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THE SURFACE MICROSTRUCTURE OF A TUNGSTEN SINGLE CRYSTAL FOLLOWING ELECTRICAL DISCHARGE MACHINING

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Electrical discharge machining is a widely used method in the production of experimental samples of metallic materials within the context of manufacturing techniques. The surface condition of the samples is usually of great importance for research. The authors studied the influence of the electrical discharge cutting process on the surface condition of samples produced from a tungsten single crystal. The metal was processed by two different machines. The surface microstructure and microhardness of the obtained samples were analyzed, also X-ray diffraction study was carried out. The surface of the samples resembles paving stones with blocks of $l \approx 100…400$ μm in size. Cracks dividing the blocks are appreciated to be of $d \sim 200$ μm in depth. The microhardness varies within the range of $H_\mu \approx 3600…4500$ MPa depending on the surface condition. Examination of residual stress revealed the presence of tensile stress at the surface, with a magnitude of $\sigma_\varphi = 765$ MPa. Analysis of experimental data indicates that the surface condition of tungsten samples is determined by the technology of the initial ingot manufacturing and the features of the electrical discharge machining process.

INTRODUCTION

The study and use of metallic materials requires certain methods of manufacturing (e.g., cutting) samples from them. In a number of cases, special attention should be paid to the surface condition of these samples, in particular when cutting samples from refractory and relatively hard single crystals, such as tungsten, which are promising for various applications [1, 2]. Among the various methods of manufacturing such samples, the method of electric discharge machining is widely used. It allows to produce samples of arbitrary shape with high accuracy. This method was used in [3] to manufacture experimental samples from a tungsten single crystal. The authors carefully studied the influence of technological parameters of the cutting process on the characteristics of surface damage obtained in single-crystal samples. However, it is of interest to study the picture of such damage in more detail using methods such as X-ray diffraction and electron microscopy. These considerations stimulated the conduct of this work.

MATERIALS AND INVESTIGATION METHODS

The raw material for obtaining samples was a flat single crystal tungsten ingot, manufactured at the E.O. Paton Electric Welding Institute using plasma arc melting using additive technology [1, 2]. To cut the samples, the electric discharge cutting machine BP-95d (Poland) was used where a brass wire with a diameter of $d = 0.25$ mm is used as the electrode tool. The working medium is distilled water, the jets of which are directed from above and below into the cutting zone in a sample and provide its cooling. This machine provided cutting speed $\upsilon = 5.3 \cdot 10^{-8}$ $m^2 \cdot s^{-1}$ for tungsten. For comparison, electric discharge cutting machine EFH43B (PRC) with a molybdenum wire of $d = 0.18$ mm as the electrode tool and a special emulsion as the working medium. This device provided cutting speed $\upsilon = 20.0 \cdot 10^{-8}$ $m^2 \cdot s^{-1}$ for tungsten. Three samples of tungsten single crystal were manufactured for the study: #1, cut perpendicular to the single crystal growth axis and #2, cut along the axis, these two were cut with BP-95d machine, while the third, #3, was cut perpendicular to the single crystal growth axis with EFH43B machine.

The effect of annealing at a temperature $T = 1000$ °C for 1 h in a vacuum $P \geq 9 \cdot 10^{-5}$ Torr on the properties of the samples was also studied.

Elemental composition of the samples was determined using Amptek EXP-2 XRF Experimenter's Kit EDX spectrometer. XRD analysis was performed using DRON-UM1 X-ray diffractometer in a filtered copper Cu-Kα radiation ($\lambda = 1.54187$ Å). Residual stress measurement was carried out using the $\sin^2\psi$ method. For single-crystal and highly textured samples, the selection of certain angles ψ_i is carried out by calculating the angles between the axis of the single crystal (texture) and reflections in the precision region [4]. To calculate residual stresses, the following values of Poisson's ratio and Young's modulus for polycrystalline tungsten were found in the literature [5]: $\nu = 0.28$ and E = 411 GPa.

