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INFLUENCE OF HEAT TREATMENT ON THE STRUCTURE, PROPERTIES AND DURABILITY OF FLAT TUNGSTEN CATHODES

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ABSTRACT

The effect of heat treatment on the structure and properties of flat tungsten thermionic cathodes (W-cathodes) obtained by hot pressing (flattening) of blanks from 99.99 % pure tungsten wire was investigated. The effect of temperature (800–1200 °C) and vacuum annealing time (0.3–2.0 h) on the formation of a recrystallization structure in W-cathodes was studied; comprehensive comparative studies of the structure, mechanical and operational properties were carried out. It was found that to increase the service life of W-cathodes a mixed structure should be formed in them in the following quantitative ratio: 5–25 % — equiaxed recrystallized grains 1–8 μm in size; the remaining volume of the cathode material should retain the original oriented structure.

KEYWORDS: thermionic cathodes, tungsten, electron beam installations, recrystallization, microstructure

INTRODUCTION

Electron beam guns (EPG) are widely used to conduct the processes of melting, evaporation and condensation in vacuum. Their consumable elements are tungsten thermionic cathodes (W-cathodes), which are the source of electrons. This most important EBG component determines the electric, energy and service properties of vacuum electron beam installation (EBI). W-cathodes with a flat surface (in the form of plates) are widely applied now as electron sources for industrial processes. Electron emission occurs, when these cathodes are heated to high temperature [1–3].

The cathode service life is determined by the speed of its geometry change (elongation, distortion, etc.) during high-temperature service and repeated heating-cooling cycles, leading to deterioration of the beam shape and need for cathode replacement. According to patent information, the main attention is paid to development of variants of the flat cathode design, while preserving polycrystalline tungsten as the material [2–5]. In practice it turned out that not only the design determines the cathode service properties, but also the material from which it is made. However, in application of alloys of tungsten (for instance single-crystal W-4 % Ta alloy [6]) and tungsten doped with SiO₂, K₂O, Al₂O₃ additives [7], as well as tungsten with To, Ta, Re coating [8] as flat cathode materials, a significant limitation of their application sphere

was found (REM coatings are used predominantly for indirectly heated cathodes). Therefore, tungsten remains a priority material for directly heated thermionic cathodes. Unfortunately, there are practically no data in publications as regards high-temperature studies of the structure-properties-performance complex of cathodes from pure W.

Two processes: deformation and recrystallization are widely used during cathode manufacture. In order to improve the required material characteristics, it is important to select the optimal modes of impact on it. Local W-cathodes are produced from wire under plant conditions during a multistage process, alternating pressing and heat treatment [3, 9]. However, the need to upgrade the technologies of manufacturing W-cathodes for EBG in terms of improvement by the cost/effectiveness criterion remains important and relevant to this day, and it became particularly urgent now under the conditions of import substitution and saving of costly metals.

The objective of this work was development of the modes of heat treatment (HT) for material of the already supplied W-cathodes, to improve the properties required to increase their service life.

MATERIALS AND METHODS OF INVESTIGATION

W-cathodes supplied for use in EBG, are plates 0.6 mm thick, 3 mm wide and 100–140 mm long. Their manufacturing method is multistage hot press-

ing of blanks from tungsten wire of 99.99 % purity described in literature [3, 9]. Total degree of deformation is estimated to be equal to 60–65 %.

Cathodes for investigations were selected randomly from three cathode batches of the total amount of 1000 pcs, produced by “TANGSTEN” Ltd. (Kharkiv) in 2023.

To determine the initial temperature of recrystallization and study the dynamics of its processes, vacuum annealing of the supplied W-cathodes was conducted, for 1 h at different temperatures in the range from 800 to 1200 °C, as well as isothermal annealing in vacuum at 1200 °C in the time interval of 20–120 min.

Structural changes were controlled using optical (Polyvar Met) and scanning electron microscopy (Cam Scan 4D). Computer analysis of the images and statistical data processing with “Media cybernetics image analysis program” Image-Pro Plus version 6.0 software was applied to study the structural changes in W-cathodes.

