

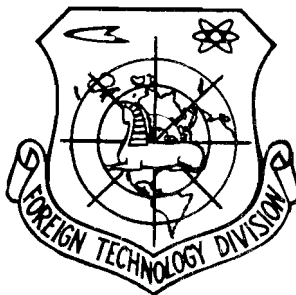
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A STUDY OF THE PHASE DIAGRAM FOR THE Mo-Zr-C SYSTEM
IN A REGION RICH IN MOLYBDENUM

A. M. Zakharov and Ye. M. Savitskiy



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EDITED TRANSLATION

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ABSTRACT

(U) ^{Mo} A series of Mo-Zr-C ^{addition} alloys containing 0-0.55 wt. percent C, 0-6.0 wt. percent Zr, and about 0.011 percent O, 0.008 percent N and 0.0006 percent H impurities, were melted from single crystal molybdenum, 99.94 percent - pure Zr, lamp black and spectrally pure zirconium carbide (ZrC) in a nonconsumable-electrode arc furnace in a helium atmosphere. The alloys were investigated by various methods of physicochemical and phase analyses in the as-cast condition and also after vacuum annealing and quenching from temperatures ranging from 1970 to 1250 degrees C. On the basis of the results obtained, isotherms were plotted for the solidus surface of molybdenum alpha-solid solution in the ternary Mo-Zr-C system, and also isothermal sections at 1970 and 1250 degrees C, and a polythermal Mo-Zr-C section. L. V. Kustova participated in the work. Orig. art. has: 5 figures and 2 tables.

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A STUDY OF THE PHASE DIAGRAM FOR THE Mo-Zr-C SYSTEM IN A REGION RICH IN MOLYBDENUM

A.M. Zakharov and Ye.M. Savitskiy (Moscow)

Recent investigations of the high-temperature strength of molybdenum alloyed with small additions of Ti, Zr and C have shown [1, 2] that the tensile properties of these alloys are considerably increased if the alloys are subjected to strengthening heat treatment (hardening and subsequent artificial tempering). The increase in the high-temperature strength of Mo-Ti-C, Mo-Zr-C and Mo-Ti-Zr-C alloys after hardening and tempering is achieved through decomposition of the supersaturated molybdenum α -solid solution and separation from it of excess phase-strengtheners of the carbide (mainly TiC and ZrC) [3, 4] and evidently nitride [5] and oxide [6] type.

In this connection a study of the phase diagrams for molybdenum with group IV α metals and C and, in particular, of the triple system Mo-Zr-C which can serve as a basis for a search for new high-temperature heat hardenable molybdenum alloys.

There are several articles in the literature devoted to a study of the diagram of state of the triple system Mo-Zr-C. In Reference [7] isothermal sections of this system have been plotted at temperatures of 2100°C (in the Mo-ZrC-C region) and 1500°C (in the Mo-ZrC-Zr region) and in Reference [8] - the isothermal section of the whole system at a temperature of 1400°C. The results of a study of the reaction of the carbide ZrC with molybdenum have been published in Reference [9]. It has been established that the Mo-ZrC section in the triple system Mo-Zr-C is quasi-binary and has a diagram of the eutectic type; the eutectic α +ZrC(γ) (where α and γ are solid solutions in a base of Mo and the carbide ZrC, respectively) contains¹ about 34 wt. % of ZrC and melts at a temperature of 2250°C. It follows from the data of Reference [7-9] that the carbide ZrC (γ) possesses limited solubility in molybdenum in the solid state; however, this solubility, like the joint solubility of Zr and C in Mo, at other Zr/C ratios remains unstudied.

The results of a study² of the molybdenum angle of the triple system Mo-Zr-C and, in particular, of the joint solubility of Zr and C in Mo in the solid state are set forth in the present article. The alloys studied contained 0-0.5% C and 0-6% Zr. The principal methods of studying the triple system Mo-Zr-C were: a microscopic analysis of cast alloys and alloys hardened after prolonged annealing from temperatures of 1970 and 1250°C, the "drop" method for determining the temperature of the beginning of melting of the

alloys, x-ray phase and structural analyses and the microhardness method.