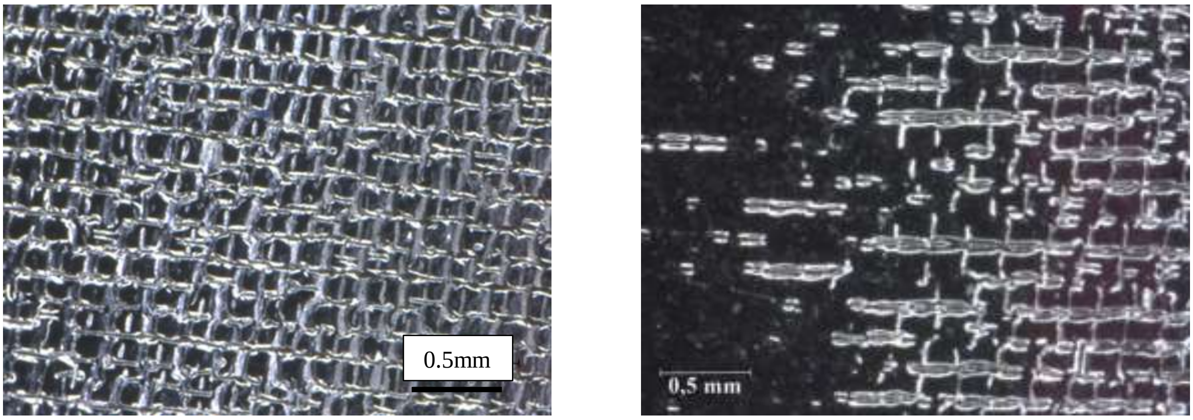
Microhardness was measured using a PMT-3 device under load of 2 N.

The surface roughness R_a of the samples after cutting was investigated using TR200 profilometer from TIME Group Inc.

The microstructure of the surface after cutting was studied using LEICA Z6 APO microscope for metallography, and Semplor NANOS microscope for scanning electron microscopy (SEM).

