THE USE OF IONIZING RADIATION FOR THE TUNGSTEN PREPARATION
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ABSTRACT
The paper presents the results of experimental studies in the field of radiation material science, in particular, relating to the processes of the ionizing radiation action on the change of the properties of the powder components in the tungsten wire preparation. It is known that the shape and size of the particles are among the most important physical characteristics of the powder, influencing its technological properties, behavior of the pressings during sintering, and often determine the quality of finished products. The use of radiation treatment helps reducing the granularity of the powder, which leads to a significant densification of the solid phase during sintering due to high surface energy and chemical reactivity. The paper shows the applicability of gamma-ray effect in the technology of production of refractory metals, allowing to manage the change in the structure, physico-chemical and mechanical properties.

INTRODUCTION
As The development and use of advanced methods for improving the technology in the powder metallurgy is a priority area, limiting the development of many areas of science and technology. The better breakthrough technologies are needed to form high-quality characteristics of powdered materials, among which the radiation treatment of powders is of significant interest. Using the energy of braking gamma rays from an electron accelerator allows ensuring the formation of a new structural condition of sintered polycrystalline materials, occurring at high excitation conditions. This is due to the fact that the mechanism that binds the energy and crystal-chemical aspects provides for the binding energy transformation and the stability of the properties and structure of products obtained by powder metallurgy. The proposed method makes it possible to create a new trend for the production of powders and to obtain metals and alloys with homogenous fine-grained structure, high strength, density, resistance to wear, corrosion and radiation resistance (Chesnokov, 2000; Chesnokov, 2006).
In the production technology of refractory metals and alloys, the radiation treatment of powders is of particular interest. The most important refractory metals include tungsten, which is produced by powder metallurgy. Its common drawback is a tendency to recrystallization (Espe, 1962; Zelikman and Nikitina, 1978). The incomplete decomposition of the raw product (salt of tungstic acid) in the preparation of the metal tungsten powder should be considered as the main cause of this phenomenon. Recrystallization process is qualitatively expressed in the appearance of (the birth of) new metal grains on the sliding faces of crystals, as well as the presence of the undecomposed particles of the raw material inside the large agglomerates, and their growth, causing softening of the structure in the finished product. It follows that the proposed mechanism for
this phenomenon is precisely the dependence of the recrystallization from the granularity of the metal powder.

It is possible to reduce the tendency of products to the crystal growth if the powders of salt powders or refractory metal oxide are treated with gamma rays. The main criteria that determine the evolution of the structure of irradiated powders in the process of their preparation are the following: Firstly, the grain size variation in the composition of powders (optional grinding), and secondly, the change of the powders surface condition after exposure (spongy structure). The last factor having a significant impact on the development of sintering and crystallization processes is of particular importance. The programmed formation of the properties of powders and products therefrom, by introducing the irradiation with braking gamma rays in some key moments of production, has a significant effect on the electronic structure, causing redistribution of electric charge inside the crystal.

**RESEARCH METHODS**

The experimental procedure was planned to take into account the possibility of the use of irradiation at the stage of the synthesis of powders with respect to the plant conditions. During radiation treatment of the powder particles, by moving to higher energy levels, the electrons promote for the targeted change of their properties. Therefore, the power level saturation and the in-depth energy uniform promotes for breaking the chemical bonds and destructing the secondary particles. This implies that targeting of volume-crystal defects which are achieved by gamma radiation levels the nuclear points density distribution, increases their number, and leads to changes in powder grading.

**RESULTS**

Studies carried out using Fischer instrument showed that the average crystallite size decreased by 10% to 15%. Herewith, the higher energy states as compared with the states on the surface of large particles correspond to the electron orbits in small particle surfaces, and as a result, the gap inside the crystal bonds is facilitated, and the activity and reactivity of the particles increases. As Table 1 shows, in the processing of powders processed by gamma rays, the content of small and medium grains has dramatically increased, and the content of coarse fractions has decreased.