Method of work. The following were used as the charging materials for the preparation of the alloys: monocrystalline molybdenum, obtained by electron beam zone refining (0.0008% N, 0.0017% O and 0.0004% H), 99.94% pure Zr iodide, lamp black and the carbide ZrC prepared from spectrally pure elements. Alloy samples weighing 50g were prepared in an arc furnace with an unconsumable (tungsten) electrode in a copper water-cooled sole in an atmosphere of purified helium (pressure 350 mm Hg column). To obtain uniform composition the ingots were turned over the recast not less than five times. The composition of the alloys is presented in Table 1. Besides Zr and C, the alloys contained as impurities an average of 0.011% O, 0.008% N and 0.0006% H.

The temperature of the alloys' solidus was determined by the drop method using an OPIR-017 optical pyrometer calibrated according to the melting points of Zr (1855°C), Hf (2220°C), Nb (2468°C) and Mo (2620°C). The accuracy of the determination was $\pm 20^\circ\text{C}$.

Cast alloys and alloys hardened after annealing in a TVV-2 furnace in a vacuum of $5 \cdot 10^{-6}$ mm Hg column from temperatures of 1970 and 1250°C. The length of the annealing at 1970°C was 47h; before hardening from a temperature of 1250°C the alloys were first annealed at 1970, 1750 and 1250°C, respectively, for 47, 27, 27 and 150 h. The rate of cooling the alloys from a temperature of 1970°C was more than 700 deg/min, and from 1250°C - more than 350 deg/min. A temperature of 1970°C is close to the supposed hardening temperatures; while a temperature of 1250°C is close to the tempering temperatures (~ 0.55 from T_{p1}) of these alloys.

TABLE 1

The Composition of the Investigated Mo-Zr-C Alloys

1 № сплав	2 Содержание компонентов, вес. %				1 № сплав	2 Содержание компонентов, вес. %			
	3 по шихте		4 по анализу			3 по шихте		4 по анализу	
	Zr	C	Zr	C		Zr	C	Zr	C
1	0.25	—	0.38	—	21	5.00	—	5.75	0.049
2	0.25	0.100	0.27	0.11	22	5.00	0.100	4.48	0.20
3	0.25	0.250	0.16	0.20	23	5.00	0.250	5.22	0.30
4	0.25	0.500	0.23	0.44	24	5.00	0.500	2.21	0.50
5	0.50	—	0.75	0.027	25	7.00	—	6.20	0.081
6	0.50	0.100	0.51	0.15	26	0.50	0.050	0.34	0.10
7	0.50	0.250	0.54	0.22	27	0.75	0.100	0.38	0.10
8	0.50	0.500	0.73	0.54	28	0.80	0.125	1.06	0.12
9	1.00	—	1.05	0.018	29	1.00	0.150	1.35	0.19
10	1.00	0.100	1.07	0.11	30	1.25	0.175	1.69	0.22
11	1.00	0.250	1.37	0.30	31	1.75	0.250	1.86	0.23
12	1.00	0.500	1.29	0.49	32	2.25	0.300	2.58	0.34
13	2.00	—	1.92	0.016	33	2.50	0.375	2.82	0.34
14	2.50	—	2.75	0.019	34	3.00	0.425	3.34	0.45
15	3.00	—	2.82	0.065	35	4.25	0.600	5.65	0.70
16	3.00	0.100	3.20	0.13	36	6.00	0.850	6.55	0.96
17	3.00	0.250	3.25	0.24	37	—	0.100	—	0.10
18	3.00	0.500	2.36	0.36	38	—	0.250	—	0.26
19	4.00	—	4.25	0.033	39	—	0.500	—	0.55
20	4.50	—	5.33	0.056					

1) Alloy No.; 2) content of components, wt. %; 3) from charge; 4) from analysis.