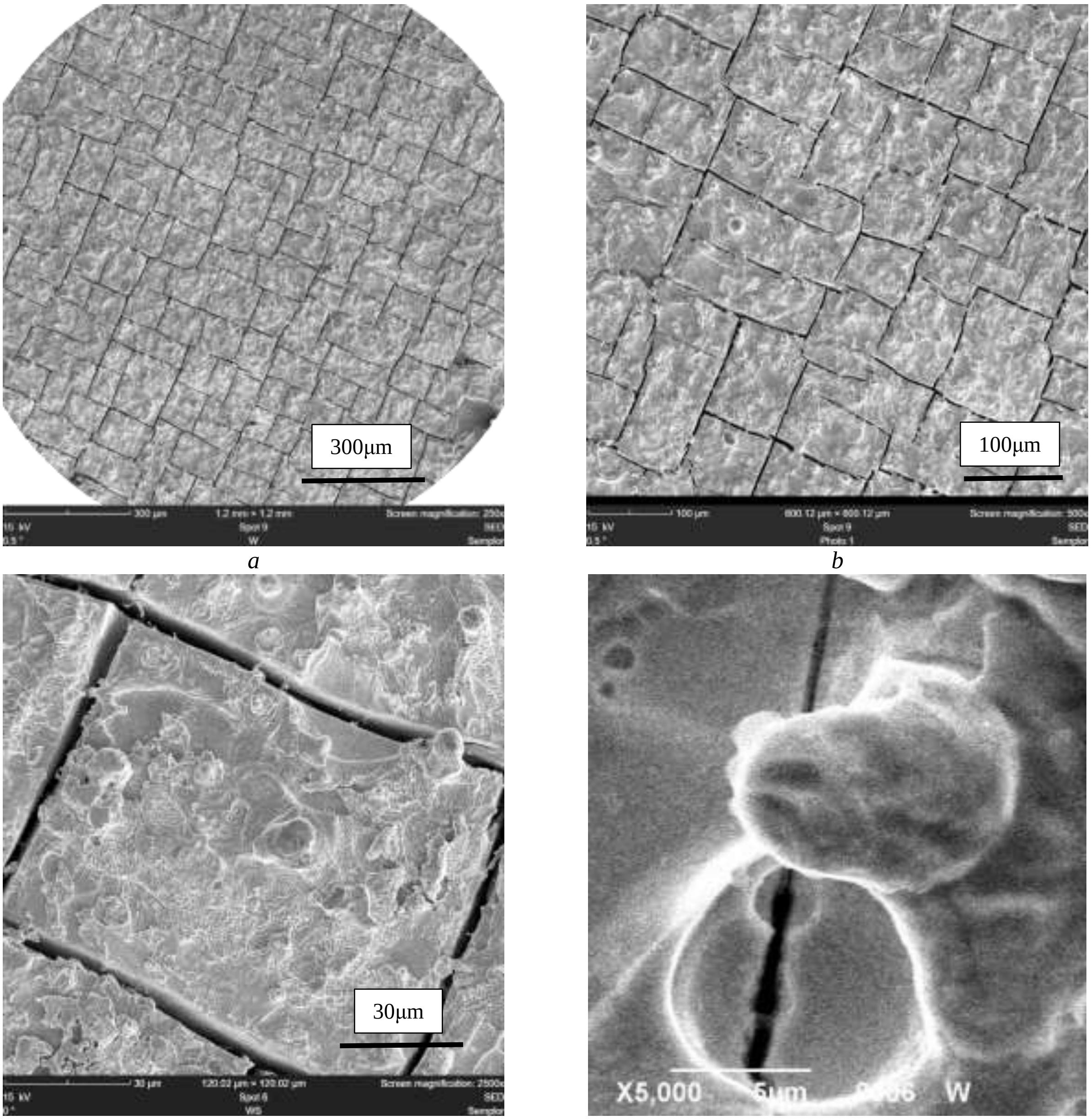
RESULTS AND DISCUSSION

X-ray studies showed that the single crystal has a fairly high purity: the tungsten content is 95.3 wt.%, the main impurities, Hf, Re, Ta, compose 0.6, 2.1, 2.0 wt.%, respectively.



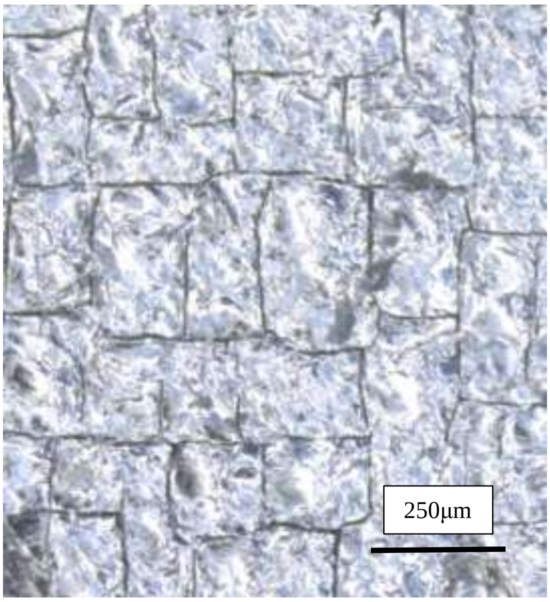
a *b*

Fig. 1. Optical micrographs of the sample #1, cut with the BP-95d machine perpendicular to the growth axis of the single crystal: a – general view of the surface after cutting; b – view of the surface after partial electropolishing (more metal is removed on the left edge of the sample).
The plane of the section is parallel to the crystallographic plane (110)

