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# THE EFFECT OF TEMPERATURE ON MECHANICAL CHARACTERISTICS OF NIOBIUM ALLOYS OF THE SYSTEM Nb-W-Mo-Zr

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Data obtained for the short-term strength and ductility of thin sheet, weld metal, and welded joints of niobium alloys 5VMTs and 10VMTs of the sysiemNb-W-Mo-Zr in the temperature range 290 - 2 270 K are analyzed. The structure, deformation mechanisms governing plastic deformation, and the nature of alloy failure in the temperature range are determined.

***Key words:*** *niobium alloys, effect of temperature, mechanical characteristics*

**Utjecaj temperature na mehanička svojstva slitina niobija u sustavu Nb-W-Mo-Zr.** Analizirani su podaci dobiveni za kratkoročnu čvrstoću i plastičnost tankog lima, zavarnog metala, zavarenih spojeva slitina niobija 5VMTs i 10VMTs u sistemu Nb-W-Mo-Zr u području temperature od 290 - 2 270 K. Određeni su mehanizmi strukture deformacije koji upravljaju plastičnom deformacijom i priroda grešaka u slitini u tom području temperature.

***Ključne riječi:*** *slitine niobija, utjecaj temperature, mehanička svojstva*

## INTRODUCTION

Metal structural materials based on niobium are distinguished by high elastic and strength properties which are very promising for preparing welded articles for critical purposes intended for high-temperature operation. Alloys 5VMTs and 10VMTs of the Nb-W-Mo-Zr system are of the most extensively used niobium alloys and they relate to low and medium-alloys of niobium, respectively, with solid solution strengthening. Due to the optimum content of alloying elements they conveniently combine high strength indices with low-temperature ductility and technological efficiency during welding and machining [1-3].

Data obtained by experiment for the short-term strength and ductility of semi-finished product and welded joints of niobium alloys 5VMTs and 10VMTs over a wide temperature (290 - 2 270 K) are analyzed in this work.

## MATERIAL, TREATMENT, AND TESTING

The chemical composition of niobium alloys is as follows: for the 5VMTs (TU 48-4-310-72), wt. %: 4.5 - 5.5 W, 1.7 - 2.3 Mo, 0.7 - 1.15 Zr, 0.02 C, 0.01 $O_2$, 0.015 $N_2$, for the l0 VMTs (TU48-0533-108-84), wt %:10 - 12 W, 1.7 - 2.2 Mo, 0.5 - 1.0 Zr, 0.02 C, 0.02 $O_2$, 0.02 $N_2$.

The semifinished product was prepared by vacuum-arc melting followed by forging and stepwise hot deformation into sheet 1 - 2 mm thick and a recrystallizing anneal at 1 670 K for 2 h in the concluding preparation stage. Welding was performed by the arc method in a controlled helium atmosphere at a rate of 0.28 cm/sec which corresponded to a running energy of 2.35 and 3.87 kJ/cm for sheet 1.0 and 2.0 cm thick.

In the as-supplied condition sheets of alloys 5VMTs and 10TVMTs are isotropic. The material structure is fine-grained with equiaxial polyhedral grains with a size of 25 - 50 μm. Welded joints have solid solution strengthening manifested as a cellular structure typical for niobium alloys (Figure 1.)

The-mechanical properties were determined from the results of testing fiat five-fold proportional specimens with a gauge length of 12 and 15 mm in high-temperature devices 1246-R and VTU-2V in a vacuum not worse than 0.1 Pa [4, 5]. The deformation rate was 2 mm/min which corresponded to a relative strain rate of about $2.2x10^{-3}$ $s^{-1}$.

For the basic 5VMTs and 10VMTs material, for which the most representative selections of tests results for specimens from industrial melts were obtained, statistical treatment of the experimental data was performed. The average sample value (mathematical expectation) $\overline{x}$ , the mean

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square sample deviation (MSSD), $S_x$ probability ranges for mathematical expectation, and mean square deviation of the mechanical properties for a significance level $\alpha$ = 0.05, were calculated. The lower guaranteed ultimate strength and ductility characteristics $x_l$ were determined as the lower tolerance limit found for a probabi1ity level $\gamma$ = 0.99 and probability $p = 1 - \alpha = 0.95$ [6].