Sections of the alloys were prepared on coarse and fine emery paper and polished on cloth with a suspension of Cr_2O_3 in water. A mixture (1:1) of a 10% KOH solution and a 30% $\text{K}_2\text{Fe}(\text{CN})_6$ solution was used as the mordant; the etching time varied from 5-7 to 10-12 s depending on the composition of the alloys.

The degree of the alloys' approach to a state of equilibrium during annealing was checked by periodically measuring the period of the lattice and the microhardness of the molybdenum α -solid solution in individual alloys. The lattice period was determined with an accuracy of $\pm 0.0003 \text{ \AA}$ in an URS-50I unit (copper emission); the angles of reflections from the planes (321) and (400) were used for the calculation. The microhardness was measured in a PMT-3 instrument under a load of 20 g; to remove the superficial cold-worked layer the sections were etched with the above-named mordant for 1.5 min. The loading time was 2-3 s, the exposure under stress 7-8 s; the value of the microhardness was calculated from the data of measurements of the diagonals of 15-20 impressions.

The measurements showed that the lattice period and the microhardness of a molybdenum α -solid solution in the alloy Mo + 5.65% Zr + 0.70% C during annealing at a temperature of 1970°C remain practically constant (3.1488 \AA and 187 kg/mm^2) after 27 h. Similarly, the lattice period and the microhardness of a molybdenum α -solid solution in the same alloy remained unchanged (3.1471 \AA and 164 kg/mm^2) during 100 h of annealing at 1250°C .

Powders of the alloys were prepared for x-ray phase analysis. Mapping of the Debye powder patterns was carried out in a chamber with a diameter of 57.3 mm in copper emission with a nickel filter, using an asymmetrical arrangement of the film; the voltage in the tube was 35 kV, the current was 10 mA and exposure time 5 h.

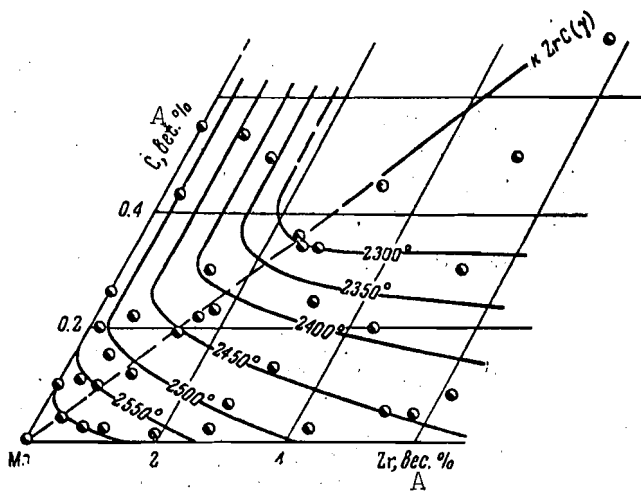


Fig. 1. Isotherms of the solidus surface of a molybdenum α -solid solution in the triple system Mo-Zr-C. A) wt. %.

Experimental data and discussion. The results of the determination of the temperatures of the start of melting of alloys of the

triple system Mo-Zr-C in the form of isotherms of the solidus surface of a molybdenum α -solid solution carried out for every 50° are shown in Fig. 1. The high melting point of the eutectic $\alpha + \text{Mo}_2\text{C}$ in the binary system Mo-C, 2510°C draws attention, whereas according to the literature data [10], it is ~2200°C. The decreased value of the melting point of the eutectic $\alpha + \text{Mo}_2\text{C}$ can be explained by the fact that in Reference [10] alloys prepared from contaminated materials were investigated. The data in Reference [3] also indicate a high melting point of the eutectic $\alpha + \text{Mo}_2\text{C}$ in the binary system Mo-C: after annealing of the alloy Mo + 0.04% C at a temperature of 2370°C, no signs of fusion were observed. It follows from this that the melting point of the carbide Mo_2C , equaling ~2400°C [10] also requires refinement.

