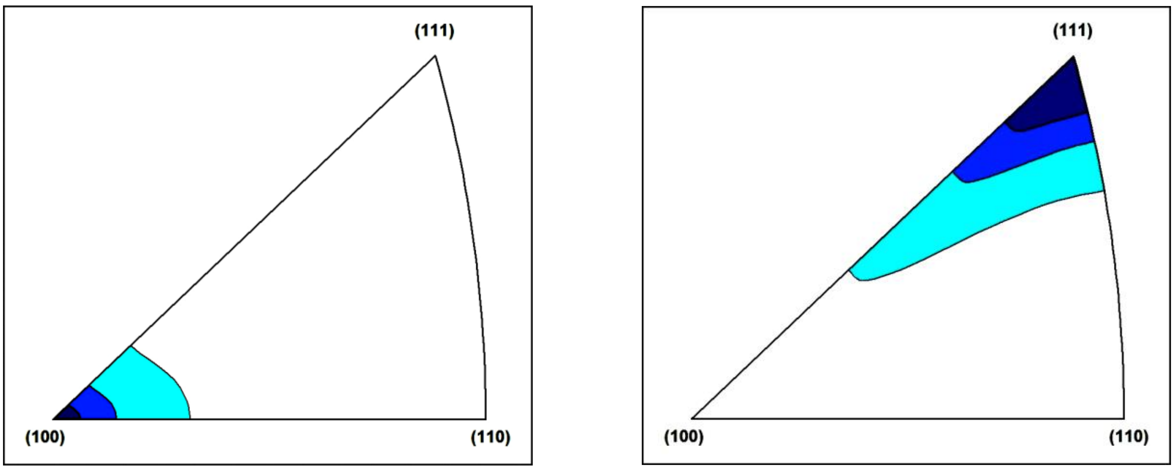

Fig. 4. Typical IPFs of the tungsten coatings: left is texture (100), 30 g of WF_6 on 30 l of H_2; right is texture (111), 60 g of WF_6 on 60 l of H_2. T = 500 °C, the deposition time 1 h

Roughness values of tungsten coating of a sample on graphite substrate

No. meas.	R_a, μm	R_z, μm
1	3.145	16.64
2	3.261	17.59
3	3.068	18.12
4	2.973	16.61

In Table, the values of the roughness of the coatings correspond to the fifth class for the sample obtained with a graphite substrate in the conditions of 30 g of WF_6 on 30 l of H_2, T = 500 °C.

A test of the tungsten-coated cathode was carried out with a pulsed voltage in the range of 350…450 kV with a pulse length of 70…75 ns at a chamber pressure of (3…6)$\cdot 10^{-6}$ Torr. The registered field emission current was 3…5 kA. After testing, no damage or delamination of the coating was found on the cathode surface.

CONCLUSIONS

Coatings of crystalline tungsten have been obtained by deposition from the gas phase on the graphite cathode. No other phases were discovered. Crystallographic textures (100) and (111) were revealed in the coatings, depending on the parameters of the coating deposition process. The obtained values of the field-emission current indicate the possibility of a decrease in the cathode-anode potential and an increase in the emission current due to the creation of a coating surface with high roughness.

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ОСАДЖЕННЯ ТЕКСТУРОВАНИХ КОНДЕНСАТІВ ВОЛЬФРАМУ ВОДНЕВИМ ВІДНОВЛЕННЯМ ЙОГО ГЕКСАФТОРИДУ

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Виконано дослідження щодо текстурованих вольфрамових покриттів, осаджених водневим відновленням гексафториду вольфраму. Формування конденсату здійснюється при природному відборі стовпчастих зерен у їх зростанні перпендикулярно поверхні осадження. Деякі грані пірамід у залежності від пересичення газової фази над зростаючою твердою отримують переважну швидкість зростання, що призводить до формування текстури. Істотне значення має пересичення газової фази над твердою, конденсованою. У діапазоні досліджених параметрів сформовані текстури (100) і (111) вольфраму. Виміряна шорсткість поверхні знаходиться у межах 5 класу.