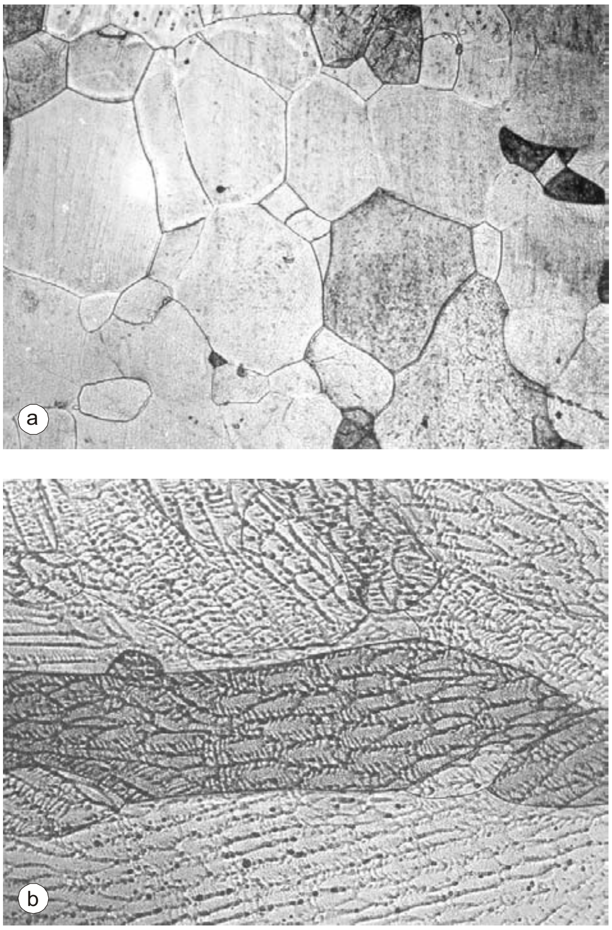


Figure 1. **Microstructure of niobium alloy 10VMTs: a-base metal; b - welded joint. Optical microscopy. Magnification: a - x400, b - x200**

Slika 1. **Mikrostruktura slitine niobija 10VMTs; a - osnovni metal; b - zavareni spoj. Optički mikroskop, povećanje; a - 400x, b - 200x**

Alloy structure was studied by optical, transmission, and scanning electron microscopy, and also the Laue x-ray method.

Table 1. **Mechanical Properties of Rolled Sheet, Welded Joint Metal, and Welded Joints of Niobium Alloy 5VMTs in a Wide Temperature Range**

Tablica 1. **Mehanička svojstva valjanog lima, zavarenog metalnog spoja i zavarenih spojeva slitine niobija 5VMTs u širokom području temperature**