The temperature of the eutectic horizontal in the section Mo-ZrC was equal to 2260°C, which practically coincides with the data of Reference [9] (2250°C). It is evident that the melting point of the triple eutectic $\alpha + \text{Mo}_2\text{C} + \text{ZrC} (\gamma)$ in the triple system Mo-Zr-C must be below 2260°C.

The comparatively high solidus temperature of the alloys investigated, Mo with 1-5.7% Zr and 0.02-0.05% C (2250-2450°C) makes it possible to assert that the maximum solubility of Zr in Mo in a solid state (at the temperature of the peritectic horizontal, equal to 1890 ± 20°C [11, 12]) exceeds 6%.

From the data of a determination of the temperatures of the start of melting, of a microscopic analysis of alloys hardened from temperatures of 1970 and 1250°C and of measurements of the lattice period and microhardness of a molybdenum α -solid solution in the hardened alloys, a polythermal section of Mo-ZrC was constructed (Fig. 2). The nature of the change in the solidus temperature of the alloys, the lattice period and the microhardness of a molybdenum α -solid solution confirms the conclusion of Reference [9] that the section of Mo-ZrC in the triple system Mo-Zr-C is quasi-binary. The carbide ZrC (γ) possesses variable solubility in molybdenum in a solid state which increases with a decrease in the temperature. The maximum solubility of ZrC (γ) in Mo (at 2260°C) is about 3.5%.

The data from a microscopic study (Fig. 2a) and the bends in the curves of the microhardness and lattice period of a molybdenum α -solid solution (Fig. 2b, c) indicate that the solubility of the carbide ZrC (γ) in Mo at a temperature of 1970°C is about 1.1%, and at 1250°C - about 0.65%.

Isothermal sections of the molybdenum angle of the triple system Mo-Zr-C at temperatures of 1970 and 1250°C are shown in Fig. 3. At a temperature of 1970°C (Fig. 3a) the carbides Mo_2C and ZrC (γ) are in equilibrium with the molybdenum α -solid solution in the alloys examined, while at 1250°C (Fig. 3b), the phase Mo_2Zr , in addition to these carbides. Three-phase regions $\alpha + \text{Mo}_2\text{C} + \text{ZrC} (\gamma)$ and $\alpha + \text{ZrC} (\gamma) + \text{Mo}_2\text{Zr}$ in the triple system Mo-Zr-C appear as the result of the following conversions: crystallization of the triple eutectic according to the scheme Liquid $\rightarrow \alpha + \text{Mo}_2\text{C} + \text{ZrC} (\gamma)$ at a temperature below 2260°C (in alloys of the triangle Mo- Mo_2C -ZrC) and the four-phase peritectic conversion Liquid + $\alpha \rightarrow \text{Mo}_2\text{Zr} + \text{ZrC} (\gamma)$

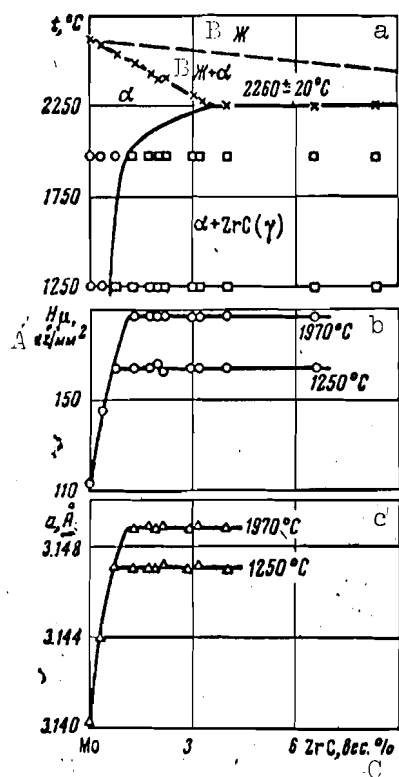


Fig. 2. Polythermal section of Mo-ZrC (a) and dependence of microhardness (b) and lattice period (c) of molybdenum α -solid solution on the composition of alloys hardened from temperatures of 1970 and 1250°C. A) H μ , g/mm²; B) liquid; C) wt. %.