Mechanical properties were assessed on the base of measurements of Vickers microhardness (HV), using Micro Duromate 4000E attachment to optical microscope, and by automatic microindentation method (Berkovich indenter) with automatic recording of the load diagram (indenter driving into the material) in MicronGamma instrument [10].

Service properties were determined when testing under the actual conditions of cathode operation in EBG of PE-123 in EBI of UE-202 and UE-210 type [2]. The cathode was mounted in the EBG cathode head, having first controlled the tension of the spring, stretching the cathode. A tungsten tablet 70 mm in diameter was used as evaporation material, which was placed into the water-cooled crucible. In operation, in addition to constant mechanical loads, the cathode is also exposed to multiple cyclic temperature loads. The test cycle simulated the temperature profile of cathode operation, and it consisted of the following stages: cathode heating and entering the operating mode (beam current of about 1.3 A, 60 mm diameter of the electron beam) for 1 min, working in this mode for 30 min, cathode switching off and cooling — 10 min. Test cycles lasted up to violation of electron beam geometry (beam spot going beyond the crucible limits in the operating mode), which was followed by recoding the total time of cathode working in the operating mode.

RESULTS AND THEIR DISCUSSION

Cathode microstructure in as-delivered state is polycrystalline and fibrous (Figure 1, *a*). Note that in both the sections made in the transverse and longi-

tudinal directions of the cathode, the structure is homogeneous, its morphology is the same, and consists of elongated grains with average width of 3–5 μm , their length is 40–100 μm , individual grains reaching the length of 200 μm . No anisotropy of mechanical properties was found, either. Obtained microhardness values of 5.4–5.6 GPa are characteristic for both the directions.

One of the important characteristics, determining metal behaviour during HT, is the start of recrystallization, which has a significant influence on the change of physical-mechanical properties of structural materials. As determined by A.A. Bochvar, the temperature of the start of recrystallization (TR) is related to absolute melting temperature by the following relationship: $TR = 0.4T_m$ ($T_m \text{ W} = 3680 \text{ K}$). Known recrystallization diagrams [11] also allow assessing TR in advance, depending on the value of preliminary deformation. According to literature data, the temperature of the start of initial recrystallization for unalloyed tungsten is in the range of $TR = 1373\text{--}1573 \text{ K}$ (1100–1300 °C) [12, 13].

However, TR depends on many parameters, which determine its more accurate value [14]. Three factors: amount of preliminary deformation, heating temperature (T) and holding duration (τ) have the strongest influence on the recrystallization process and determine the final grain size and mechanical properties of the material. At fixed amount of deformation (unchanged manufacturing conditions) two more factors remains, which require precising TR for the studied W-cathodes — T and τ .

Conducted analysis of the dependence of W-cathode microhardness on vacuum annealing temperature (T) in the selected temperature range of 800–1200 °C at fixed holding ($\tau = 1 \text{ h}$) showed that microhardness values do not change right up to 1000 °C (Figure 2), and only after reaching the temperature of 1100 °C, the microhardness slightly decreases (by 3–4 %). No visible changes in the microstructure were found up to the temperature of 1100 °C with the selected investigation methods. Noticeable changes in the microstructure occur in the case of temperature rising up to 1200 °C. New equiaxed grains form against the background of the initial structure (Figure 1), which is accompanied by microhardness lowering by 10 % compared to the initial one (Figure 2). Obviously, in our case, increasing of annealing temperature of W-cathodes to 1200 °C, and holding for 1 h actively stimulate the start of the initial recrystallization process. Further detailed studies of the kinetics of this process were conducted at the temperature of 1200 °C.

