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THE EFFECT OF THERMAL TREATMENT ON THE MECHANICAL PROPERTIES OF LOW-ALLOYED MOLYBDENUM ALLOYS OVER THE WIDE RANGE OF TEMPERATURES

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The article presents data on the short-time strength and ductility of thin rolled sheet made of low-alloy molybdenum TsM-10 of the system Mo-Al-B, TsM-6 of the system Mo-Zr-B, and TsM-12 of the system Mo-Zr-Hf-B in deformed, polygonized, and recrystalized states in the temperature range 290 - 2 270 K. It was established that heat treatment can increase the high-temperature strength of these alloys by a factor of 1.2 - 2.2 and 1.1 - 1.5, respectively.

Key words: *low - alloyed molybdenum alloys, thermal treatment, mechanical properties*

Utjecaj toplinske obradbe na mehanička svojstva niskolegiranih slitina molibdena u širokom temperaturnom području. Članak daje podatke o vlačnoj čvrstoći i plastičnosti tankovaljanog lima napravljenog od niskolegiranog molibdena TsM-10 u sistemu Mo-Al-B, TsM-6 u sistemu Mo-Zr-B i TsM-12 u sistemu Mo-Zr-Hf-B u deformiranom, poligoniziranom i rekristaliziranom stanju u temperaturnom području 290 - 2 270 K. Utvrđeno je da toplinski tretman može povećati vlačnu čvrstoću na visokoj temperaturi svih slitina za faktor 1.2 - 2.2 i 1.1 - 2.5.

Ključne riječi: *niskolegirane slitine molibdena, toplinska obradba, mehanička svojstva*

INTRODUCTION

Structures operating at high temperatures and exposed to aggressive gaseous media are made of low-alloy molybdenum TsM-10 of the system Mo-Al-B, TsM-6 of the system Mo-Zr-B and TsM-12 of the system Mo-Zr-Hf-B [1, 2]. Alloy TsM-10 is commercially pure molybdenum deoxidized by aluminium and boron. Alloy TsM-6 is alloyed with up to 0.2 wt. % zirconium and TsM-12, is alloyed with up to 0.03 wt. % zirconium and 0.09 wt. % hafnium belongs to the heat-resistant molybdenum alloys with solid-solution hardening. The mechanical characteristics of molybdenum alloys depend on the structural state of the material and on the technological factors determining it [3-5].

In particular, in materials of this class after thermo mechanical treatment in certain regimes and after welding, particles of the second phase may form which have a substantial influence on the strength and ductile characteristics of the material [3, 6].

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The present communication contains experimental data on the short-time strength and ductility of semi product sheet of the molybdenum alloys TsM-l0, TsM-6 and TsM-12 in different structural states and a wide temperature range (from 290 - to 2 270 K).

MATERIALS, THEIR TREATMENT AND TESTING

The semi products of the alloys were obtained by vacuum-arc melting with subsequent high-temperature stepped forging and rolling to sheet 1 - 2 mm thick, with reduction of 80 - 90 % . At the concluding stage of the technological process sheers of alloys TsM-6, TsM-12 and TsM-10 were annealed at 1 420 and 1 220 K respectively, for 1 h for relieving internal stresses, and then they were chemically etched for removing scale and the dirty surface layer.

The mechanical characteristics were determined from the results with flat fivefold proportional specimens with 12 and 15 mm long working part, on high-temperature installations 1246-R and VTU-2V in a vacuum not poorer than 0.1 Pa, by a method described in [7]. The strain rate was 2 mm/min which corresponded to the relative strain rate $2.2 x 10^{-3}$ s^{-1}. The specimens were cut out in the direction of the technological deformation of the sheet. Heat treatment was carried out in a vacuum not poorer than 0.1 Pa at 1 570 and 1 770 K with alloy TsM-6, at 1 570, 1 770, 1 870 and 2 270 K with alloy TsM-12, and 1 420, 1 970 and 2 170 K with alloy TsM-10 for 1 h.

In the state as supplied the macrostructure of the investigated alloys are fibers drawn in the direction of deformation. The dislocation structure is cellular with the cells about 2 μm and moderate dislocation density.