at a temperature below 1890°C, which in alloys of the triangle Mo-ZrC-Mo₂Zr ends with the disappearance of the liquid phase. Since in the alloys which we studied these conversions do not take place, the appearance of the three-phase regions $\alpha + Mo_2C \pm ZrC(\gamma)$ and $\alpha + ZrC(\gamma) + Mo_2Zr$ in the isothermal sections at temperatures of 1970 and 1250°C (Fig. 3) is evidently connected with the separation of the carbides Mo₂C, ZrC (γ) and the phase Mo₂Zr from the molybdenum α -solid solution as a result of a decrease in the joint solubility of C and Zr in molybdenum in a solid state at a decreased temperature.

The solubility of C in Mo at a temperature of 1970°C was taken as equal to ~0.02% ([4,13] and at 1250°C - 0.01 [4, 13, 14]. The alloy Mo + 0.10%C studied in the present work at temperatures of 1970 and 1250°C had a two-phase structure $\alpha + Mo_2C$.

It is seen from the isothermal sections constructed that additions of Zr to alloys of the binary system Mo-C at first somewhat increase and then decrease the solubility of C in Mo in a solid state. For example, at a temperature of 1970°C, additions of Zr in amounts up to ~0.7% increase the solubility of C to ~0.12%, while at a large Zr content (3-6%) this solubility decreases to 0.03-0.05%. Analogously, at a temperature of 1250°C, additions of Zr

in amounts up to 0.4% increase the solubility of C to ~0.10%, while at a Zr content of 0.7%, this solubility decreases to 0.03-0.04%, etc. In alloys of Mo with 2-6% Zr, the carbide phase ZrC (γ) at a temperature of 1970°C appears at a C content of more than 0.03-0.05%, and at 1250°C, at a C content of more than 0.03-0.04%.

Photographs of the microstructures of some investigated alloys of the triple system Mo-Zr-C are shown in Fig. 4. The carbides Mo₂C and ZrC (γ), as well as the phase Mo₂Zr are difficult to distinguish from each other on unetched slides. After etching, the carbide Mo₂C inclusions take on a characteristic pinkish color, inclusions of the γ -solid solution in a ZrC base appear gray, while the phase Mo₂Zr has a dark gray color. In the three-phase alloys α + Mo₂C + ZrC (γ), inclusions of the carbide Mo₂C, as a rule, are very large, while the inclusions of ZrC (γ) have a finely dispersed form.

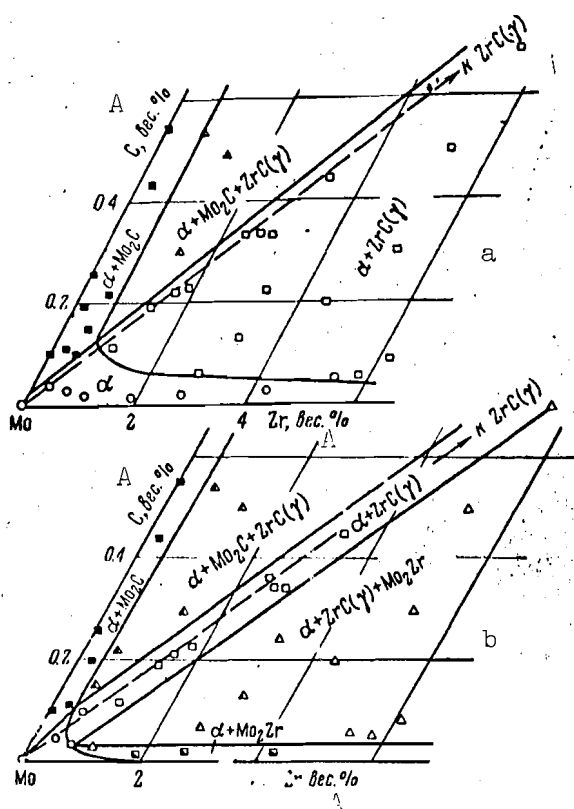


Fig. 3. Isothermal sections of triple system Mo-Zr-C at temperatures of 1970 (a) and 1250°C (b). A) Wt, %.

