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Sublimation of tungsten on a spiral heater

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The analysis of the tungsten sublimation process in a thermal unit with a spiral-shaped heater at a temperature of 1200−1720 K. A process model in the COMSOL MULTIPHYSICS system has been developed to evaluate thermal conditions. The formation of tungsten microcrystals in the regions of the spiral with a lower temperature has been established. Carbon inclusions in the surface of the spiral were noted, the source of which was the oils sorbed in the pores of the wire during its production by drawing.

Keywords: tungsten spiral, sublimation recrystallization, thermal process model.

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Tungsten has a firm spot in vacuum technology and electrical engineering. Owing to its refractory quality (melting point $T_m = 3420\,^\circ$C) and resistance to electrochemical corrosion, tungsten is the key material for fabrication of filaments, heaters, and electrodes [1]. Improvements in the composition and structure of tungsten-based alloys, their production procedure, and operating regimes have led to increased thermal stability and service life of heaters and their widespread adoption in various fields of science and technology [2]. Studies into tungsten oxidation within the temperature range of 1000−3300 K [3–5] and tungsten evaporation in vacuum under low pressures of hydrogen and water vapor at temperatures of 1650−3230 K [6,7] and in an argon environment under heating within the range of 2850−3150 K [8,9] have already been published. It was demonstrated in several papers [10–12] that a combination of high temperature and high DC current density leads to electromigration in tungsten, altering the material structure and the conductor shape. The aim of the present study is to analyze the process of tungsten sublimation in a spiral-shaped heater at a temperature of 1200−1720 K.

A tungsten wire spiral heater, which is applied in synthesis of high-temperature semiconductor and metallic materials, was used for experiments [13]. Resistive heating was performed in the chamber of a „Zona-03“ growth setup in an argon (grade 1) environment at a pressure of $\sim 1.8 \cdot 10^5$ Pa. The design of the thermal unit is shown in Fig. 1. The spiral is made of double-twisted VA tungsten wire 0.5 mm in diameter and is wound on dielectric rods positioned evenly around the circumference of a cylindrical base. To reduce heat loss, the heater was provided with a multilayer insulating shell. Experiments were carried out with the voltage increasing smoothly from 2 to 20 V, which led to an increase in current from 6 to 15 A. The holding time at maximum power was 30 min. The average temperature of the spiral was estimated using the following analytical expression [14]:

$$t = -1.6181(R_t/R_{300})^2 + 202.98R_t/R_{300} + 127.76, \quad (1)$$

the obtained value was $\sim$ 1406 K.

The temperature distribution within the spiral volume was estimated using the COMSOL Multiphysics 6.2 software package with the Heat Transfer in Solids, Electric Currents, and Surface-to-Surface Radiation interfaces. The needed parameters of these interfaces were combined through multiphysics couplings. Since the heater is placed in a multilayer heat-insulating unit, heat exchange with the environment was considered to be negligible. To simplify the model, the wire was assumed to be solid with an equivalent diameter of 0.7 mm in calculations. The argon atmosphere prevented oxidation of the spiral surface. The problem of modeling the thermal characteristics of the spiral heater was not axisymmetric. The used model included

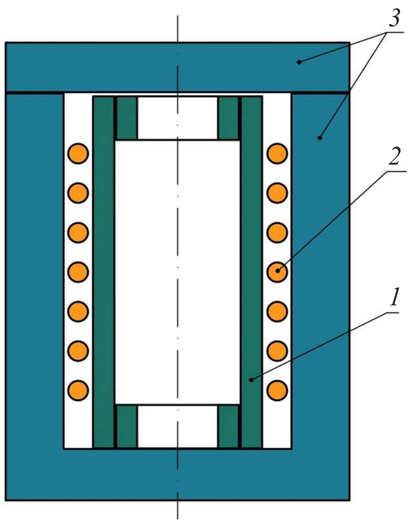

Figure 1. Thermal assembly. *1* — Ceramic rod, *2* — tungsten wire, and *3* — heat insulation.