It was found that after isothermal annealing operations a two-phase microstructure forms in the entire

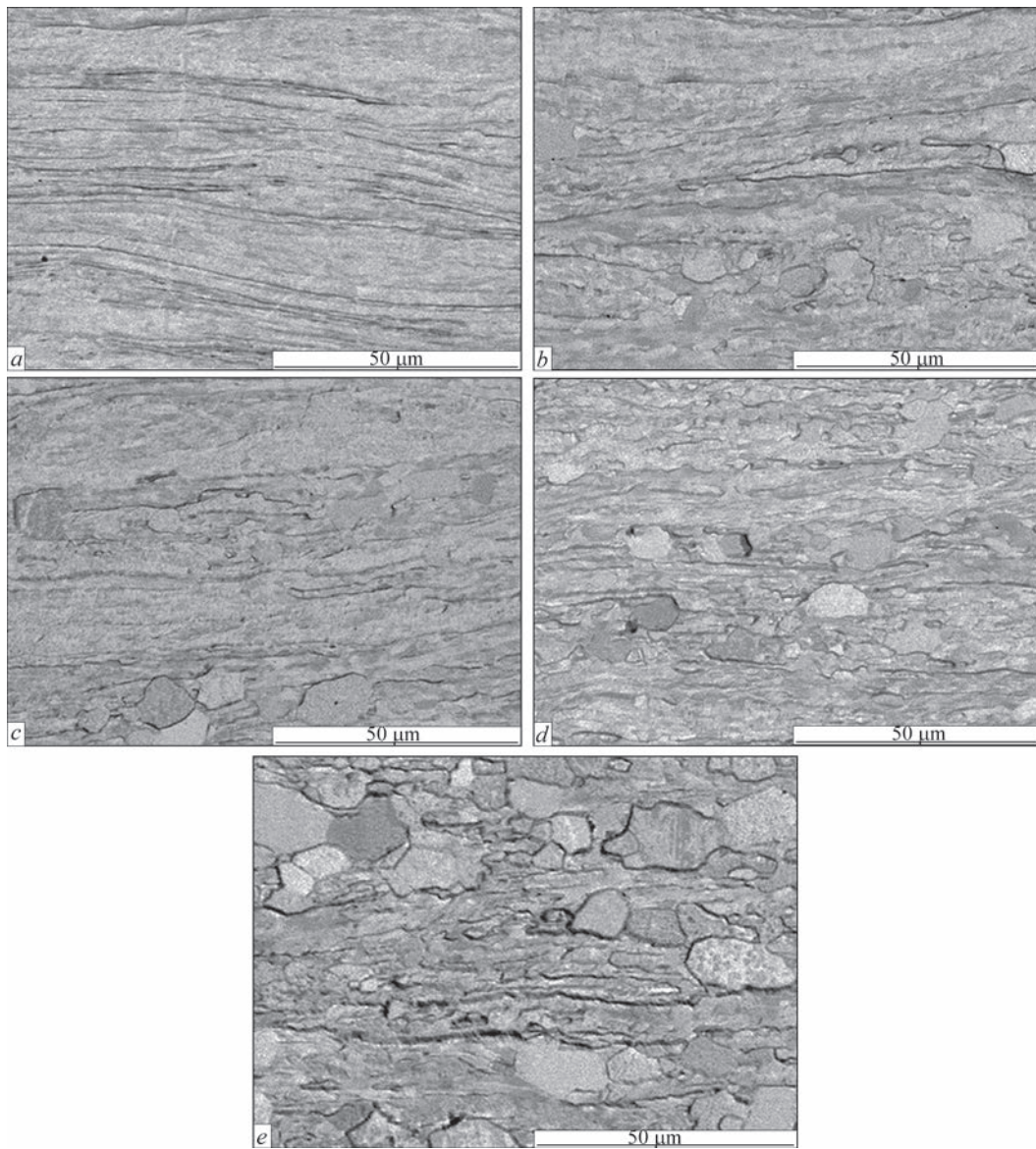


Figure 1. W-cathode structure: *a* — in as-delivered state; *b–e* — after vacuum annealing at the temperature of 1200 °C and annealing time of 30, 50, 80 and 120 min, respectively

studied time interval ($\tau = 20\text{--}120$ min), which is a mixture of initial elongated and equiaxed recrystallized grains (Figure 1).

