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PROPERTIES OF NIOBIUM ALLOY 5VMTs AFTER DEFORMATION AND CHEMICAL HEAT TREATMENT

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It is known that the temperature of the beginning of recrystallization of niobium alloys is increased by introducing refractory metals with atomic sizes close to that of niobium. The addition of nitrogen can also increase the recrystallization temperature of niobium alloys. The temperature threshold of niobium recrystallization depends on the production process, namely, the degree of deformation in cold treatment, the deformation temperature, the initial grain size, the regime of the subsequent heat treatment, etc. However, only a few works have been devoted to the joint effect of deformation, nitriding, and heat treatment on the recrystallization of industrial niobium alloys. The present paper concerns determination of the temperature threshold of recrystallization of industrial niobium alloy 5VMTs after chemical heat treatment and estimation of the relaxation strength of arch springs fabricated from this alloy.

The effect of alloying on the temperature of the beginning of recrystallization of niobium alloys is considered in [1, 2], and the production process is described in [3].

We studied two batches of flat specimens 15 × 15 × 1 mm in size of alloy 5VMTs (5% W, 2% Mo, 1% Zr, the remainder Nb). The specimens were cut from sheets deformed with a degree $\varepsilon = 25$, 50, and 70%.

The first batch of deformed specimens was annealed in vacuum at $133 \times (10^{-5} - 10^{-6})$ Pa by the following regimes: (*1*) 1000°C for 2 h; (*2*) 1200°C for 2 h; (*3*) 1300°C for 1.5 h; (*4*) 1400°C for 1.5 h; (*5*) 1500°C for 1 h; (*6*) 1700°C for 1 h; (*7*) 1800°C for 1 h; (*8*) 2000°C for 1 h.

We measured the microhardness of specimens deformed and annealed by the indicated regimes. They were also subjected to tensile tests with determination of σ_r and δ after deformation with $\varepsilon = 70\%$ and annealing by all the regimes.

The second batch of deformed specimens was subjected to chemical heat treatment (CHT) by a regime involving saturation with nitrogen in a helium-ammonia medium (85% He and 15% NH_3 at a pressure of 10^5 Pa) at 800 °C for 10 h followed by vacuum annealing conduced in two stages, namely, 1000°C for 1 h + 1200°C for 4 h. In order to study the recrystallization process in the alloy the specimens were annealed after the CHT for 1 h at 1000, 1200, 1300, 1450, and 1500°C in quartz ampoules (preliminarily evacuated to 6.65×10^{-3} Pa) and at 1700, 1800, and 2000°C in a high-temperature furnace at a residual pressure in the chamber equal to 6.65×10^{-4} Pa.

Analyzing the mechanical properties of the alloy subjected to deformation and annealing (Table 1), we established that the recrystallization temperature had a range of 1400 – 1500°C for deformations with $\varepsilon = 25 - 50\%$. The strength and ductility of the alloy deformed with $\varepsilon = 70\%$ changed considerably at 1300 – 1400°C.

Specimens of alloy 5VMTs from the second batch (after CHT) were also subjected to mechanical tests with measure-

TABLE 1

t_{an}, °C	τ_{an}, h	Microhardness *H*. Degree of preliminary deformation, %			σ_r, N/mm^2	δ, %
		25	50	70		
Initial state*		210	225	235	625	6.6
1000	2.0	205	220	220	620	14.0
1200	2.0	200	185	200	615	15.0
1300	1.5	195	185	185	605	18.0
1400	1.0	165	180	165	450	25.2
1500	1.0	165	165	155	420	26.7
1700	1.0	150	145	130	415	25.2
1800	1.0	150	140	115	335	26.0
2000	1.0	140	140	105	300	27.1

* After deformation.

Notes. 1. Values of medians of a variational series of 15 measurements of the microhardness and mean values of the mechanical properties after 3 – 5 tensile tests at room temperature are presented. The error of measurement of the mechanical properties does not exceed ± 1.5%. 2. The specimens used in the mechanical tests were deformed preliminarily with $\varepsilon = 70\%$.