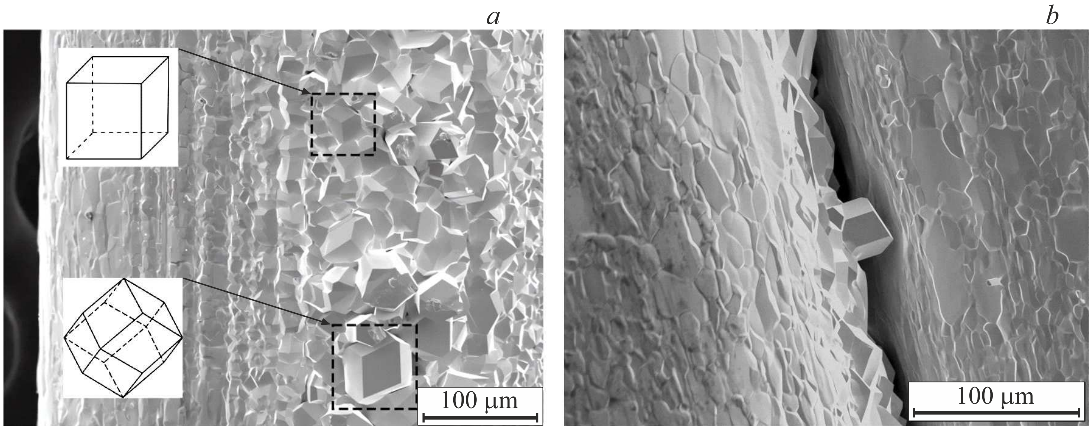

Figure 2. Wire surface after heat treatment. *a* — Crystals on the inner surface of one of the twisted wires; *b* — inner surface of wires in the twist gap.

equations of thermal conductivity, electric conductivity, and radiative heat transfer:

$$\begin{cases} \rho C_p \frac{\partial T}{\partial t} + \rho C_p \mathbf{u} \cdot \nabla T + \nabla \cdot \mathbf{q} = Q + Q_{ted}, \\ \mathbf{q} = -k \nabla T, \\ Q_{ted} = -\alpha T : \frac{dS}{dt}, \\ \nabla \cdot \mathbf{J} = Q, \\ \mathbf{J} = \sigma \mathbf{E} + \frac{\partial \mathbf{D}}{\partial t} + \mathbf{J}_e, \\ \mathbf{E} = -\nabla V, \\ \sigma = \frac{1}{\rho_0 \left(1 + \alpha_r (T - T_{ref})\right)}, \\ J_{ems} = \varepsilon e_b(T) + \rho_d G, \\ G = G_m + G_{amb} + G_{ext}, \\ G_{amb} = F_{amb}\, \varepsilon_{amb}\, e_b(T_{amb}), \\ e_b(T) = n_0^2 \sigma_{\mathrm{SBc}} T^4, \\ q_{r,net} = \varepsilon \big(G - e_b(T)\big), \end{cases} \qquad (2)$$

where ρ is the density, C_p is the thermal capacity, T is the absolute temperature, **u** is the velocity vector of translational motion of the body material, **q** is the heat flux, k is the thermal conductivity, α is the coefficient of temperature expansion, S is the second Piola–Kirchhoff stress tensor, **J** is the current density, **E** is the field intensity, **D** is the electric induction, $\mathbf{J}_e$ is the external current density, V is the voltage, Q is the resistive heating, σ is the electric conductivity, ρ_0 is the initial resistivity, α_r is the resistive temperature coefficient, T_{ref} is the initial body temperature, σ_{SBc} is the Stefan–Boltzmann constant, ε is the surface emissivity, T_{amb} is the ambient temperature, G_m is the mutual surface irradiation, G_{ext} is the ambient irradiation, n_0 is the refraction index of the medium, ρ_d is the diffuse reflectivity, ε_{amb} is the ambient emissivity, F_{amb} is the ambient view factor, J_{ems} is the emissive power, and $q_{r,net}$ is the net heat flux.

The initial conditions for the radiative thermal conductivity equation were as follows:

$$J_{init} = \varepsilon e_b(T_0) + (1 - \varepsilon) e_b(T_{amb}),$$

where $T_0 = 300\,\mathrm{K}$ is the initial body temperature.

The boundary conditions for the thermal conductivity equation in solids are

$$-\mathbf{n} \cdot \mathbf{q} = h(T_{amb} - T) + q_{r,net},$$

where **n** is the normal to the surface and h is the heat transfer coefficient.

As for the electric conductivity equation, the initial condition was $V_0 = 0\,\mathrm{V}$ at boundary conditions

$$V = f(x, y, z, t).$$

The tungsten wire surface was imaged with a JEOL JCM-6000 scanning electron microscope. Microcrystals were examined by energy-dispersive X-ray (EDX) microanalysis with scanning electron microscopy using a JEOL JCM-6000 PLUS microscope fitted with an energy-dispersive X-ray spectrometer.

Although the rate of tungsten evaporation in the temperature interval from 1000 to 2000 K is fairly low and varies within the range from $5 \cdot 10^{-34}$ to $1.75 \cdot 10^{-13}\,\mathrm{g/(cm^2 \cdot s)}$ [15], active processes of evaporation and condensation on the wire surface were noted. Since the spiral material is rigid, gaps formed between the wire surfaces facing the inside of the twist. Figure 2 shows microcrystals on the inner surface of one of the twisted wires, which have discernible cubic and rhombododecahedric shapes characteristic of tungsten crystals. No crystal formation was noted on the surface of the opposite wire (Fig. 2, *b*).

This may be attributed to the fact that the wires were located one above the other in certain regions of the twisted spiral; in this configuration, the surface of the lower wire was the source of evaporated material, and the upper one was the seed. The most intense deposition and crystallization proceeded along the shortest route between the two surfaces, which explains the formation of a ridge (Fig. 2, *a*) of microcrystals.