The experiments show that the major centers responsible for irradiation processes are associated with the micro-irregularities of the initial components (various kinds of impurity atoms, gas inclusions, etc.), which translate their neighboring areas in the oscillatory mode. In the sintered particles, the thermal avalanche is rapidly developing as the excitation wave, which leads, on the one hand, to the enrichment of the powder particles volume with the vacancies and, on the other hand, heals the micropores in the surface layer, followed by a decrease in wavelength due to recombination processes (Eighth International Conference on Fusion Reactor Materials, Japan, Sendai International Center, October 26-31, 1997; Zaykin, et al., 1999). Exactly the impurity centers set up by one-electron atoms in a crystalline powder form the structure in which the electron binding energy is much higher than that in samples obtained using industrial technology (Fig. 1).

The established pattern allows to expand understanding of the mechanism of activation and modification of submicroscopic particles that determine the grain size. Irradiation can "trigger" the phase transition of a substance from one state to another at lower energy costs, and by manipulating the irradiation dose it is possible to manage the process of formation and growth of crystals.

During sintering of excited particles in hydrogen a wide range of spatial perturbations occurs due to quantum-mechanical effect of the solid body on the diffusion processes that lead to changes in the formation of the spatial structure of the rod. The main imperfections known to modern science are the borders between the individual grains and the violation of the order the crystals arrangement in the crystal lattice. The experiments have shown that defects caused by irradiation, found directly in the grain boundary, lead it to an excited non-equilibrium state, and most importantly have an impact on such important processes as plastic deformation, recrystallization, and others.

<table>
<thead>
<tr>
<th>Metal Powder</th>
<th>Treatment Technology</th>
<th>Content of grains, he in %</th>
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</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Above 20</td>
</tr>
<tr>
<td>Tungsten</td>
<td>Industrial</td>
<td>33.7</td>
</tr>
<tr>
<td></td>
<td>Radiation</td>
<td>22.3</td>
</tr>
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Table 1. The dependence of the particle size of tungsten powders on the treatment technology
On a tungsten rod workpiece (Fig. 2a) obtained using the industrial technology, the coarse structure of the metal powder and the presence of particles in the form of impurity inclusions and undecomposed raw material within large agglomerates are clearly observed. Polycrystallines obtained after sintering, consisting of a set of interconnected grains, form a crystalline structure with defects that do not affect the order of the crystals arrangement in the crystal lattice, and the thickness of the boundaries between the grains. However, this pattern abruptly falls during sintering of excited particles and changing of the particle size distribution.

The structure of rods after sintering of powders produced by radiation technology, which vests the grains with a high surface energy, thereby changing the morphology of the system, as well as the grains interaction energy, is of particular interest. The photograph (Fig. 2b) clearly shows fine crystal grains, adherent to each other, between which the borders are located in the form of fine lines. In this case, we observe the absorption of boundaries between the grains that increases the ductility of the tungsten rod. Consequently, the obtaining of powders using ionizing radiation in the nanometer range initiates the diffusion processes in powder metallurgy and allows to controllably influence the change of the structure and change the physico-mechanical properties of solids.

The most detailed study of the structure of the tungsten rods fracture surface, which occurs by cleavage at the grain boundaries, in order to analyze the causes and course of the destruction process was carried out by fractography. (Fig. 3) shows the microscopical shots of replicas from various parts of the fracture surfaces of the tungsten rods samples at a different magnification, obtained by plant technology.

The surface of the tungsten rod (Fig. 3a) clearly shows the cleavage steps on the fracture plain of the large tungsten particle and the crack resulting from the rupture, bordering the particle contour. In the sample (Fig. 3b), the field of the particles is observed, while at the particle edge the pores and voidings are observed in the form impurity inclusions. At the fracture (Fig. 3c), the particles are seen consisting of grains of different sizes and shapes, as well as dark inclusions in the form of oxides, extracted from the sample surface by replica. The large difference in grain size (Fig. 3d) adversely affects the quality
of the finished product, and in the case of a thin tungsten wire the large grain, once in cross section, leads to rupture of the sample. Research on electron microscope showed that on the surface of destroyed samples obtained by factory technology, there is a manifestation of rather rough surface, where the fractures have a well-developed geometry and uneven granularity, which significantly affects the processing properties in the preparation of bar and wire.

Fig. 4 shows the fracture surface of tungsten rods produced by irradiation of powders using gamma rays.