| $T$ [ K ] | Sheet $\delta$ = 1.0 (annealing at 1 637 K for 2 h) | | | |
|---|---|---|---|---|
| | $R_m$ [MPa] | $R_{p0.2}$ [MPa] | $A$ [%] | $A_{un}$ [%] |
| 290 | 445 | 320 | 29.0 | 14.5 |
| 520 | 330 | 245 | 32.0 | 21.0 |
| 770 | 275 | 180 | 15.5 | 5.0 |
| 1020 | 300 | 200 | 17.0 | 8.5 |
| 1270 | 240 | 170 | 18.5 | 8.0 |
| 1520 | 120 | 105 | 50.0 | 3.5 |
| 1770 | 65 | 60 | 77.0 | 1.8 |
| 2020 | 37 | 35 | 86.0 | 1.6 |
| 2270 | 18 | 18 | 117.5 | 0.3 |
| | Welded joint metal | | | |
| 290 | 545 | 435 | 24.0 | 11.5 |
| 520 | 395 | 290 | 22.5 | 7.5 |
| 770 | 365 | 265 | 17.5 | 5.8 |
| 1020 | 395 | 260 | 17.8 | 7.5 |
| 1270 | 350 | 275 | 11.5 | 5.0 |
| 1520 | 195 | 175 | 21.2 | 2.5 |
| 1770 | 90 | 80 | 45.5 | 9.5 |
| 2020 | 41 | 40 | 60.5 | 0.4 |
| 2270 | 20 | 20 | 160.5 | 0.2 |
| | Welded joint | | | |
| | | Failure zone | | |
| 290 | 410 | Joint | | |
| 520 | 330 | Joint | | |
| 770 | 295 | Joint | | |
| 1020 | 300 | Joint | | |
| 1270 | 275 | Joint, fusuion zone | | |
| 1520 | 155 | Joint, basic metal | | |
| 1770 | 76 | Basic metal | | |
| 2020 | 42 | Basic metal | | |
| 2270 | 20 | Basic metal | | |

## RESULTS AND DISCUSION

Typical deformation curves and temperature dependence of the mechanical properties obtained for alloys 5VMTs and 10VMTs (ultimate strength $R_m$ nominal yield strength $R_{p0.2}$, relative elongation A, and relative uniform deformation $A_u$ taking account of the statistical parameters of scatter are provided in Tables 1., 2, and in Figure 2. - 4.

The limits for the probability ranges of mathematical expectation of the ultimate strength and relative elongation of the alloy are shown by lines in Figure 3., 4., and the temperature dependences for the lower guaranteed limit of mechanical properties are shown by broken lines.

As for the majority of niobium alloys, 5VMTs and 10VMTs are quite ductile both in the recrystallized condition and in the cast condition over almost the whole test temperature range. The nature of the deformation curve for the alloys depends markedly on the structural condition of the material and test temperature. At 290 - 770 K in the initial sections of the curve zones are observed like a yield-

Table 2. **Mechanical Properties of Rolled Sheet, Welded Joint Metal, and Welded Joints of Niobium Alloy 10VMTs in a Wide Temperature Range**

Tablica 2. **Mehanička svojstva valjanog lima, zavarenih metalnih spojeva i zavarenih spojeva slitine niobija 10VMTs u širokom rasponu temperature**

| *T* [ K ] | Sheet δ = 1.0-1.7 (annealing at 1 637 K for 2 h) | | | |
|---|---|---|---|---|
| | $R_m$ [MPa] | $R_{p0.2}$ [MPa] | *A* [%] | $A_{un}$ [%] |
| 290 | 565 | 425 | 34.0 | 15.8 |
| 520 | 465 | 315 | 33.8 | 14.8 |
| 770 | 345 | 243 | 31.4 | 14.0 |
| 1020 | 385 | 223 | 20.8 | 11.5 |
| 1270 | 290 | 180 | 24.5 | 10.3 |
| 1520 | 175 | 150 | 44.5 | 5.2 |
| 1770 | 96 | 89 | 68.0 | 2.4 |
| 2020 | 62 | 60 | 74.0 | 0.9 |
| 2270 | 38.4 | 36 | 100.0 | 0.7 |
| | Welded joint metal | | | |
| 290 | 550 | 420 | 11.0 | 11.0 |
| 520 | 527 | 395 | 15.0 | 9.5 |
| 770 | 455 | 335 | 28.0 | 10.0 |
| 1020 | 470 | 320 | 24.0 | 9.5 |
| 1270 | 365 | 285 | 5.5 | 3.5 |
| 1520 | 200 | 175 | 14.5 | 5.5 |
| 1770 | 105 | 100 | 40.0 | 8.0 |
| 2020 | 60 | 59 | 31.0 | 0.5 |
| 2270 | 40 | 40 | 109.5 | 0.4 |
| | Welded joint | | | |
| | | Failure zone | | |
| 290 | 560 | Basic metal, HAZ | | |
| 520 | 430 | Weld, HAZ | | |
| 770 | 300 | Basic metal, weld | | |
| 1020 | 395 | Basic metal, HAZ | | |
| 1270 | 350 | Basic metal, weld | | |
| 1520 | 200 | Weld | | |
| 1770 | 93 | Basic metal, weld | | |
| 2020 | 56 | Basic metal, weld | | |
| 2270 | 34 | Basic metal, weld | | |