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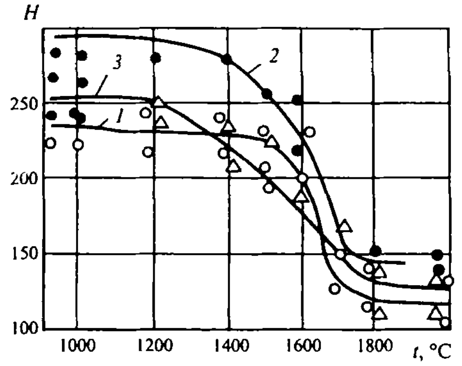

Fig. 1. Microhardness of alloy 5VMTs as a function of the temperature of annealing conducted after deformation with different degrees and CHT: *1*) ε = 25%; *2*) ε = 50%; *3*) ε = 70%.

ment of their microhardness. In addition, the electrical conductivity was measured on wire specimens (∅ 1 × 200 mm) with analysis of their x-ray diffraction patterns. Results of the study of alloy 5VMTs after the CHT are presented in Figs. 1 – 3.

The dislocation density was calculated by the method of [4] on the basis of determination of the physical half-width of interference lines (211) by the formula

$$N = k\, b_0 \cot \theta_{hkl},$$

where k is a coefficient (for niobium and its alloys $k = 1.65 \times 10^{12}$), b_0 is the physical half-width of the line.

Specimens 10 × 10 × 1 mm in size were subjected to an x-ray diffraction analysis using annealed sheet specimens as a standard. The micrographs were obtained in cobalt K_α radiation (BSV-22 x-ray tube) with rotation of the specimens. The working voltage was 40 kV, and the tube current was 20 mA. The working slits were 0.5 × 6.0 × 0.25 with a primary Soller's slit. The photography was pointwise with an accumulation regime of 10 sec, using an automatic scintillation counter for recording.

The x-ray diffraction analysis has shown that after nitriding at 800°C for 10 h (compared with annealing) the crystal lattice constant increased from 0.3315 to 0.3330 nm and the dislocation density increased from 3×10^{15} to 1.5×10^{16} m^{-2} (i.e., by an order of magnitude). As a rule, in strain hardening of high-strength alloys it attains 10^{14} m^{-2} [5]. A higher dislocation density (about 10^{17} m^{-2}) can cause formation of cracks and fracture of the metal.

The change in the microstructure and the growth of the recrystallization temperature of alloy 5VMTs after nitriding show that the disperse particles formed in the process of CHT impede the loss of strength in the metal. Analyzing the results obtained, we established that the temperature of the beginning of recrystallization in alloy 5VMTs increased by 300°C after the CHT and attained $t_{\text{b.r.}} = 1750 - 1880$°C.

Using the data of the present study we plotted a recrystallization diagram (Fig. 4) of a nitrided alloy and took it into account in choosing the regime for chemical heat treatment.

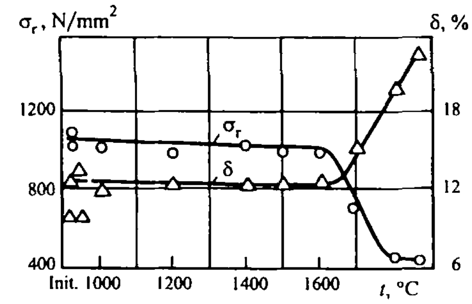

Fig. 2. Dependences of the ultimate rupture strength σ_r and elongation δ of alloy 5VMTs on the temperature of annealing conducted after deformation with ε = 70% and CHT.

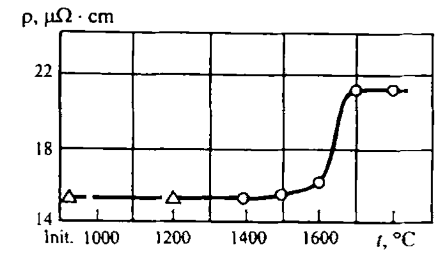

Fig. 3. Electrical conductivity ρ of alloy 5VMTs as a function of the temperature of annealing conducted after deformation with degree ε = 70% and CHT.