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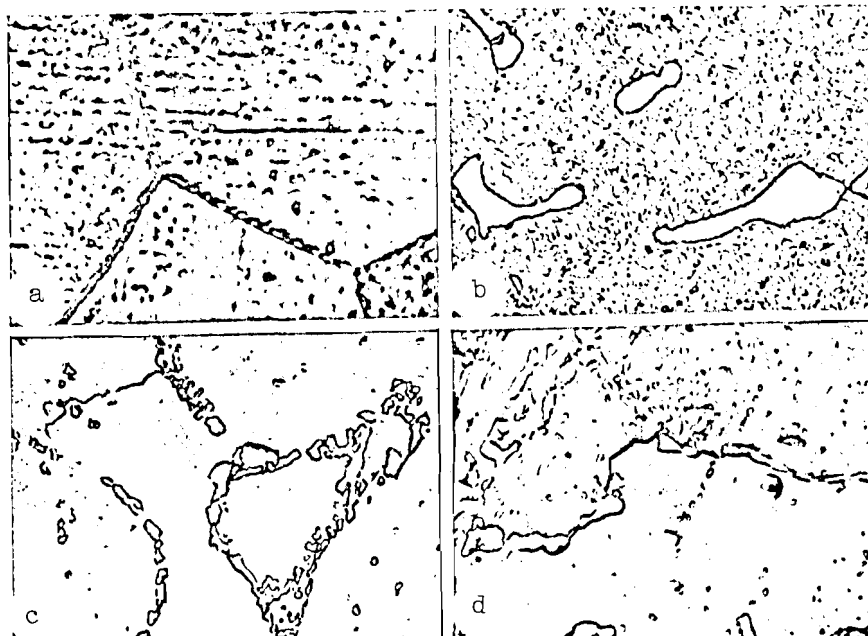


Fig. 4. Microstructures of some alloys of the triple system Mo-Zr-C (mordant - mixture of 10% KOH solution and 30% $K_3Fe(CN)_6$ solution in ratio of 1:1. a) Alloy 1 (see Table 1), hardening from 1250°C, α ($\times 900$); b) alloy 8, the same, but $\alpha + Mo_2C + ZrC (\gamma)$; c) alloy 34, hardening from 1970°C, $\alpha + ZrC (\gamma)$, ($\times 600$); d) alloy 25, hardening from 1250°C, $\alpha + ZrC (\gamma) + Mo_2Zr$ ($\times 90$).

The results of a microscopic study of alloys of the triple system Mo-Zr-C were completely confirmed by x-ray phase analysis. Diagrams of the roentgenograms of individual alloys having different phase compositions are shown in Fig. 5. Alloys located on the isothermic sections in the monophasic region α are uniform α -solid solutions in an Mo base and have a bcc lattice (Fig. 5a); alloys located in the two-phase region $\alpha + ZrC (\gamma)$, in addition to crystals of a molybdenum α -solid solution contain deposits of a γ -solid solution in a base of the carbide ZrC with an fcc lattice (Fig. 5b); finally, alloys of the three-phase regions $\alpha + Mo_2C + ZrC (\gamma)$ and $\alpha + ZrC (\gamma) + Mo_2Zr$ contain in their structure in addition to crystals of a molybdenum α -solid solution and inclusions of the carbide phase ZrC (γ), deposits of the carbide Mo_2C with a hcp lattice and the phase Mo_2Zr with an fcc lattice, respectively (Fig. 5c, d).