After annealing at 1 570 K for 1 h the macrostructure of the alloys, TSM-6 and TsM-12 barely changes. However, as a result of polygonization the size of the dislocation cells increases to 4 - 6 μm. Annealing at 1 770 K is accompanied by recrystallization of the material of this alloys with the grain size increasing to 40 - 50 μm. In alloy TsM-12 annealing at 1 870 and 2 270 K for 1 h increases the grain size to 70 and 300 μm, respectively.

In alloy TsM-10 after annealing at 1 420 K for 1 h the process of primary recrystallization is completed, and polyhedral grains up to 10 μm in size form. Annealing at 1 970 and 2 120 K for 1 h increases the grain size to 200 and 300 μm, respectively, on account of accumulative and secondary recrystallization [5].

For the alloy TsM-10, for which in the state as supplied the most representative samples were obtained according to test results with specimens of three industrial melts, the experimentally determined mechanical characteristics were statistically processed. This involved the calculation of the mean value (the mathematical expectation) $\bar{x}$, the sample standard deviation (SD), S_x the confidence intervals for the mathematical expectation and the standard deviation at the significance level $\alpha = 0.05$. The lower guaranteed limit of the strength and ductile characteristics x_l was determined as the lower tolerance limit for the confidence level $\gamma = 0.99$ and probability level $P = 1 - \alpha = 0.95$ [8].

RESULTS AND DISCUSSION

The obtained mechanical characteristics of the molybdenum alloys (ultimate strength R_m conventional yield strength $R_{p0.2}$ relative elongation A, and relative uniform deformation A_{un} with the statistical parameters of dispersion taken info account) are presented in Tables 1. - 3. and Figures 1. - 3. The drawings also show the temperature dependences of the strength and ductile characteristics of the cast metal of the seam and of welded joints of the alloys TsM-6, TsM-12 and TsM-10 plotted according to previously obtained experimental data [9].

An analysis of the temperature dependences of the mechanical characteristics of the investigated materials in the deformed, recrystallized, and cast states showed that they are qualitatively similar for both molybdenum alloys. As a rule, the change of strength and ductile characteristics of the alloys TsM-6, TsM-12 and TsM-10 with rising temperature is of a nonmonotonic nature. When the temperature rises from room temperature to 520 - 770 K, the strength of the material decreases greatly. The strength indices of the alloys decrease by one third to one half. In the range 520 - 1 520 K, which corresponds to the homologous temperatures ~ (0.2 - 0.5) T_{pl} the loss of strength proceeds with less intensity, and then intensity increases again in the range 1 520 - 2 270 K (~(0.5 - 0.8) T_{pl}).

The temperature dependences of the ultimate strength, and especially of the conventional yield strength of the alloys in the recrystallized and cast states, in the range 1 070 - 1 770 K (~(0.4 - 0.6) T_{pl}) have extreme connected with processes of dynamic strain-aging, which manifest them-

Table 1. **Mechanical Characteristics of the Molybdenum Alloy TsM-10 in the Range 290 - 2 270 K**

Tablica 1. **Mehanička svojstva slitine molibdena TsM-10 u rasponu 290 - 2 270 K**

T [K]	Annealing at 1 220 K, 1 h				Annealing at 1 420 K, 1 h			
	R_m [MPa]	$R_{p0.2}$ [MPa]	A [%]	A_{un} [%]	R_m [MPa]	$R_{p0.2}$ [MPa]	A [%]	A_{un} [%]
290	800	730	19.0	6.8	607	563	29.8	16.6
470	-	-	-	-	418	350	25.6	22.4
520	625	555	9.5	4.3	-	-	-	-
670	-	-	-	-	341	210	57.1	22.5
770	595	535	7.0	2.4	-	-	-	-
870	-	-	-	-	297	200	36.9	21.6
1020	530	480	7.8	1.5	-	-	-	-
1070	-	-	-	-	250	185	38.4	20.4
1270	440	410	9.5	1.3	182	104	40.2	21.3
1470	-	-	-	-	143	87	37.5	16.5
1520	160	68	36.0	15.3	140	75	37.5	5.5
1770	73	40	52.3	10.4	68	46	54.5	13.5
2020	33	18	46.5	7.7	45	33	41.0	8.7
2270	8.2	4.6	58.3	6.2	13	8	68.0	6.0
	Annealing at 1 970 K, 1 h				Annealing at 2 120 K, 1 h			
290	481	372	10.4	9.8	360	342	1.2	1.2
470	276	195	44.2	24.7	215	102	37.6	21.4
520	-	-	-	-	-	-	-	-
670	198	120	50.6	38.6	174	56	41.2	25.7
770	-	-	-	-	-	-	-	-
870	181	84	42.5	35.2	148	48	38.4	27.9
1020	-	-	-	-	-	-	-	-
1070	175	62	37.9	30.6	132	42	32.8	28.2
1270	164	51	30.4	29.2	112	34	29.4	27.5
1470	141	54	22.7	20.1	98	36	29.1	28.1
1520	93	64	16.5	10.0	95	49	23.4	7.9
1770	54	42	15.0	7.0	66	46	13.0	5.7
2020	42	33	52.0	10.0	41	29	25.8	9.8
2270	14	9	72.0	7.0	15	10	43.8	2.5