As recrystallized grains present in the structure have a wide range of sizes (for instance, from 2 to 21 μm at $\tau = 90$ min), the value of the average grain size can distort the information about the occurring processes. As a result of the performed computer processing of microstructure images and statistical analysis, curves of frequency distribution of grains by size were plotted for W-cathodes, annealed during different τ at the temperature of 1200 °C. The distribution curves were used to determine the probable grain size d for each of the studied τ , and the results are given in Figure 3 (curve 1). Change in the volume fraction of recrystallized V phase with greater holding time is shown in Figure 3 (curve 2).

Given dependencies demonstrate that for W after deformation processing, as in our case, fine equiaxed grains of 1 μm size were detected already after 20 min of isothermal holding. The recrystallized phase volume was 5 %.

With increase of the duration of isothermal annealing (τ), grain size d and their volume fraction in the cathode material are increased simultaneously (Fig-

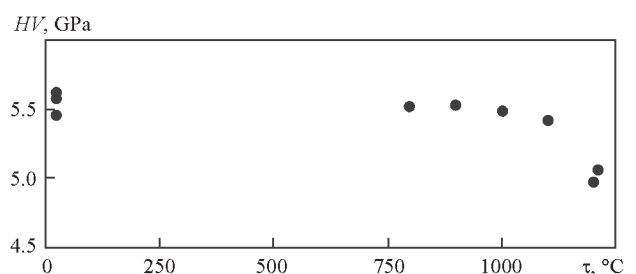


Figure 2. Dependence of tungsten cathode microhardness on vacuum annealing temperature ($\tau = 60$ min)

ure 3, curves 1, 2). The processes run more actively in τ interval from 20 to 60 min, and their slowing down is observed at $\tau > 60$ min.

Note that with τ increase part of the grains are coarsened, but new ones also appear. Thus, the structures recorded by us in the entire interval of annealing time (Figure 1) form as a result of superposition of two parallel processes: the process of new fine grain formation proceeds together with growing of the already formed grains. Thus, even at maximum $\tau = 120$ min, initial recrystallization is incomplete, and the volume of the recrystallized phase in this case is not more than 60 %. Based on the generated data on the structure in the longitudinal and transverse directions we can assume that partial recrystallization runs homogeneously through the entire volume of cathode material.

Structural changes correlate with the change in mechanical properties of W-cathodes. With τ increase, a monotonic lowering of microhardness HV from 5.5 GPa in the initial state to 4.7–4.9 GPa takes place ($\tau = 120$ min) (Figure 4). This corresponds to the known fact that the processes of initial recrystallization are accompanied by lowering of internal stress and strength of material. However, taking into account the incomplete recrystallization and the volume taken up by it the resulting reduction of cathode material microhardness is not higher than 13 %.

The key factor ensuring the object strength, as well as a measure of elasticity of the material, from which it is made, is the Young's modulus (E). In the work E was calculated by the obtained load-hold-unload diagrams in the coordinates of load — depth of indenter penetration by the procedure described in a number of works [10, 15]. Resultant average E value for each of τ is taken from calculations of not less than ten measurements. Figure 4 shows changes in the modulus of elasticity of W-cathodes, depending on the time of annealing at unchanged temperature of 1200 °C.

Average value of the modulus of elasticity of W-cathodes in the initial state is equal to 213 GPa. The difference in E values obtained in the longitudinal and transverse sections of the cathodes is small, being within the instrument error.

With increase of holding time at annealing E changes nonmonotonically: after increasing to maximal value of 263–270 GPa in τ time interval from 20 to 50 min its noticeable lowering occurs, and after 90 min of holding the modulus of elasticity of annealed tungsten cathodes is below the initial one.

