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Abstract

Full Text

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RECRYSTALLIZATION DIAGRAM OF TANTALUM

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The high corrosion resistance of tantalum in aggressive media, its refractory nature, high ductility, which makes it possible to subject it to cold plastic deformation with large degrees of reduction while retaining sufficient strength, and a number of other properties place tantalum among the metals very important for technology. As is known, a very substantial factor affecting the mechanical properties of metals and alloys is the grain size; therefore, the construction of a recrystallization diagram relating the grain size to the degree of deformation and to the temperature of subsequent annealing is especially necessary for metals used mainly in the form of articles obtained by deformation (sheets, foil, rods, tubes, wire, etc.). The data obtained make it possible to select deformation and annealing conditions that ensure optimal mechanical properties of the articles. A recrystallization diagram of tantalum has not yet been published. The literature contains only some data on the recrystallization of cold-worked tantalum, referring to tantalum obtained by the powder-metallurgical method ([1–3]). There are no data in the literature on the recrystallization of tantalum obtained by the casting method.

Fig. 1. Recrystallization diagram of tantalum

Until now, the principal method for obtaining compact tantalum, as well as other refractory metals (tungsten, niobium, molybdenum), has been powder metallurgy. Recently, methods of arc melting in vacuum or in protective media have been widely developed for such refractory and readily oxidized metals as titanium, zirconium, molybdenum, and others. Arc melting of molybdenum, and especially of its alloys, in most cases is already replacing the production of powder-metallurgical rods. Therefore it was of great interest to study the recrystallization of tantalum,

To the article by E. M. Savitskii, M. A. Tylkina, and I. A. Tsygankov, p. 720

Figure 3

Figure 2: Figure 3

Figure 1

Figure 3: Figure 1

Fig. 3. X-ray diffraction patterns of tantalum specimens. **a** –specimen deformed by 34%; **b** –specimen deformed by 34%, annealed at 1275°.

To the article by S. A. Buivailo and F. Z. Meerson, p. 823

Fig. 1. Myocardium 2 days after creation of aortic stenosis: glycogen is absent in the swollen muscle fibers.

Figure 2 Figure 3

Fig. 2. Myocardium 3 months after creation of aortic stenosis: a considerable amount of glycogen in the protoplasm of the muscle fibers.

Fig. 3. Myocardium 2 days after creation of aortic stenosis: fatty degeneration of the muscle fibers.

obtained by the casting method. We constructed a recrystallization diagram of type I for cold deformation, by rolling, of cast tantalum (Fig. 1). The casting of the specimens was carried out in an arc furnace in argon at a pressure of 200 mm Hg, from metalloceramic tantalum foil of 99.8% purity.

The conditions of cooling the metal on a copper hearth promote the formation of a coarse-grained structure (Fig. 2a).

The cast ingots were subjected to cold deformation by forging until rods with a cross section of 7×7 mm were obtained; these were annealed in vacuum at 1300° for 2 h. As a result of this treatment the coarse-crystalline structure of the cast metal was completely destroyed. The rods possessed a recrystallized, fine-grained, polyhedral structure with a grain diameter of 10—11 μ (Fig. 2b) and served as the starting material for the entire work.

The deformation of the initial rods was carried out by cold rolling without intermediate anneals, with 13 degrees of reduction: 2.6; 5.7; 8; 10; 15; 34; 50; 68; 83; 90; 96; 98; 6%. The deformed rods were cut into specimens 8–10 mm long and annealed in vacuum in the interval 1000—2500° with a holding time of 1 h. Grain counting was performed with an object micrometer on the transverse section of the polished specimen at the intersection of the diagonals, which corresponds to the region of maximum deformation. The temperature of the onset of recrystallization was determined by microstructural and X-ray methods, from the appearance of the first spots on the diffraction rings. The X-ray patterns were taken in Cu radiation from a polished specimen that had first been subjected to deep etching in order to remove the surface layer.

The line of the onset of recrystallization of tantalum as a function of the degree of deformation is plotted on the recrystallization diagram as a dashed line (Fig. 1).