Fig. 4a shows a cleavage of a tungsten sample, on whose surface the undestroyed particle is seen. The surface of the sample shown in Figure 4b is characterized by fine-grained structure with a characteristic type of fracture along the contour of the particles gap. Figure 4c shows the same surface (Fig. 4b) fragment, in which fine particles are clearly visible. The break surface of large particles is shown in Figure 4d where the subtle break steps are seen. Using the pre-processing of the tungsten anhydride by gamma rays reduces granularity, and increases the density of the material and its aligning by entire volume. Thus, the fractographic study of the tungsten rods allows assessing their suitability for the production of the wire with the desired structure before treatment. Further processing of rods welded into wire of different diameters is carried out in a rotary forging machine, followed by pulling through dies of carbide material at 700°C to 800°C, and wires of smaller diameters-through the diamond die. Tungsten wire is characterized by fibrous microstructure, and at the heating it is prone to recrystallization and sag by its gravity. Metallographic studies were carried out on the BA tungsten wire, 0.3 mm in diameter.

Tungsten wire, obtained using industrial technology (Fig. 5a), is characterized by cross-borders, and the adhesive force perpendicular to the length of the wire is significantly reduced, causing recrystallization brittleness. Samples obtained from the raw material after the radiation treatment (Fig. 5b) have a stacker structure, typical for unsnagging tungsten. Big adhesion strength of individual structure fibers with the crystals elongated direction along the axis, in combination with high strength of crystals themselves, contributes to the acquisition of high mechanical tensile strength by the metal and the ability to bend without breaking.

Fig. 6 shows microscopical shots of crystal structures obtained by industrial technology. During the manufacture of wire, the impurity inclusions are located along the boundaries of metal crystals and retain their ability to isothermal decomposition. The metallographic analysis was conducted by the research replicas along the axis of the tungsten wire in a known manner. The analysis of the structures demonstrates that the wire (Fig. 6a), produced in the

![Fig. 3 Fractometrical studies of the replica of tungsten rod produced in industrial conditions, from unirradiated WO3.](image-url)
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factory conditions, is characterized by the structure consisting of thick and loose fibers. The structures (Fig. 6b and 6c) are heterogeneous in thickness, and Fig. 6b is characterized by the appearance of both longitudinal and transverse boundaries, and Fig. 6c by the presence of thick fibers which become thinner inboard with a lot of loose inclusions. Sample (Fig. 6d) has a transverse deformed border on center, and the stepped configuration of one of the fibers. Fig. 7 shows the microstructure of the tungsten wire produced by radiation technology.

Tungsten wire, produced using the radiation, is characterized by typical fine fiber structure (Fig. 7a and 7b); moreover, the axially elongated grain is observed between the fibers. The stratification is inherent to the fragment caused during the separation of the wire in the production of the preparation, and the oxide film is visible on the surface of the wire (Fig. 7d), under which the subsurface fine fiber structure is seen. The wire samples produced using irradiation are less prone to recrystallization and have increased strength due to targeting of the heterodesmical structure of chemical bond (Aliev, et al., 2006; Chesnokov, et al., 2014).

Fig. 4 Fractometrical studies of the replica of tungsten rod produced in industrial conditions, from unirradiated WO₃.

Fig. 5 The microstructure of the surface of a tungsten wire, obtained using industrial (a) and radiation (b) technology: a-sagging wire; b-unsagging wire (Zoom × 100).
Fig. 6 The crystalline microstructure of a tungsten wire, obtained using industrial technology. (Zoom × 2,000).

Fig. 7 The crystalline microstructure of a tungsten wire, obtained using industrial technology.
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DISCUSSION
Most The comparisons of the structures shown in Fig. 6 and 7 demonstrates that the wire made according to industrial technology is characterized by the presence of lateral boundaries that cause recrystallization brittleness, and the wire obtained from rods made of WO$_3$ irradiated with gamma rays has a stacker structure (the boundaries extend along the wire axis), characteristic for unsagging tungsten.

CONCLUSION
Thus, using gamma radiation as a treatment tool it is possible to affect the kinetics of decomposition of the raw material in a programmed way, influence the granularity of the metal powder, reduce the tendency for the tungsten recrystallization, and controllably generate physical and mechanical properties. The prospect of the use of ionizing radiation will create a new trend on the production of powders and products for various sectors of mechanical engineering and instrumentation, as well as microelectronics, nuclear, aerospace, semiconductor and computer technology.

REFERENCES