selves in the blocking of motion of dislocations by interstitial impurity atoms [10]. At that the strength characteristics of the alloys increase somewhat, and the ductile characteristics decrease (Tables 1. -3., Figures 1. - 3).

Table 2. **Mechanical Characteristics of the Molybdenum Alloy TsM-6 in the Range 290 - 2 270 K**
Tablica 2. **Mehanička svojstva slitine molibdena TsM-6 u području temperature od 290 - 2 270 K**

T [K]	Annealing at 1 420 K for 1 h			
	R_m [MPa]	$R_{p0.2}$ [MPa]	A [%]	A_{un} [%]
290	830	755	24.5	9.5
520	660	585	16.0	4.7
770	275	520	14.0	2.8
1020	560	440	9.0	1.6
1270	465	390	5.0	0.8
1520	340	300	16.0	1.5
1770	120	85	40.5	11.0
2020	60	50	54.5	6.0
2270	29	24	61.5	6.0
	Annealing at 1 570 K for 1 h			
290	750	685	27.5	12.5
520	570	440	20.5	9.0
770	510	450	18.0	6.4
1020	435	395	14.5	3.6
1270	400	370	13.0	2.6
1520	355	340	15.5	1.5
1770	165	120	24.0	8.5
2020	70	55	51.5	7.0
2270	38	32	65.0	6.8
	Annealing at 1 770 K for 1 h			
290	570	460	43.0	16.5
520	445	195	56.0	34.5
770	325	145	49.0	34.5
1020	270	105	48.0	29.5
1270	245	135	39.0	21.5
1520	180	115	31.0	7.0
1770	125	90	33.0	10.5
2020	75	60	44.0	8.0
2270	43	39	55.5	5.5

The strength of alloys increases by rolling, and consequently the differences in the strength properties of deformed and recrystallized material are retained up to 1520-1770 K.

Heat treatment and the attendant change of structure of the material have an ambiguous influence on the mechanical properties of molybdenum alloys, and in dependence on the test temperature they may either enhance or impair the strength and ductile characteristics.

Table 3. **Mechanical Characteristics of the Molybdenum Alloy TsM-12 in the Range 290 - 2 270 K**
Tablica 3. **Mehanička svojstva slitine molibdena TsM-12 u području temperature od 290 - 2 270 K**