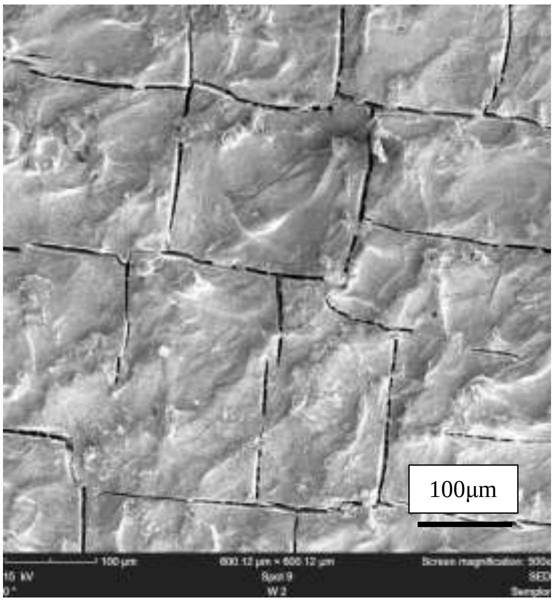


c *d*

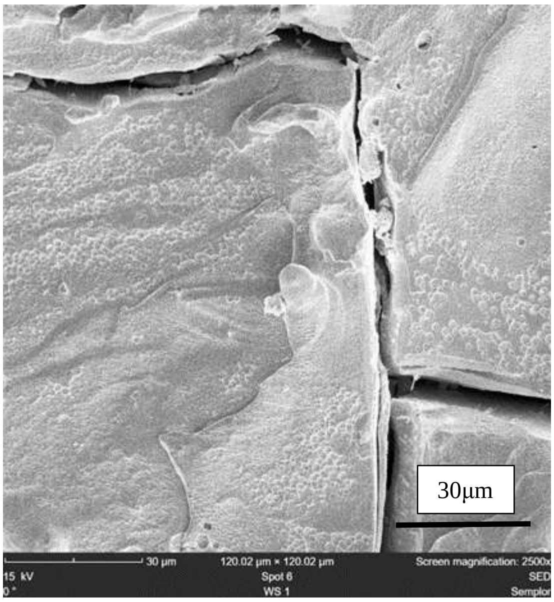
Fig. 2. SEM images of the surface of the tungsten single crystal after cutting with the BP-95d machine, sample #1: a–d – different magnifications



a

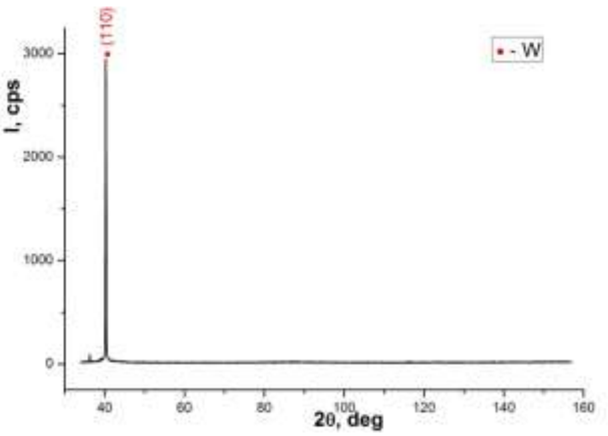


b

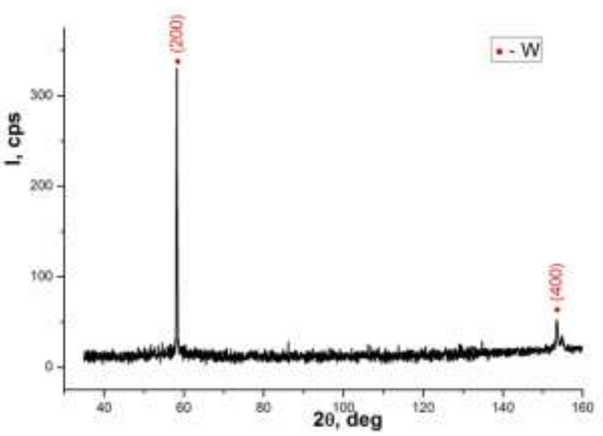


c

Fig. 3. Sample #3, after cutting with the EFH43B machine: a – general view; b, c – SEM images of the surface of the tungsten single crystal sample at different magnifications



a



b

Fig. 4. Diffraction patterns in the θ-2θ scanning mode *from tungsten samples: a – sample #1, cut perpendicular to the growth axis; b – sample #2, cut along the growth axis*

First of all, it should be noted that neither the direction of cutting the samples (#1, 2, or 3) nor annealing significantly affect the studied properties of the samples, therefore further observations will generally concern the sample #1.