ing platform or tooth. In the range 770 - 1 270 K for both alloys there is dynamic strain aging (DSA) typical for the majority of niobium alloys and connected with blocking of the mobile dislocations by impurity atom atmospheres [7, 8]. In this temperature range the deformation curves have a specific wavy nature pointing to nonuniform occurrence of plastic deformation processes. Here there is a marked reduction in ductility characteristics and an increase in the strength indices for the alloys (Figure 2.).

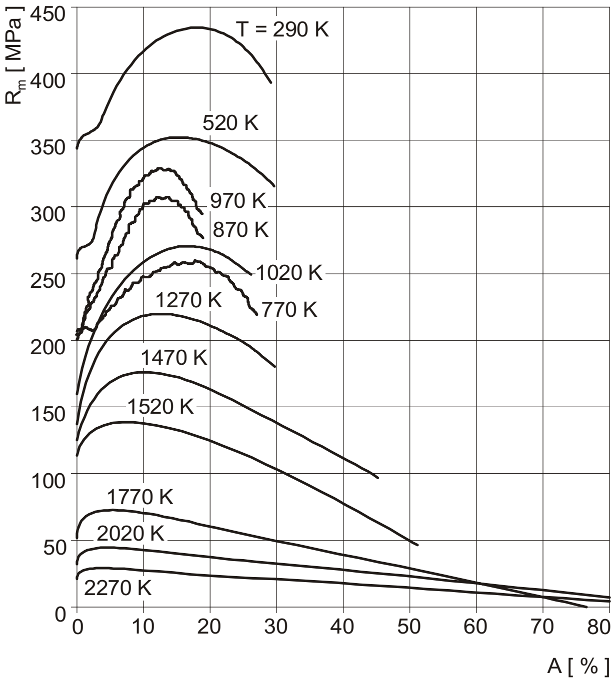


Figure 2. **Typical deformation curves for niobium alloy 5VMTs in the range of temperatures 290 - 2270 K**

Slika 2. **Tipične krivulje deformacije slitine niobija 5VMTs u području temperature od 290 - 2 270 K**

On deformation curves for cast metal of a welded joint for niobium alloy 10VMTs at room temperature there is no descending section which indicates that specimen failure occurs in the stage of necking without local plastic deformation with a relative reduction of area close to zero.

For both niobium alloys in the temperature range 290 - 770 K there is typically uniform loss of strength accompanied by some reduction in relative elongation and relative uniform strain for the basic material, and a marked, by more than a factor of two, increase in ductility index for cast metal of the welded joint for alloy 10VMTs (Tables 1. and 2., and Figure 3., 4.). At 770 - 1270 K there are extremes on the temperature dependences for strength properties connected with development of strain aging effects followed by a sharp reduction in the strength index in the range 1 270 - 1 700 K as a result of polygonization are recrystallization. With a further increase in temperature in the range 1 770 - 2 270 K alloy loss of strength is again uniform in nature.

The relative elongation of alloys 5VMTs and 10VMTs at room temperature is 29.0 and 34 %, and at 2 270 K it is 117.5 and 100.0 %, respectively. The minimum ductility properties are observed in the temperature range of dynamic strain aging: at 1 020 K the relative elongation of the niobium alloys is 17.0 - 20.8 %, and the relative uniform strain is 8.5 - 11.0 %.