T [K]	Annealing at 1 420 K for 1 h *			
	R_m [MPa]	$R_{p0.2}$ [MPa]	A [%]	A_{un} [%]
290	770	730	25.0	10.0
520	650	565	19.0	7.5
770	585	535	13.5	4.5
1020	495	465	10.5	2.5
1270	425	425	2.7	0.8
1520	440	350	4.5	0.7
1770	360	150	24.0	1.2
2020	86	78	35.0	2.2
2270	40	34	35.0	4.8
	Annealing at 1 570 K for 1 h			
290	780	750	29.0	14.0
520	675	570	21.0	10.0
770	590	520	16.0	7.0
1020	510	480	12.0	3.0
1270	450	430	6.0	1.5
1520	360	350	5.0	1.0
1770	160	150	19.5	1.5
2020	82	75	37.0	2.5
2270	42	36	32.0	4.0
	Annealing at 1 770 K for 1 h			
290	710	580	29.0	11.0
520	615	450	32.0	15.0
770	485	315	30.0	17.0
1020	420	340	26.0	12.0
1270	310	250	16.0	6.5
1520	235	210	9.0	1.5
1770	155	130	15.0	2.0
2020	100	90	22.0	2.2
2270	51	48	43.0	2.5
	Annealing at 1 870 K for 1 h			
290	620	445	34.5	12.5
520	525	300	42.5	24.5
770	430	285	31.5	16.0
1020	390	270	29.0	14.5
1270	285	215	24.5	11.5
1520	235	190	13.0	2.3
1770	150	135	12.5	1.9
2020	90	80	19.5	2.0
2270	47	41	30.5	5.5
	Annealing at 2 770 K for 1 h			
290	470	325	7.0	7.0
520	380	255	12.5	7.5
770	305	115	39.0	30.5
1020	260	85	37.0	22.5
1270	165	100	36.5	22.5
1520	130	85	34.5	14.5
1770	90	70	34.5	10.0
2020	60	50	37.5	9.0
2270	37	29	39.5	8.5

* State as supplied

For instance, in the range from 290 to 1 520 - 1 770K the conventional yield strength and the ultimate strength of the deformed alloys TsM-10, TsM-6 and TsM-12 with cellular dislocation structure are considerably higher than the analogous indices of the polygonized, recrystallized,

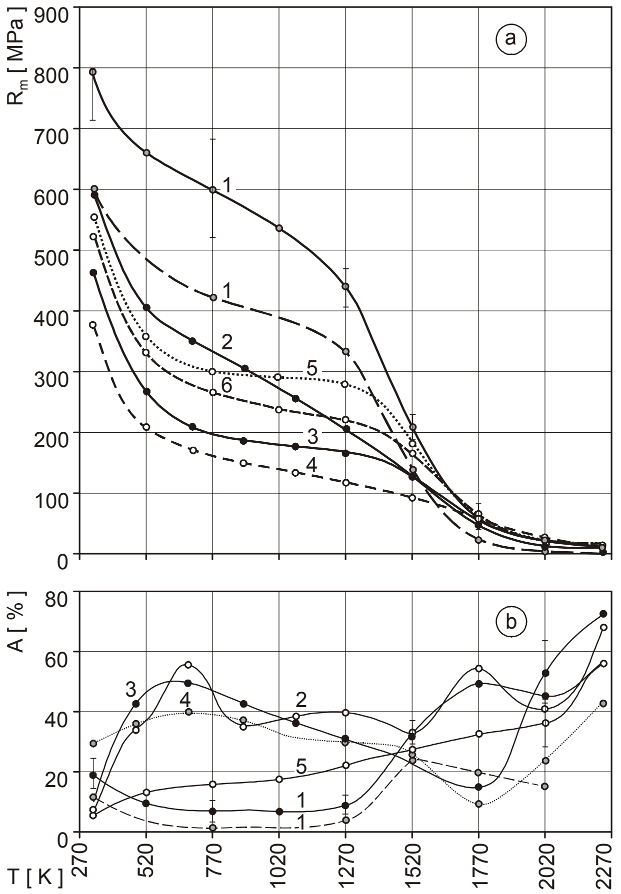

Figure 1. **Temperature dependences of ultimate strength (a) and of relative elongation (b) of the molybdenum alloy TsM-10: 1) annealing at 1 220 K for 1 h, 2) the same at 1 420K, 3) the same at 1 920 K, 4) the same at 2 120 K, 5) metal of the seam, 6) welded joint (1 - 4 denote the base metal). (Primes denote the confidence limits for the mathematical expectation of ultimate strength and relative elongation of the alloy TsM-10; the dashed lines are the temperature dependences of the lower guaranteed limit of the mechanical characteristics.)**