Studying the surface of the samples after cutting at macroscopic level revealed significant difference in its roughness depending on a machine used. This observation is confirmed by the results of measuring the roughness R_a: cutting with the BP-95d machine provides $R_a = 2.199$, while the EFH43B machine gives $R_a = 3.906$.

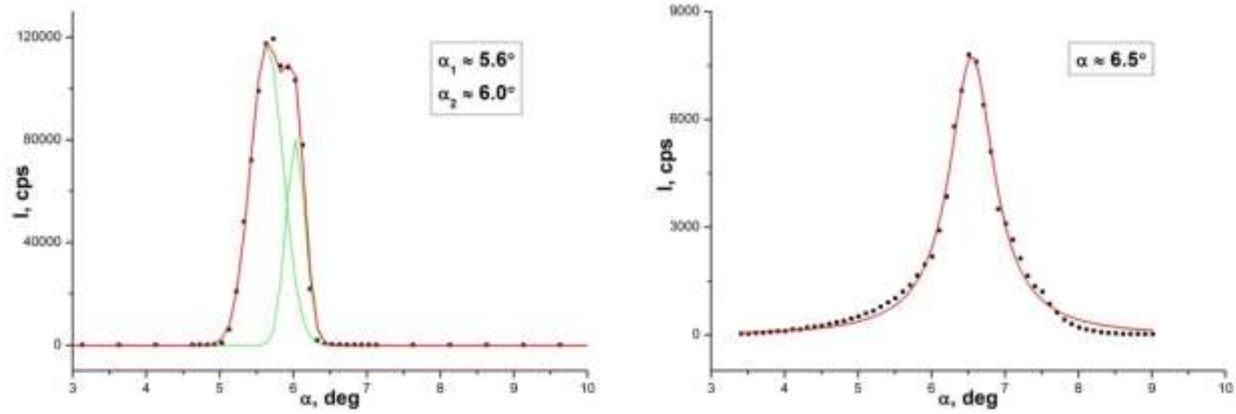
Optical micrographs of the surfaces of samples #1 and #2, given in Fig. 1, reveal, as in work [3], a structure similar to a paving stone, the individual blocks of which form a grid and have a rectangular shape or a shape close to a square.

SEM images of the surfaces of the samples are presented in Fig. 2 (cutting on the BP-95d machine) and in Fig. 3 (cutting on the EFH43B machine).

As shown by the SEM study of the surface (see Figs. 2, 3), the grid observed in the metallographic images (see Fig. 1,a) is formed by a system of cracks that intersect almost at right angles and form block fragments. Comparison of Figs. 2 and 3 shows that the average block sizes are approximately in the range $l \approx 100…300$ μm for cutting with the BP-95d machine, and they are in the range of $l \approx 300…400$ μm for the EFH43B machine cutting. Cracks dividing the blocks are appreciated to be of d ~ 200 μm in depth.

Diffraction patterns of the samples #1 and 2 in the θ-2θ scanning mode are shown in Fig. 4. Diffraction pattern of sample #1, cut perpendicular to the growth axis (see Fig. 4,a), contains only the (110) diffraction peak. Whereas, diffraction pattern of sample #2, cut along the growth axis (see Fig. 4,b), contains weak diffraction peaks from the {100} family of planes. Measured lattice parameter of tungsten samples is $a = (3.1652 \pm 3 \cdot 10^{-4})$ Å.

X-ray single crystal orientation analysis of the samples revealed that sample #1 (cut perpendicular to the growth axis) has {110} crystallographic orientation. There are 2 maxima (blocks) on the rocking curve (Fig. 5,a); the deflection angles of the [110] directions according to the sample axis are $\alpha_1 \approx 5.6°$ and $\alpha_2 \approx 6.0°$, respectively (e.g., misorientation angle of blocks is only $\Delta\alpha \approx 0.4°$). Sample #2 (cut along the growth axis) has {100} crystallographic orientation, the deflection angle of the [100] direction relative to the sample axis is $\alpha \approx 6.5°$ (Fig. 5,b). The half-width of the rocking curve is FWHM = 0.74°. No blocks or grains were detected during scanning. That is, the ingot is oriented with the crystallographic direction [110] parallel to the growth direction of the single crystal, while the side faces are oriented perpendicular to the crystallographic directions <100>.



