Application of the technology of induction chemical-thermal treatment to improve the physical and mechanical properties of tantalum

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Abstract. The results of the chemical thermal treatment (CTT) of tantalum in a solid carbon-containing medium in the temperature range from 1000 to 1300 °C were presented. CTT consisted in heating a workpiece with a working medium in a container made of a refractory material. The induction heating method was used for heating. After strengthening treatment, tantalum samples were characterized by increased hardness, which grew from 140 to 1100 HV0.02.

1. Introduction
Tantalum interacts with non-metallic elements that form interstitial solid solutions (H, C, N, O) [1]. For example, the absorption of hydrogen is slow under normal conditions, but at a temperature of about 500 °C, the absorption rate is maximal. As a result of this interaction, not only absorption occurs, but also the formation of chemical compounds – TaH hydrides. Tantalum absorbs nitrogen at a temperature of 600 °C, and at a higher temperature, TaN nitride is formed. Oxidation with atmospheric oxygen begins at a temperature of 200–300 °C, whereas above 500 °C intense oxidation occurs with the formation of the higher oxide Ta2O5. Simultaneous saturation with oxygen and nitrogen leads to the formation of a film with high mechanical strength compared to other coatings of tantalum oxide [2]. Carbon-containing gases (CH4, CO) at the temperature of 1200–1400 °C react with metal, which results in the formation of hard and refractory carbides, e.g. TaC [3].

A method of chemical-thermal treatment (CTT) allows the production of the necessary compounds on the surface of metals and their alloys. One of the effective ways of CTT of tantalum is carburization in a solid carburizer such as graphite. The use of eddy currents arising during induction heating is effective as a heating source [4].

2. Methodology
For CTT, the prepared sample was placed in a heat-resistant metal container and then carbon-containing powder was poured from all sides. The container with the samples was subjected to induction heating in the air atmosphere at the given electrophysical parameters (power, inductor current) at temperatures of 1000, 1100, 1200, and 1300 °C, with an exposure time of 4 min. After processing, the chemical elemental composition of the coatings was investigated using energy dispersive X-ray fluorescence analysis (EDX), the surface morphology was studied by scanning electron microscopy (SEM), and mechanical properties (microhardness) – by the Vickers method.
[5,6]. The hardness was assessed with two loads on the indenter, a low load (20 gf) made it possible to determine the hardness of the coating and a large load (100 gf) was used to determine the hardness of the underlying layer (the effect of the tantalum base).

3. Results
The chemical composition of the surfaces of tantalum samples after CTT was represented by tantalum, carbon and oxygen. The amount of tantalum in the coatings decreased as the treatment temperature grew from 29 to 23 at.% (Figure 1). The amount of carbon increased from 39 to 61 at.% at a temperature of 1200 °C; with a further growth of temperature, the percentage of carbon reached 58 at.%. The amount of oxygen was minimal (11 at.%) at a temperature of 1200 °C, an increase and decrease in the CTT temperature led to a higher percentage of oxygen.

Figure 1. Effect of CTT temperature on the change in the amount of tantalum (a), carbon (b) and oxygen (c) in sample coatings

The surface morphology after CTT had significant changes – coatings with nanosized particles were formed on the surface. The minimum processing temperature ensured the formation of a uniform coating, while the scratches less than 1 µm wide were visible resulting from grinding the tantalum base. The obtained coating had single pores not more than 2 µm in size and nanosized particles in the form of single protrusions 10–20 nm in size (Figure 2a). Raising the temperature to 1100 °C in the course of carburization led to the formation of a coating with an increased number of surface pores with the size up to 3 µm and a decrease in the number of small particles (Figure 2b). In some areas, structural elements with a size of not more than 3 µm were observed. Figure 2c shows the surface morphology of a tantalum sample after CTT at 1200 °C. In this case the nanostructure was similar to the first sample but the particle size increased; there were also areas with pores up to 4 µm in size. The
most defective structure is shown in Figure 2d. At a given CTT temperature, pores occupied a significant part of the surface. The particles had a wide spread in size, and their minimum size equaled about 100 nm.

Figure 2. Surface morphology of tantalum samples after CTT at processing temperatures of 1000 °C (a), 1100 °C (b), 1200 °C (c), and 1300 °C (d)

The initial hardness of the tantalum samples was 140±7 HV. The summarized measurement results were presented graphically (Figure 3). The obtained hardness curves showed that at the highest CTT temperature (1300 °C) coatings with a high hardness of 1100±64 HV_{0.02} were formed. At this temperature, the hardness of the base increased significantly to 740±57 HV_{0.01}. A decrease in the process temperature to 1200 °C led to lower hardness of the coating (900±48 HV_{0.02}) and the base (522±70 HV_{0.02}).
Figure 3. Effect of the CTT treatment temperature on the change in surface hardness of tantalum samples at HV$_{0.02}$ (a) and HV$_{0.1}$ (b)

Hard coating and soft base were observed at temperatures close to the minimum, where the difference between the values of the coating and the base equaled 650 HV. The minimum CTT temperature (1000 ºC) contributed to the formation of a coating with a low hardness of 611±64 HV$_{0.02}$. In this case, the hardness of the base was close in value to that of the coating and amounted to 569±59 HV$_{0.1}$.

4. Conclusions
Thus, in the course of the work, it was found that as a result of CTT, three-component coatings were formed consisting of tantalum, carbon and oxygen. The structure of the resulting coatings contained nanograins, the size of which grew with an increase in the temperature of the CTT process. The hardness of the coatings had the highest value at the maximum temperature T = 1300 ºC.

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