Slika 1. **Temperaturna ovisnost vlačne čvrstoće (a), relativne istezljivosti (b), molibdenove slitine TsM-10 o temperaturi: 1) nakon žarenja na 1 220 K tijekom 1 sata, 2) nakon žarenja na 1 420 K tijekom 1 sata, 3) nakon žarenja na 1 920 K tijekom 1 sata; 4) nakon žarenja na 2 120 K tijekom 1 sata; 5) metala zavara, 6) zavarenog spoja (1 - 4 se odnosi na osnovni metal), prim brojevi označavaju granice pouzdanosti za matematička očekivanja vlačne čvrstoće i relativne istezljivosti slitine TsM-10; isprekidane crte predstavljaju temperaturne ovisnosti donje granične crte mehaničkih karakteristika**

and cast materials. The strength characteristics of the alloys decrease monotonically with higher annealing temperature and the attendant increase of the grain size. And the differences between the mechanical properties of deformed, recrystallized, and cast materials decrease, the higher the test temperature is. The obtained experimental results are in good agreement with the results of investigations carried out with materials of the given class [4, 11].

In the high-temperature we find the opposite picture. At 1 770 - 2 270 K heat treatment enhances the strength characteristics of the alloys TsM-6, TsM-12 and TsM-10 by a factor of 1.1 - 1.5 and 1.2 - 2.2, respectively. The strength of the molybdenum-tungsten alloy increases regularly with rising annealing temperature and test temperature in the investigated range. However, annealing of alloy TsM-12 at 2 270 K for 1 h results in a significant softening of the material over the whole studied temperature range (Table 1. - 3., Figures 1.a, 2.a, 3.a).

Analogous results concerning the influence of recrystallization annealing on the strength of a molybdenum-tungsten alloy of the system Mo-30% W-NbC -C in the range 290 - 2 270 K were obtained earlier on by the authors of [12].

It is characteristic that the strength characteristics of the heat-resistant alloys, TsM-6 alloyed with up to 0.2 wt. % zirconium and TsM-12, alloyed with up to 0.03 wt. % zirconium and 0.09 wt. % hafnium, respectively, are substantially better in the high-temperature range than the analogous indices of the alloy TsM-10 which in chemical composition is very close to commercially pure molybdenum.

With higher temperature the characteristics of ductility of the molybdenum alloys also change nonmonotonically (Figures 1.b, 2.b and 3.b). At room temperature the ductility of deformed material is fairly good because of the low temperature of cold shortness in this stage. In the range of elevated and medium temperatures (520 - 1 270 K) the relative elongation of the deformed alloys TsM-6, TsM-12 and TsM-10 decreases from 25.0 and 19.0 % to 2.5 - 5.0 and 7.0 %, respectively; this, however, is not a consequence of embrittlement but is connected with the poor strain-hardenability of the material. In fact, in tensile tests the strength of the metal, which is intensely cold-worked during rolling, increases only slightly, and on account of that the deformation of the specimen is rather unstable, and a neck forms at a comparatively early stage of deformation [4].

At temperatures above 1 270 K, which corresponds to ~ $0.5T_{pl}$, the ductility of rolled sheet greatly increases on account of the thermally activated processes of polygonization and recrystallization of the material. In the high-temperature range the relative elongation of the alloy TsM-6 increases monotonically up to 2 270 K. On the temperature dependence of commercially pure molybdenum, the alloy TsM-10, we find that ductility somewhat decreases in the region of 2 020 K. High-temperature embrittlement of molybdenum is usually ascribed to the segregation of carbides type Mo_2C along grain boundaries, these carbides being thermodynamically stable in the range 1 970 - 2 370 K, and to the change of the nature of failure from ductile transcrystalline to intercrystalline [3, 13].

Heat treatment of molybdenum alloys in dependence on the annealing conditions may either improve their characteristics of ductility at room and elevated temperatures, or it may lead to embrittlement of the material. Polygonization annealing of the alloys TsM-6 and TsM-12 at 1 570 K for 1 h increases its relative elongation and relative uniform deformation almost in the entire investigated temperature range without changing the nature of their temperature dependence.