The least scattering of mechanical properties occurs in the range 290 - 1 270 K, and the greatest occurs at 2 020 K. There is an extreme in the temperature dependence for the lower guaranteed limit of relative elongation for alloy 10VMTs at 2 020 K characterizing, as at 1 020 K, a minimum in material ductility (Figure 4.). An increase in the scatter of mechanical properties in the high-temperature region is probably connected with physicochemical transformations in the material, in particular with precipitation of particles of a second phase along grain boundaries [3, 9].

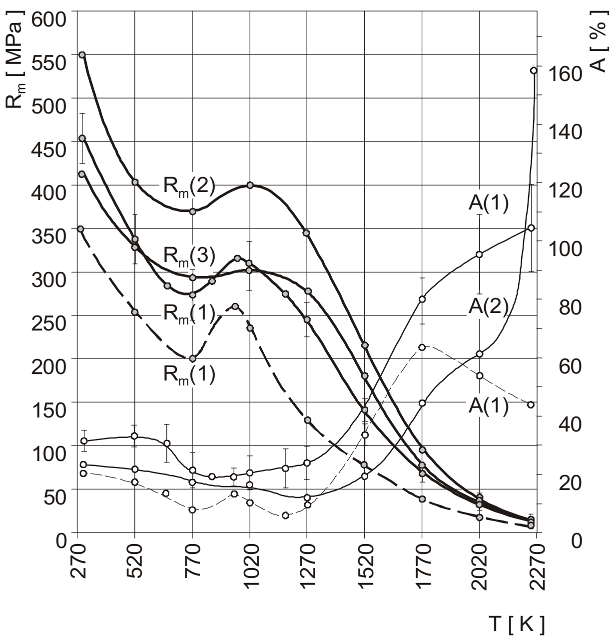


Figure 3. **Temperature dependence for the ultimate strength (shaded points) and relative elongation (open points) for niobium alloy - 5VMTs: 1) basic metal (sheet 1.0 mm thick, annealing at 1 670 K for 2 h), 2) weld metal, 3) welded joint**

Slika 3. **Ovisnost vlačne čvrstoće (zasjenjene točke) i relativnog istezanja (otvorene točke) osnovnog metala slitine niobija 5VMTs: 1) osnovni metal (lim 1.0 mm debljine, žarenje na 1 670 K tijekom 2 sata), 2) metal za zavarivanje, 3) zavareni spoj**

As a rule in the test temperature range welded joints of niobium alloys 5VMTs and 10VMTs are of equal strength with sheet material. In the range 290 - 1 270 K they are equally probable to fail both through the basic metal and through the weld, and also in the heat-affected zone (HAZ). At 1 520 K joint failure occurs through the weld, at 1 770 K it is predominantly through the basic metal, and only at $T > 0.5T_m$ (in the range 2 020 - 2 270 K it only fails through the basic material.

The strength properties of cast metal of a welded joint of niobium alloys in the range 290 - 1 770 K exceeds the similar index for the basic metal and welded joints by 20 - 50 %. At room temperature and in the high-temperature range (2 020 - 2 070 K) there is almost no difference in the strength of cast, recrystallized material, and welded joints.

The ductility properties of the cast metal of a welded joint of alloys 5VMTs and 10VMTs are markedly lower than for the basic material. At room temperature its relative elongation for alloy 10VMTs is 11 %, and at 2 270 K it is 110 %. The minimum relative elongation (5.5 %) is observed at 1 270 K.

Temperature dependences for the mechanical properties of alloys in the cast and recrystallized conditions are qualitatively similar. However, a minimum connected with development of dynamic strain aging effects shifts on the temperature dependence for relative elongation into a higher temperature region by about 250 K. Here the extreme in the region of 2 020 K characterizing high-temperature embrittlement of alloy 10VMTs is clearly expressed for cast material (Figure 4.).