In spite of the comparatively small amounts of the carbide phases in the alloys studied, lines not only of the molybdenum α -solid solution base, but also a large part of the lines of Mo_2C and ZrC (γ) appeared on the roentgenograms (Fig. 5b, c) and only on roentgenograms plotted from alloys of the three-phase region $\alpha + ZrC (\gamma) + Mo_2Zr$; some weak lines of the phase Mo_2Zr (Fig. 5d) were absent.

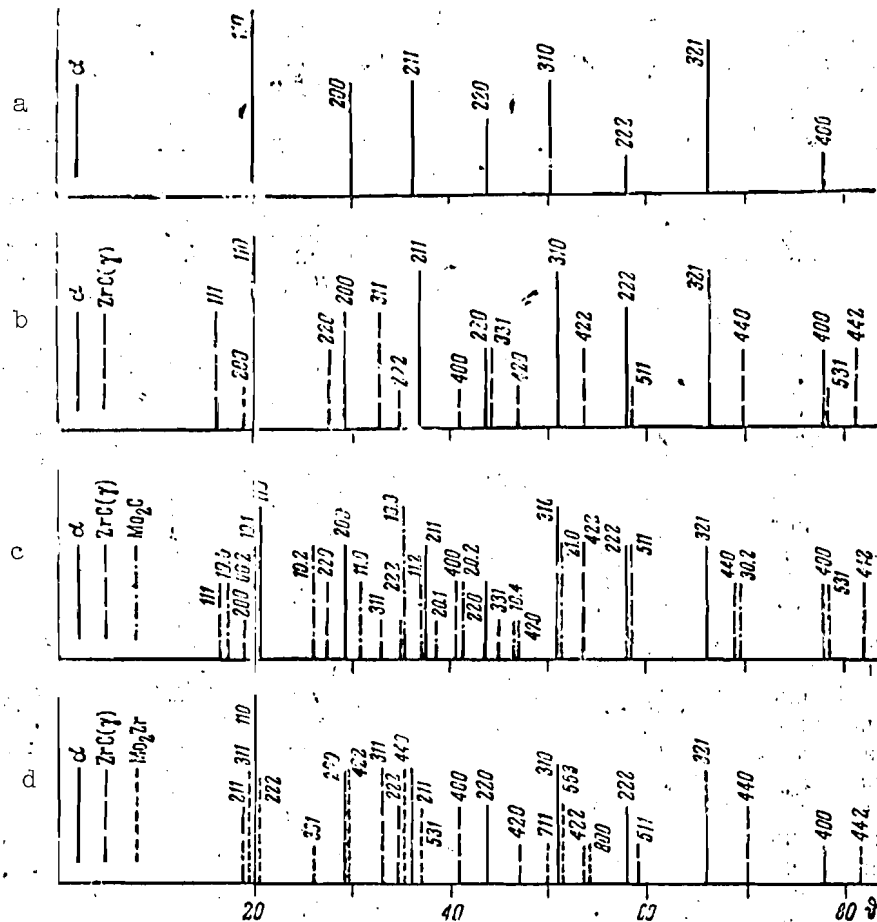


Fig. 5. Diagrams of roentgenograms of some alloys of the triple system Mo-Zr-C. a) Alloy 1, hardening from 1970 or 1250°C; b) hardening from 1970°C; c) alloy 12, hardening from 1250°C; d) alloy 23, hardening from 1250°C.

The results of a determination of the lattice periods of various phases in alloys of the triple system Mo-Zr-C are presented in Table 2. The rear lines of the roentgenograms (321) and (400) corresponding to the angles $\vartheta \sim 66$ and 78° were used to calculate the period of the bcc lattice of the molybdenum α -solid solution; the periods of the hcp lattice of the carbide Mo_2C were calculated from the lines (210) and (302) (the angles ϑ are equal to ~ 52 and 70°); the period of the fcc lattice of the carbide ZrC (γ) from the lines (440) and (442) (the angles ϑ are equal to ~ 70 and 81°) and the period of the fcc lattice of the phase Mo_2Zr accordingly from lines (533) and (800) (the angles ϑ are equal to ~ 52 and 55°).