a *b*

Fig. 5. Rocking curves of tungsten samples: a – {110} crystallographic plane of the sample #1, cut perpendicular to the growth axis; b – {200} crystallographic plane of the sample #2, cut along the growth axis

Residual stress analysis was carried out only on sample #1. It was established that this sample has significant tensile stresses of the magnitude $\sigma_\varphi = 765$ MPa. Calculated value of the lattice parameter in the unstressed state is $a_0 = 3.1705$ Å, which is larger than the literature value for pure tungsten ($a_{lit} = 3.1648$ Å), hence the difference $\Delta a = 5.7 \cdot 10^{-3}$ Å. The larger lattice parameter can be explained by the presence of Ta and Hf impurities in the ingot, which have larger metallic radii than tungsten ($r_W = 1.39$, $r_{Re} = 1.37$, $r_{Ta} = 1.46$, $r_{Hf} = 1.59$ Å).

Microhardness H_μ measurements showed that after mechanical polishing the single crystal has $H_\mu = (4500 \pm 170)$ MPa. After partial electro-polishing (right edge of Fig. 1,b) inside the blocks $H_\mu = (4080 \pm 210)$ MPa, and after full electro-polishing (left edge of Fig. 1,b) the microhardness decreases to $H_\mu = (3660 \pm 170)$ MPa. Note that after partial electro-polishing (as in Fig. 1,b), the shape of the diamond pyramid imprints indicates that the metal removal rate during polishing around the block boundary, i.e. near the cracks, is significantly higher than inside the blocks.

Explanation of the microstructure features of the tungsten single crystal surface after electric discharge cutting (Figs. 1–3) should be sought in at least two factors: the process of manufacturing a single crystal and the process of its cutting. The production of massive ingots is accompanied by the occurrence of significant internal stresses in them, tensile in this case, as evidenced by X-ray research. And electric discharge cutting is accompanied by very high temperatures (according to some data, up to 10000 °C [6]) and may be accompanied by the occurrence of shock thermal stresses. Therefore, the residual internal stresses σ_r in the material caused by these factors may exceed its tensile strength σ_b, so the relaxation of these stresses will occur through the appearance of cracks, the breakdown of the surface volume into small fragments/blocks [3]. The emergence of cracks is significantly affected by the cutting mode and, possibly, the wire tension and its oscillations. The above-mentioned greater dimensions of blocks after cutting with the EFH43B machine are obviously related to the difference in technological parameters of the used machines. A more detailed study of this phenomenon requires additional research.

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МІКРОСТРУКТУРА ПОВЕРХНІ МОНОКРИСТАЛА ВОЛЬФРАМУ ПІСЛЯ ЕЛЕКТРОЕРОЗІЙНОЇ ОБРОБКИ

І.П. Кісляк, І.В. Колодій, О.М. Великодний, С.О. Соколов, І.М. Тихоновська, С.В. Худяков, Л.О. Пироженко

Серед методів виготовлення дослідних зразків металевих матеріалів широке розповсюдження має метод електроерозійного різання. Стан поверхні зразків, зазвичай, має велике значення для досліджень. Автори вивчали вплив процесу електроерозійного різання на стан поверхні зразків, що виготовлялися з монокристала вольфраму. Метал оброблявся двома різними верстатами. На отриманих зразках вивчалась мікроструктура поверхні, мікротвердість, проводилися рентгенівські дифрактометричні дослідження. Поверхня зразків нагадує бруківку з блоками розміром $l \approx 100…400$ мкм. Оцінка глибини d тріщин, що розділяють блоки, дає величину $d \sim 200$ мкм. Мікротвердість варіюється у межах $H_\mu \approx 3600…4500$ МПа залежно від стану поверхні. Макронапруження (розтяг) поверхні складають $\sigma_\varphi = 765$ МПа. Аналіз експериментальних даних свідчить про те, що стан поверхні зразків вольфраму зумовлюється технологією виготовлення вихідного зливку та особливостями процесу електроерозійного різання.