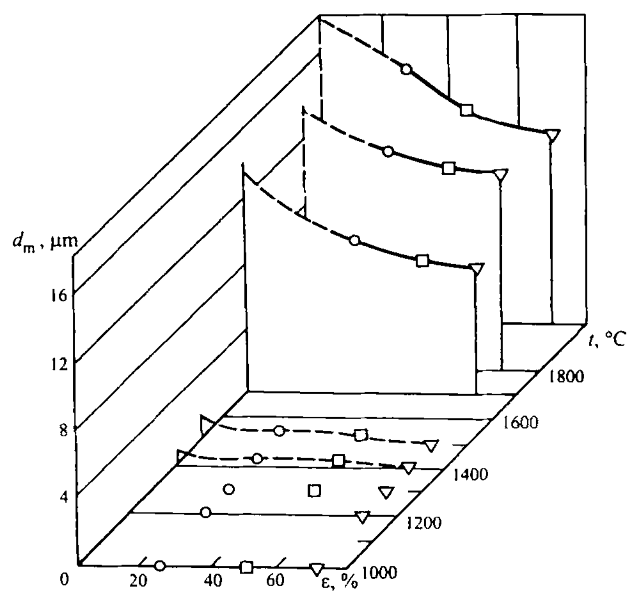

Fig. 4. Recrystallization diagram for alloy 5VMTs after CHT (d_m is the mean grain size, ε is the degree of preliminary deformation, t is the temperature of vacuum annealing).

A phase analysis of specimens of alloy 5VMTs subjected to CHT by various regimes has shown the presence of niobium nitrides (NbN, Nb_2N) and zirconium nitride (ZrN). The strained metal did not contain nitride phases (Table 2). After nitriding of strained specimens their surface contained a small amount of niobium nitrides of various modifications. In

TABLE 2

Treatment regime	N_m, %	Phases
Deformation with ε = 50% (initial)	0.005	Not found / Not found
Nitriding at 800°C for 10 h	0.030	Nb_2N; NbN (sm); Nb[C, N] (tr) / Not found
Nitriding at 800°C for 10 h + two vacuum annealings at 1000°C for 1 h	0.024	NbN; Zr[NO](sm) / ZrN; Nb_2Zr_6[NO] (sm)
Nitriding at 800°C for 10 h + two vacuum annealings at 1000°C for 1 h + three vacuum annealings at 1250°C for 4 h	0.23	ZrN; NbZr[ON](tr) / NbN (tr); ZrN; ZrO_2 (tr)

Notation: sm) in a small amount; tr) traces.
Note. The numerators present phases determined on the surface of the specimen (at a distance $h = 30-50$ μm), the denominators give phases in the center of the specimen ($h = 500$ μm).

the central part of the specimens nitrogen was present in the solid solution.

After the first vacuum annealing niobium nitride and a certain amount of zirconium nitride were retained in surface layers up to 50 μm thick. The center of the specimens contained zirconium nitride and traces of complex compounds of niobium and zirconium with nitrogen that were difficult to identify exactly.

After the second vacuum annealing zirconium nitrides of cubic modification were observed over the entire cross section of the specimens.

To check the efficiency of the CHT, we studied the relaxation strength of arch springs 0.3 × 5.8 × 3.0 mm in size of alloy 5MVTs nitrided at 700°C for 10 h and annealed in vacuum at 1000°C for 1 h.

To evaluate the relaxation strength the springs were kept at 20°C for 70 h and then stabilized at 800°C for 5, 10, 15, and 20 h. The temperature and time parameters of the stabilizing treatment were determined from the optimum values of the degree of relaxation. The degree of relaxation R (%) was calculated by the formula

$$R = \frac{H_0 - H_f}{H_0} \times 100\%,$$

where H_0 and H_f are the initial and final heights of the spring flexure in mm.

Below we present results of evaluation of the degree of relaxation R.

State	R, %
Kept at 20°C for 70 h	6.6
Stabilization at 800°C for (h):	
1	7.8
5	15.7
10	0
15	0
20	0

It can be seen that the maximum degree of relaxation corresponds to a 5-h hold at 800°C. After a 10-h hold at the same temperature the spring does not relax.

CONCLUSIONS

1. The recrystallization temperature of industrial niobium alloy 5VMTs is 1400°C after deformation and it is 1750°C after a chemical heat treatment (nitriding).

2. Nitriding of strained alloy 5VMTs increases the temperature threshold of recrystallization owing to reinforcing of the grain boundaries by disperse particles.

3. Evaluation of the relaxation strength of arch springs of alloy 5MVTs after CHT at an operational temperature of 800°C has shown their high operating properties.

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