The temperature of the onset of recrystallization of tantalum decreases as the degree of deformation increases, from 1300 to 1200°. At a deformation of 2.6% the temperature of the onset of recrystallization is 1300°; at 10–34%, 1275°; at 50–68% it falls to 1250°; and at 84% and above it reaches 1200°. Some X-ray patterns of tantalum are shown in Fig. 3.

Fig. 2. Microstructures of tantalum (200×): *a*—cast structure, *b*—initial structure after forging, annealed after forging for 2 h at 1300°, deformation by 67.6%, annealed at *e*—1 h, 1300°, *d*—cold-rolled; *z*—deformed by 67.6%, annealed at 1800°, 1 h; *b*—deformed by 67.6%, annealed at 2000°, 1 h; *e*—deformed by 67.6%, annealed at 2500°, 1 h.

Cold rolling of tantalum at small degrees of deformation (up to 15%) leads to distortion of its lattice and deformation of individual grains, but does not introduce substantial changes into its microstructure. At deformation with a reduction of more than 30%, a clearly expressed rolling texture is observed (Fig. 2 c). The high ductility of tantalum causes a sharp change in the shape of individual grains and their elongation without fragmentation up to a reduction of ~50-60%; with greater deformation, the grains are crushed. At 90% deformation the grain diameter is 1-2 μ. Annealing at 1000-1600° does not cause noticeable grain growth. The recrystallization process during treatment at 1200° in specimens with a high degree of deformation, and at 1600° at all degrees of deformation, leads to the complete destruction of the rolling texture and to the appearance of new fine recrystallized grains with a diameter on the order of 13-6 μ. A sharp change in grain size, associated with the occurrence of the process of coalescence recrystallization, takes place during annealing at 1800 and 2000° (Fig. 2 d, e). The size of the initial grain, equal at 1000-1600° to approximately 10 μ, increases threefold at 1800° (to 31 μ) and tenfold at 2000° (to 115 μ). At different degrees of deformation, considerable grain growth is also observed. On the annealing isotherms at 1800 and 2000°, maxima of grain size are observed, corresponding to the critical degrees of deformation. At 1800° the maximum corresponds to 8% deformation and shifts toward a lower degree of deformation (5.7%) as the annealing temperature is raised to 2000°. On the 2500° isotherm the maximum grain size approaches very small degrees of deformation. An interesting fact, apparently representing a specific feature of tantalum, may be considered the appearance of annealing twins at 2500° at all degrees of deformation (Fig. 2 f) and the exceptional grain growth at this temperature, changing their size by several orders of magnitude. The size of the initial grain then reaches approximately 500 μ, while the grain size at 83% deformation is 320 μ. We are continuing the study of recrystallization at large degrees of deformation during annealing at 2500°. Investigation of the effect of the degree of deformation and annealing temperature on the strength and ductility of tantalum in tension (on small Gagarin-type specimens with a diameter of 3 mm) and on

hardness showed that tantalum has optimum properties after annealing at 1300°. (Properties of cold-deformed tantalum: $\sigma_B = 95$ kg/mm^2, $\delta = 5\%$; $\psi = 45\%$; after annealing at 1300°: $\sigma_B = 60$ kg/mm^2, $\delta = 22\%$; $\psi = 58\%$.) Raising the annealing temperature to 1600° and higher leads to an increase in hardness and ultimate tensile strength, with a corresponding decrease in elongation. This phenomenon is associated with the ability of tantalum to absorb gases especially intensively in the range 1600–1800° and with its strengthening owing to the formation of solid solutions with oxygen ([3-5]). Therefore, the preservation of high ductility at an annealing temperature of about 1600° is possible only under conditions of a very high vacuum. On the basis of the data obtained, it may be considered that the optimum annealing regime, ensuring the production of a fine-grained polyhedral structure and high mechanical properties of tantalum, is a temperature on the order of 1300–1400°.

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Note: Figure translations are in progress. See original paper for figures.