The values obtained of the period of the bcc lattice of the molybdenum α -solid solution in the alloys studied (Table 2) confirm the joint effect of additions of Zr and C on the value of the lattice period of pure Mo: both C and Zr increase the lattice period of Mo [15]. The periods of the hcp lattice of the carbide

Mo₂C are close to the literature data ($a = 3.002$, $c = 4.724$ Å, $c/a = 1.574$ [16]; $a = 2.998 \pm 0.002$, $c = 4.734 \pm 0.003$ Å, $c/a = 1.58$ [17], if it is considered that in the triple system Mo-Zr-C a small region of triple solid solutions is formed in a carbide Mo₂C base and additions of Zr somewhat increase the lattice period of Mo₂C [7]. The period of the fcc lattice of the α -solid solution in a carbide ZrC base is also close to the lattice period of ZrC of stoichiometric composition ($a = 4.678$ Å [18], $a = 4.683$ kX [19, 20] and $a = 4.695$ Å [21, 22]) while the closer values obtained in the present work can be explained by the fact that additions of Mo and Zr to ZrC increase its lattice period [19].

TABLE 2

Phase Composition and Lattice Periods of Individual Phase in Investigated Alloys of the Triple System Mo-Zr-C

1 Сплав *	2 $t_{\text{зак}}$, °C	3 Периоды решетки a , Å, фаз			
		α	Mo ₂ C **	γ	Mo ₂ Zr
1	1970, 1250	3.144 ± 0.003	—	—	—
12	1250	3.145 ± 0.003	3.021 ± 0.005	4.661 ± 0.004	—
35	1970	3.1488 ± 0.003	—	4.668 ± 0.004	—
	1250	3.1471 ± 0.0003	—	—	—
23	1250	3.149 ± 0.003	—	4.667 ± 0.004	7.596 ± 0.008

*See composition in Table 1.

** $c = 4.785 \pm 0.007$, $c/a = 1.584$.

1) Alloys*; 2) $t_{\text{зак}}$, °C; 3) lattice periods a , Å, phase.

With regard to the period of the fcc lattice of the phase Mo₂Zr, the value obtained is in good agreement with the literature data ($a = 7.60 \pm 0.05$ Å [23], $a = 7.583$ Å [7] and $a = 7.590 \pm 0.005$ kX [24]).

Conclusions. 1. The molybdenum angle of the triple system Mo-Zr-C at a content in the alloys of 0.0-5 wt. % C and 0-6 wt. % Zr has been investigated by methods of microscopic, x-ray phase and structural analyses and by a determination of the temperature of the beginning of melting and the microhardness; isotherms of the solidus surface, isothermal sections at temperatures of 1970 and 1250°C and a polythermic section of MoZrC have been plotted.

2. At a temperature of 1970°C the carbides Mo₂C and ZrC (γ) are in equilibrium with the molybdenum α -solid solution, and at 1250°C - the phase Mo₂Zr, in addition to the named carbides.

3. The melting point of the eutectic $\alpha + \text{Mo}_2\text{C}$ in the binary system Mo-C has been established as equal to 2510 ± 20 °C.

4. The maximum solubility of Zr in molybdenum in a solid state (at a temperature of 1890°C) exceeds 6 wt. %.

5. It has been confirmed that the section of Mo-ZrC in the triple system Mo-Zr-C is of a quasi-binary nature and has a diagram of

the eutectic type; the eutectic $\alpha + \text{ZrC} (\gamma)$ in this section melts at a temperature of 2260°C , which practically coincides with the data of article [9] (2250°C).

6. The carbide $\text{ZrC} (\gamma)$ possesses variable solubility in molybdenum in a solid state, which decreases with a decrease in temperature; the maximum solubility of $\text{ZrC} (\gamma)$ in Mo (at a temperature of 2260°C) is about 3.5 wt. %, at 1970°C it is 1.1 and at 1250°C it is 0.65 wt. %.

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