All the obtained E values are in the range of 180–270 GPa. This is less than the values of the modulus of elasticity E_0 for a massive single-crystal tungsten sample, according to the reference data, E_0 400–411 GPa [13], and the experimentally derived value

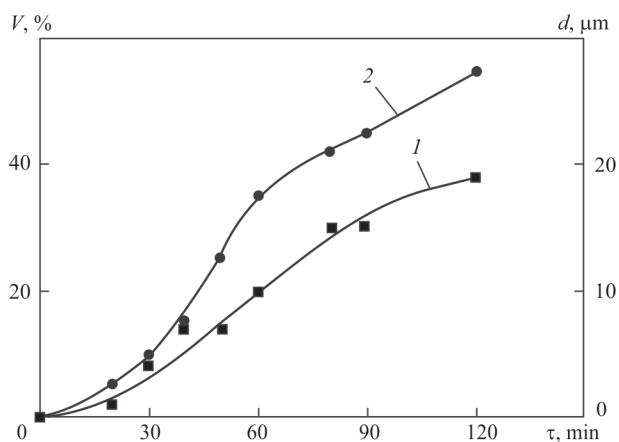


Figure 3. Influence of annealing time (τ) on recrystallized grain size d (1) and volume fraction V (2)

$E_0 = 355$ GPa for polycrystalline W [16]. It is, however, higher than the value of the modulus of elasticity 100 ± 30 GPa, which the authors of work [17] determined for nanostructured tungsten based on experimental data. A general trend is observed of decreasing of the modulus of elasticity with reduction in the grain size and respective increase in the intercrystalline boundaries. In this case, the correlation of the modulus of elasticity with the size and amount of the recrystallized phase was established experimentally, and a region of maximal E values was determined, which corresponds to the presence of 5–25 % of recrystallized grains of 1–8 μm size in the structure of W-cathodes.

When solving the question of materials application, several parameters must be taken into account, as usual. During operation, the cathode is exposed not only to temperature loads, but also to mechanical loads, namely constant tension due to EBG design.

One of the causes for shortening of the service life of the supplied W-cathodes is their stretching during operation right up to sagging. Although creep resistance is the determining property of refractory materials, reduction of the creep rate is one of the tasks aimed at improvement of the cathode performance.

When conducting an additional experiment for comparison with the initial one, a cathode was select-

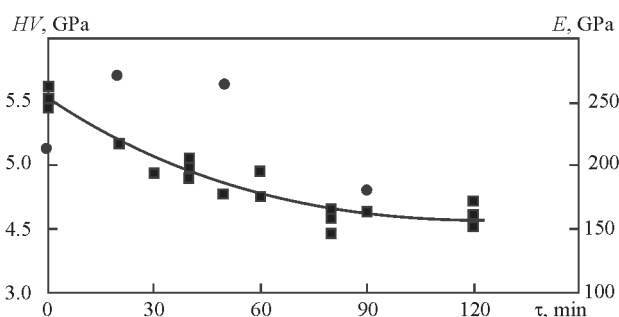


Figure 4. Influence of annealing time (τ) on mechanical properties of W-cathodes: line — microhardness; dots — modulus of elasticity

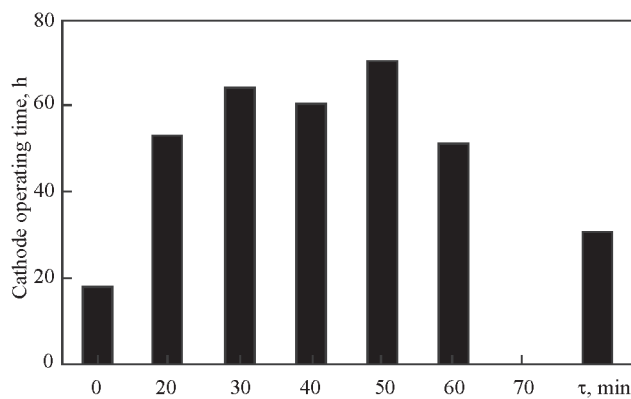


Figure 5. Service life of W-cathodes in the initial state and depending on vacuum annealing time

ed with maximal rigidity of the area with high E value, which has been subjected to HT by the following mode: $T = 1200\text{ }^{\circ}\text{C}$, $\tau = 30\text{ min}$. Both the samples were tested under similar conditions: at a high temperature (operating temperature of directly heated cathode of $2200\text{--}2300\text{ }^{\circ}\text{C}$ ($0.77T_m$)) and under constant tensile stress of estimated value of 35.4 MPa , with 10 min time of operation in such a mode (it was noted that in case of a significant elongation of the cathode, it occurs in the first $10\text{--}20\text{ min}$ of operation). Measurement results showed that the magnitude of deformation $\Delta//l$ of the initial and heat-treated cathodes was equal to 0.9 and 0.7% , respectively. Thus, conducting cathode HT leads to lowering of creep rate.