The results of EDX microanalysis (Fig. 3) revealed that the tungsten content in the studied microcrystal region is

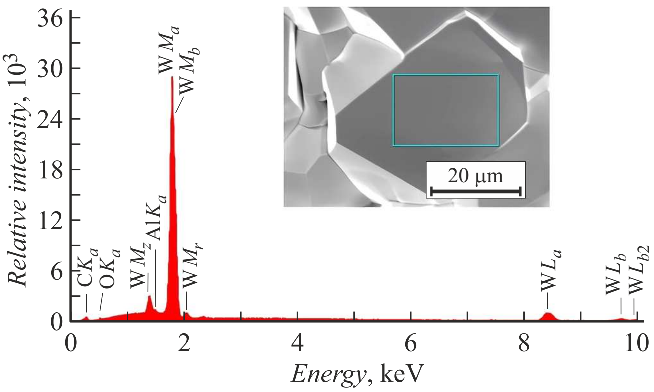

Figure 3. Results of EDX microanalysis: image of a microcrystal with the studied area highlighted (inset) and X-ray spectrum of the material in the indicated area.

97.25 mass%; the presence of Al and O impurities characteristic of the VA alloy was also noted (0.31 and 0.49 mass%, respectively). The presence of carbon (1.95 mass%) is attributable to the specifics of wire production by drawing with the use of drawing oils based on high-molecular hydrocarbons, which penetrate into surface defects and burn out during heat treatment, forming carbon.

The temperature distribution estimated based on the obtained model (Fig. 4) revealed that the spiral temperature at the hottest point did not exceed 1726 K.

The highest temperatures (1677–1695 K) were observed approximately in the middle of a tungsten wire arc between the points of its contact with the dielectric rods; at these contact points, the temperature was $\sim$ 1170 K. A stable wire temperature was established at 2000 s of heating and did not change further. The base and the cover of the stand were the least heated parts of the model with their temperature being approximately three times lower than that at the hottest point of the heater.

The median wire temperature (Fig. 4, *b*) determined in thermal equilibrium was 1524 K, while the average temperature was 1448 K. The latter value is consistent with the estimate based on formula (1); however, the simulation results demonstrate that the applicability of this analytical approach to resistive heaters is limited because of temperature variations due to differences in heat dissipation at different sections of the spiral.

The study of sublimation recrystallization of a heater made of tungsten revealed the formation of tungsten microcrystals in spiral regions with a lower temperature. Carbon inclusions on the surface of the spiral were noted. Their sources are oils sorbed in the pores of a wire during its production by drawing. A model of the process including the contributions of thermal conductivity, electric conductivity, and radiative heat transfer was proposed for the studied system.

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Conflict of interest

The authors declare that they have no conflict of interest.

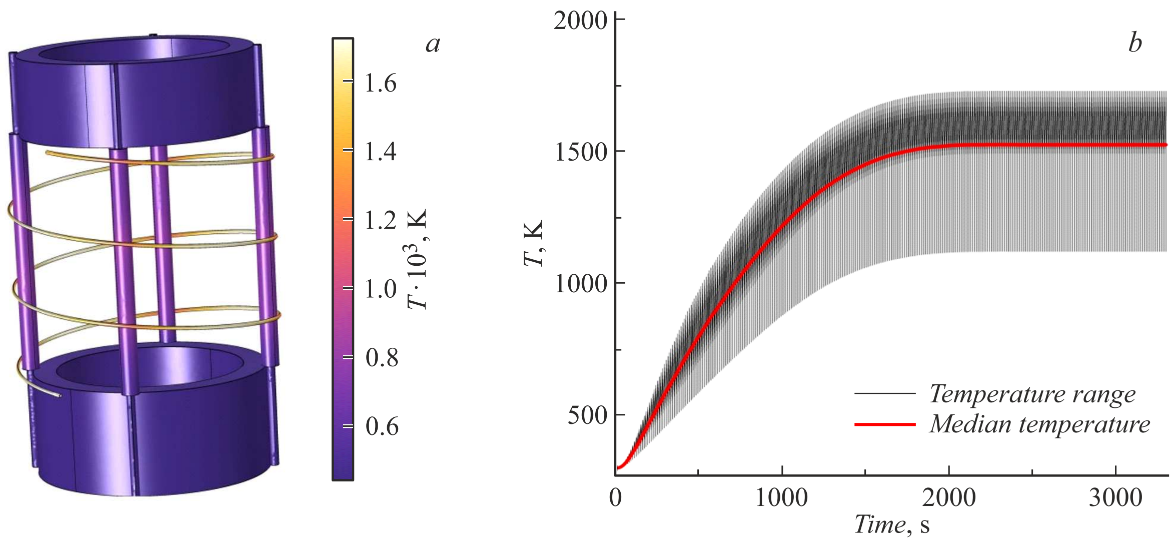

Figure 4. Simulation results. *a* — Heater model in thermal equilibrium; *b* — temporal dependence of wire temperature.

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