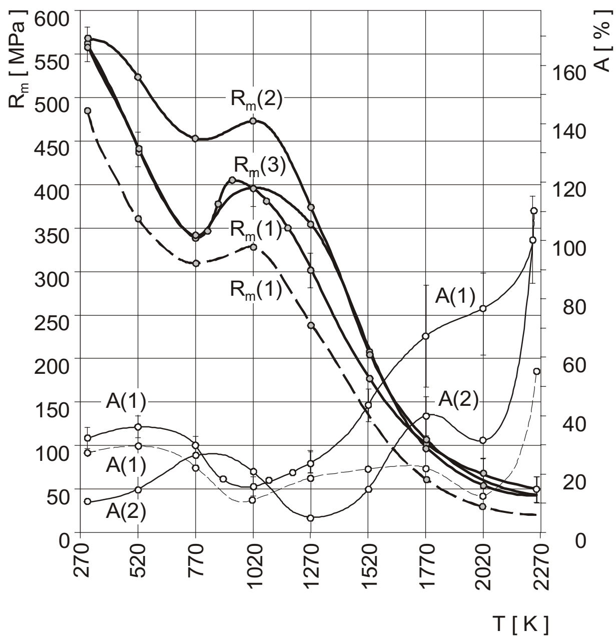


Figure 4. **Temperature dependence for the ultimate strength (shaded points) and relative elongation (open points) for niobium alloy - 10VMTs: 1) basic metal (sheet 1-2 mm thick, annealing at 1 670 K for 2 h), 2) weld metal, 3) welded joint**

Slika 4. **Ovisnost vlačne čvrstoće (zasjenjene točke) i relativnog istezanja (otvorene točke) za slitinu niobija 10VMTs: 1) osnovni metal (lim debljine 1 - 2 mm, žarenje na 1 670 K tijekom 2 sata), 2) metal za zavarivanje, 3) zavareni spoj**

It is characteristic that the strength properties of medium-alloy 10VMTs, alloyed with 10 - 12 wt. % tungsten, are substantially better over the whole studied temperature range than analogous indices of the low-alloy of niobium 5VMTs, alloyed with only 4.5 - 5.5 wt. % tungsten (Table 1., 2.).

Structural studies showed that in the temperature range from room to 1 270 K plastic deformation of niobium alloys is accomplished predominantly as a result of uniform or nonuniform singular or multiple slip (Figure 5.a). How-