Creep process in our case is associated with the impact of a whole number of different physical factors: tensile stresses; high temperature, cathode heating being characterized by a high rate and rather quickly entering the stationary operating mode; presence of an axial temperature gradient, resulting in a secondary recrystallization zone ($TSR\ 2000\text{--}2100\text{ }^{\circ}\text{C}$) in the active central part of the cathode and possible development of compressive stresses, because of intensive metal expansion in this area is prevented by cold cathode ends. The question of creep mechanism is complicated, requiring additional studies and theoretical analysis, and it was not part of the objective of this work, but owing to great practical interest, investigations in this area will be carried on.

The physical creep mechanism probably is of a predominantly dislocation nature, based on the data of the map of creep deformation mechanisms of tungsten [18], and taking into account our values of the two main parameters, working temperature and stresses τ . It is known that the rate of this process is sensitive to structural characteristics, namely grain size and shape. It is obvious that presence of recrystallized grains of $4\text{ }\mu\text{m}$ size in the amount of 10% , created additional boundaries in the direction normal to the load and this way slightly reduced the creep rate.

Experimental determination of the entire complex of processes taking place in the cathode assembly (including cathode material erosion as a result of ion bombardment) is quite complicated. Only indirect measurement of physical values determining the flow of processes inside the cathode is possible. Final response to the question of rationality of heat treatment application for W-cathodes is only given by checking their service properties under the actual conditions of EBG operation.

Results of evaluation of service life of as-delivered cathodes, as well as cathodes after HT in different modes, are shown in Figure 5. In as-delivered state the service life of the cathode varies from 10 to 18 h (8 cathodes have been tested), and after vacuum annealing ($T = 1200\text{ }^{\circ}\text{C}$) during τ from 20 to 60 min it reaches the maximal $50\text{--}60\text{ h}$. This coincides with the area of maximal E values (Figure 5) against the background of slight softening (Figure 4), due to internal stress lowering at the beginning stages of initial recrystallization process (Figure 3). Further increase of annealing time leads to lowering of operational characteristics, so it is not rational.

Thus, based on analysis of the results of the conducted complex studies, an optimal HT mode ($T = 1200\text{ }^{\circ}\text{C}$, $\tau = 20\text{--}50\text{ min}$, protective environment is vacuum) was selected, in order to improve the service properties of W-cathodes. Such HT leads to the presence of a mixed two-phase structure of the recrystallized and unrecrystallized phases. Presence of a recrystallized phase of $1\text{--}8\text{ mm}$ size in the amount of $5\text{--}25\%$ ensures the level of mechanical properties, necessary for reliable operation of the cathodes: microhardness of $5.0 \pm 0.15\text{ GPa}$, modulus of elasticity of $260\text{--}270\text{ GPa}$.

CONCLUSIONS

1. It is found that to improve the quality of flat W-cathodes produced by pressing from tungsten wire, a mixed structure should be formed in them with the following quantitative ratio: up to $5\text{--}25\%$ of equiaxed recrystallized grains, with the rest of the cathode material volume preserving an unrecrystallized structure.

2. It is shown that such a condition of W-cathodes is reached after conducting the HT: vacuum annealing at $1200\text{ }^{\circ}\text{C}$ for $20\text{--}50\text{ min}$ is recommended.

3. Service life of the cathodes heat-treated in such a mode reaches $50\text{--}60\text{ h}$ ($3\text{--}5$ times higher, compared to unheat-treated cathodes) and it corresponds to the level of endurance of foreign-made cathodes, the cost of which is several times higher.

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CONFLICT OF INTEREST

The Authors declare no conflict of interest

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