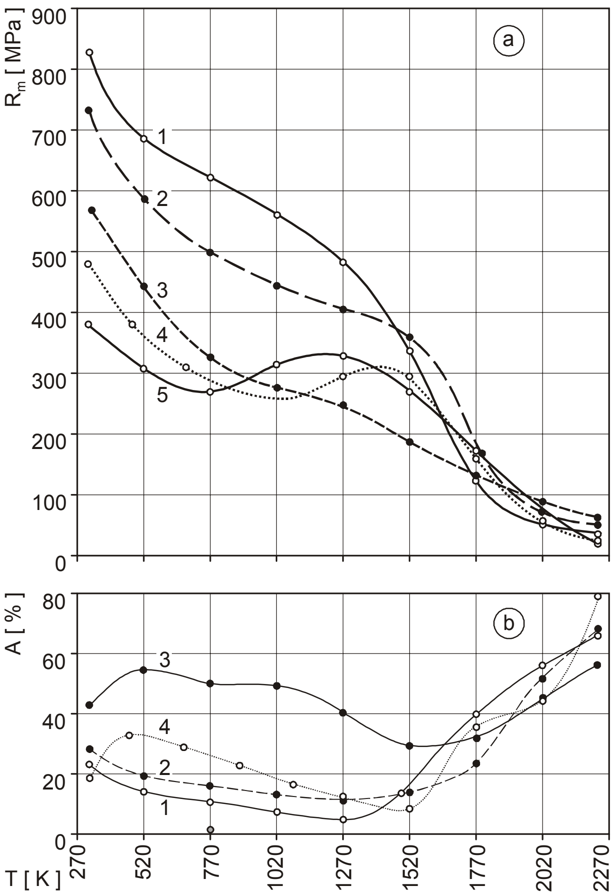

Figure 2. **Temperature dependences of ultimate strength (a) and of relative elongation of the alloy TsM-6: 1) annealing at 1420 K for 1 h, 2) the same at 1 570 K, 3) the same at 1 770 K, 4) metal of the seam; 5) welded joint, (1-3 denote the base metal)**

Slika 2. **Temperatuma ovisnost vlačne čvrstoće (a), relativne istezljivosti slitine TsM-6: 1) žarenje na 1 420 K tijekom 1 sata, 2) žarenje na 1 570 K tijekom 1 sata, 3) žarenje na 1770 K tijekom 1 sata, 4) metal zavara, 5) zavareni spoj (1-3 odnosi se na osnovni metal)**

Recrystallization of the alloys TsM-6, TsM-12 and TsM-10 after annealing (at 1 770 and 1 420 K, respectively) is accompanied by very substantial improvement of their ductile characteristics in the range from room temperature to 1 520 - 1 670 K and has little effect on the ductility of the se materials in the high-temperature range. Recrystallization annealing of molybdenum alloys at higher temperatures, as well as the cast state of the metal of the welding seam cause embrittlement of the material at room temperature [5, 9]. The temperature dependence of the relative elongation in the interval from 290 to 770 K has an extremum connected with the transition of the material into the ductile state, and the minimum discovered in the high-temperature range for the alloy TsM-10 is shifted toward the temperature of 1 770 K (Figures 1.b, 2.b, 3.b).