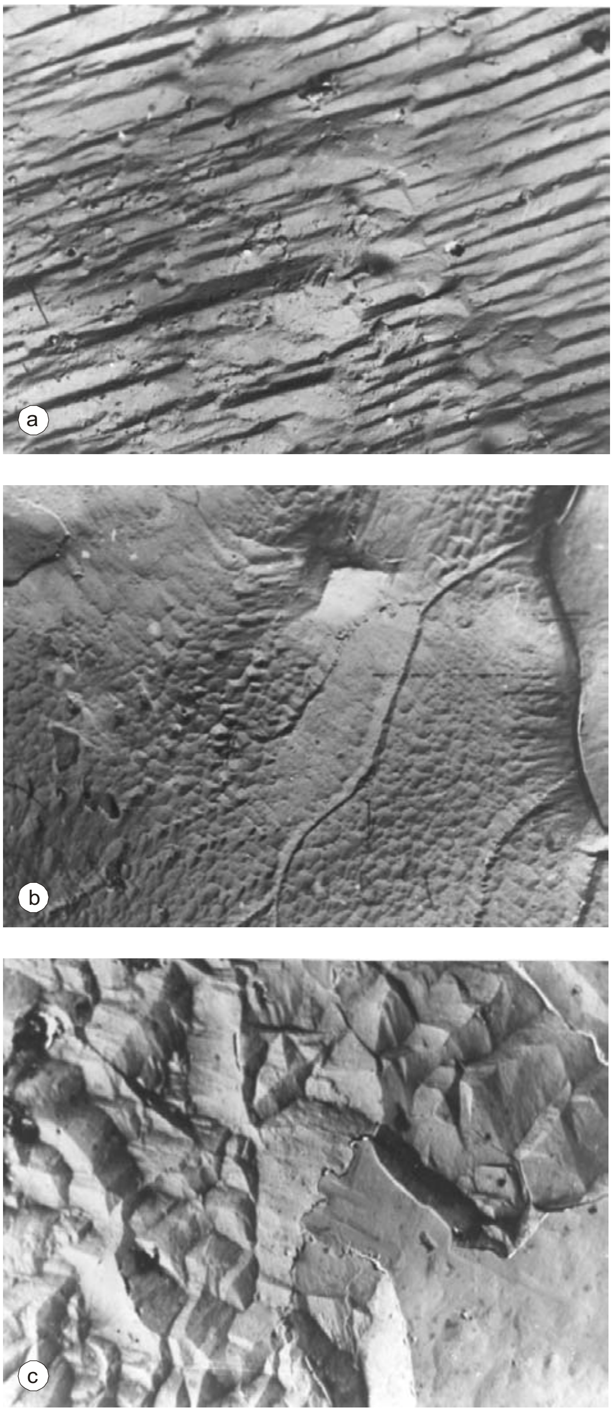

Figure 5. **Microstructure of niobium alloy 10VMTs after mechanical testing at the temperatures: a-770 K, b-1520 K, c-1770 K. Transmission electron microscopy. Magnification: a, b-x3000, c-x7000**

Slika 5. **Mikrostruktura slitine niobija 10VMTs nakon mehaničkog ispitivanja na temperaturama: a - 770 K, b - 1 520 K, c - 1 770 K. Snimano elektronskim mikroskopom; uvećanje a, b - 3000x, c - 7000x**

ever in individual cases there is grain-boundary slip. With an increase in temperature the number of areas with multiple slip and grain-boundary slip increases, and in the temperature range 1 570 - 1 720 K the grain-boundary slip mechanism predominates (Figure 5.c).

The start of dynanuc recrystallization of the test alloy is observed at temperatures above 1 670 K. After mechanical tests at 1 770 K the material has a subgranular structure with traces of slip in individual blocks with a size of 5 - 6 μm within a grain.

Testing at 2 020 - 2 270 K is accompanied by secondary recrystallization of the alloy and formation of a fine-grained substructure with traces of uniform singular and multiple slip in individual grains (Figure 5.c).

The nature of failure for niobium alloys depends on structural state of the material an the test temperature. For recrystallized sheet material dhere is typically intergranular failure over almost the whole of the test temperature range. For cast metal of a welded joint at room temperature there is brittle failure through the body of grains. With an increase in test temperature to 520 - 1 020 K the nature of failure of welded joints changes into ductile predominantly along grain boundaries.

At 1 270 K the failure of welded joints is mixed in nature. In fractures apart from areas with ductile finely-cupped separation along the boundaries of crystals there are facets of brittle transcrystalline cleavage with a developed grooved gap. With a further increase in temperature up to 2 270 K for welded joints of alloys 5VMTs and 10VMTs ductile failure is typical. At 2 020 K particles of a second phase of rounded shape are observed in a fracture which are supposed from qualitative X-ray analysis to be zirconium oxide [9, 10].

## CONCLUSIONS

1. The properties obtained for the short-term strength and ductility of sheet material, weld metal, and welded joints of alloys 5VMTs and 10VMTs of the system Nb-W-Mo-Zr in the temperature range 290 - 2 270 K are obtained taking account of the statistical parameters of experimental data scatter. It is shown that over almost all of the whole test temperature range welded joints of the alloy are equal in strength to die basic material.
2. Plastic deformation of niobium alloys in the temperature range 290 - 2 270 K is realized predominantly by intragranular singular and multiple slip, and also grain-boundary sliding. Dynamic recrystallization in die alloy occurs at 1 670 K.
3. The nature of failure for alloys 5VMTs and 10VMTs depends on the structural state of the material and the test temperature, and it may vary in die test temperature range depending on such factor as brittle failure through the body of a grain to ductile inter- and intracrystalline failure.

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