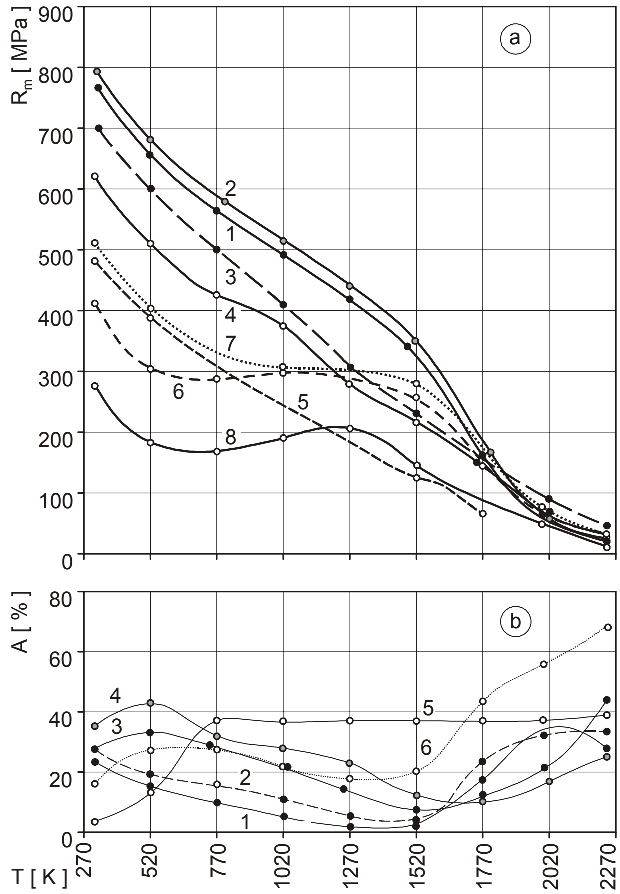

Figure 3. **Temperature dependences of ultimate strength (a) and of relative elongation of the alloy TsM-12: 1) annealing at 1 420 K for 1 h, 2) the same at 1 570 K, 3) the same at 1 770 K, 4) the same at 1 870 K, 5) the same at 2 770 K, 6) metal of the seam; 7, 8) welded joint (7 - state after welding, 8 annealing at 1 770 K, 1 h), (1-6 denote the base metal)**

Slika 3. **Temperaturna ovisnost vlačne čvrstoće (a), relativne istezljivosti (b), slitine TsM-12: 1) žarenje na 1 420 K tijekom 1 sata, 2) žarenje na 1 570 K tijekom 1 sata, 3) žarenje na 1 770 K tijekom 1 sata, 4) žarenje na 1 870 K tijekom 1 sata, 5) žarenje na 2 270 K tijekom 1 sata, 6) metal zavara 7), 8) zavareni spoj (7 - stanje nakon zavarivanja, 8 - žarenje na 1 770 K, tijekom 1 sata, (1 - 6 odnose se na osnovni metal)**

The nature of the temperature dependences of short-time strength and ductility of the molybdenum alloys is determined by physicochemical transformations that took place in the process of thermo mechanical treatment, by annealing and welding, and also by heating and by the applied external load in mechanical tests. The mechanical characteristics of the material are affected first and foremost by the processes of recrystallization and aging, and also by the content and specific traits of the distribution of interstitial impurities in the body and along the boundaries of grains [3-6, 11, 14].

It is known that high-temperature annealing under stress leads to more rapid recovery and recrystallization of deformed material than annealing without stress because of the faster migration of grain sub boundaries and boundaries. And as a role, the change of structure of material is accompanied by an impairment of its strength characteristics.

On the other hand, recrystallization of the molybdenum alloys entails a reduction of the area of the internal boundaries, and this gives rise to the appearance of segregation pile-ups of interstitial impurity atoms along grain boundaries, and in the limit case also to the segregation of the second phases in near-boundary regions, which may inhibit processes of accumulative recrystallization and enhance the strength of the material.

In the general case, when material is subjected to mechanical loading at high temperature, there occur two processes simultaneously: deformation and recrystallization. The superposition of these processes may lead either to enhanced or to impaired strength of the material [4, 15].

Since the characteristics of high-temperature long-time strength of low-alloy molybdenum are necessarily impaired with increasing ordering of the structure [16], the discovered increase of short-time strength of the alloys TsM-6 and TsM-10 in the polygonized and recrystallized states compared with the deformed state at temperatures above $0.6T_{pl}$ is due in the first place to specific traits of the dynamic recrystallization of bcc metals under conditions of active static extension and their influence on the strength indices of the material [17 - 20].

CONCLUSIONS

1. As a result of the investigation of the characteristics of short-time strength and ductility of rolled sheet of the molybdenum alloys TsM-10 of the system Mo-Al-B and TsM-6 of the system Mo-Zr-B in the deformed, polygonized, and recrystallized states it was established that heat treatment enhances the high-temperature strength of these materials by a factor of 1.2 - 2.2 and 1.1 - 1.5, respectively.

2. Annealing at 1 420 -1 770 K for 1 h can be recommended for improving the characteristics of short-time strength of molybdenum alloys when they are to be used in components and structural elements whose operating conditions are characterized by temperatures higher than $0.5T_{pl}$ and if their service life does not exceed several minutes.

3. Annealing of molybdenum alloys at t he temperature above 1 870 K would be objectionable because it causes a loss of plasticity and a reduction of the material strength characteristic over the whole studied temperature range.

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