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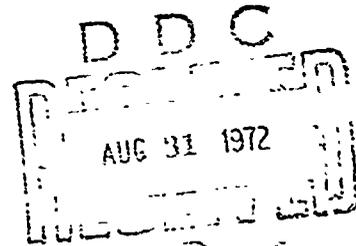
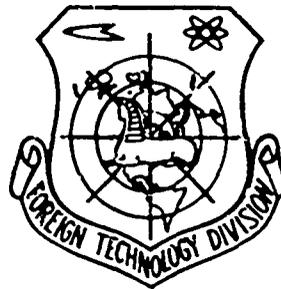
FOREIGN TECHNOLOGY DIVISION



RECRYSTALLIZATION OF HYDROSTATICALLY
EXTRUDED MOLYBDENUM

by

G. A. Mochalov and A. I. Yevstyukhin



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13. ABSTRACT

> The recrystn. of Mo of various compns. was studied, and the Type I recrystn. diagrams were detd. for samples that had been plastically deformed by extrusion. After deformation and thermal treatment, the microstructure, grain size, and hardness were detd. An increase in the heating temp. and a decrease in the degree of deformation resulted in a gradual change in the av. grain size. There was a marked increase in the grain size as the temp. was increased for forged Mo with 20% deformation. The av. grain size decreased with increasing deformation. A feature of the forged, extruded, cermet Mo is the very small change in the av. grain size for deformations of 60-80% for a heating temp. of 1600°. Another is the unusually high, anomalous growth in the grains at >1600° and deformations over 50%. Alloying with Ti and Zr stabilizes the structure and decreases the linear growth rate of the grains. As a result of the extrusion of Mo of any state with deformations of 40-80%, at 1100-1600°, a fine-grained structure was obtained.

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Б б	<i>Б б</i>	B, b	С с	<i>С с</i>	S, s
В в	<i>В в</i>	V, v	Т т	<i>Т т</i>	T, t
Г г	<i>Г г</i>	G, g	У у	<i>У у</i>	U, u
Д д	<i>Д д</i>	D, d	Ф ф	<i>Ф ф</i>	F, f
Е е	<i>Е е</i>	Ye, ye; Ё, ё*	Х х	<i>Х х</i>	Kh, kh
Ж ж	<i>Ж ж</i>	Zh, zh	Ц ц	<i>Ц ц</i>	Ts, ts
З з	<i>З з</i>	Z, z	Ч ч	<i>Ч ч</i>	Ch, ch
И и	<i>И и</i>	I, i	Ш ш	<i>Ш ш</i>	Sh, sh
Я я	<i>Я я</i>	Y, y	Щ щ	<i>Щ щ</i>	Shch, shch
К к	<i>К к</i>	K, k	Ъ ъ	<i>Ъ ъ</i>	"
Л л	<i>Л л</i>	L, l	Ы ы	<i>Ы ы</i>	Y, y
М м	<i>М м</i>	M, m	Ь ь	<i>Ь ь</i>	'
Н н	<i>Н н</i>	N, n	Э э	<i>Э э</i>	E, e
О о	<i>О о</i>	O, o	Ю ю	<i>Ю ю</i>	Yu, yu
П п	<i>П п</i>	P, p	Я я	<i>Я я</i>	Ya, ya

* ye initially, after vowels, and after ъ, ь; e elsewhere.
 When written as ѐ in Russian, transliterate as yѐ or ѐ.
 The use of diacritical marks is preferred, but such marks
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RECRYSTALLIZATION OF HYDROSTATICALLY EXTRUDED MOLYBDENUM

G. A. Mochalov and A. I. Yevstyukhin

The structural state has an extremely strong influence on the physical properties of metals, especially molybdenum [1-6]. The existence of fine-grained structure in molybdenum enables it to retain high plastic properties in the recrystallized state [3, 6]. Therefore, the proper selection of conditions for the thermal treatment for molybdenum and its alloys is extremely important. In this respect, knowing the diagrams of recrystallization, which relate the degree of plastic deformation and annealing temperature (of heat treatment) to the size of the obtained grain, is of considerable interest.

A small number of works describing the recrystallization diagrams of molybdenum is known [3, 7-11]. Ye. M. Savitskiy with coworkers gives a recrystallization diagrams of type I for the cast molybdenum with carbon additions of 0.1-0.2%, deformed by compression (to 70%) [3, 7] and a recrystallization diagram of type II for the hot-pressed molybdenum [8]. In the works [9-11] are given recrystallization diagrams of type I for the cermet molybdenum, which were obtained at relatively low annealing

temperatures (up to 1450°C) [9] and on very fine sheets [10]; moreover, the study was done at relatively small degrees of material reduction obtained by rolling.

The recrystallization diagrams obtained in these works do not encompass all of the numerous forms of shaping (for instance, hydrostatic extrusion) and the composition of the most known molybdenum alloys (TsM-2A, VM-1 and others) which are used in technology [3, 12, 13].

This work examines the recrystallization and gives recrystallization diagram of type I for the molybdenum with various composition, which was plastically deformed by the hydrostatic extrusion method.

THE INVESTIGATED MATERIALS

The recrystallization study was carried out for the hot-forged (reduction during hot forging is about 70%) cermet molybdenum, cermet (not forged) molybdenum, and hot-pressed molybdenum TsM-2A annealed at 2000°C in vacuum for 1 h [4, 5, 13]. Molybdenum TsM-2A in both states was used from one melt. The purity and composition of the tested materials (according to chemical, spectral and gas analysis data) are shown in Table 1.

Cold shaping with various degree of deformation (up to 80% reduction per section) of the molybdenum with various composition and original structure was done by means of a single hydrostatic extrusion [4, 5, 14]. Mechanical properties and structure of the indicated materials are described in works [4-6].

THE PROCEDURE OF INVESTIGATION

The study of microstructures of the material after deformation and heat treatment was done on the longitudinal sections.

Table 1. The alloying additions and admixtures content in the test materials.

Material	The obtaining method (specifications)	Content of certain elements, in wt. %								Note
		Ti	Zr	C	O	N	H			
Molybdenum TSM-2A Zi-6, 12, 137	Arc melting with the consumable electrode, hot pressing of castings with 78 mm diameter to a rod of 20 mm diameter (ChMTU 1200-64)	0,10	0,10	0,0018	0,0026	0,0045	0,00023			
Molybdenum M4 (purity 99,94%) Zi-5	Hot forging of cermet moldings (25 x 25 mm) at 1250-10500C to a rod with 17 mm diameter (TSMTU 16-64)	—	—	0,004	0,006	0,003	0,0005			The basic metallic admixtures content (Ni, Si, Ca, Mg, Zn, Cu, Al, Cr, Pb, N, Mn) less than 0.005 wt. % of each.
Molybdenum (purity 99,97%)	Moldings of cermet molybdenum (without preliminary shaping) (TU 7-135-54)	—	—	0,003	0,009	0,006	0,0008			The total content of metallic admixtures (Ca, Si, Fe, Cu, Al, Ni, Cr, Pb, Mg, Mn, Sn, Cd, Bi, Sb, Zn, As, S, P) is less than 0.009 wt. %.

The procedure for preparation and readying of the sections for study was described earlier [4].

The average grain diameter of the material was determined by the nodal points method of Saltykov and, in certain cases, additionally by calculating the number of grains per the unit of the surface section, using the Jeffreys's method [6, 15]. The grain size was determined only for the completely recrystallized material, therefore the spatial diagrams do not show the dependence of the average size grain for some degrees of deformation and temperatures because, in this case, the material being annealed has not recrystallized completely.

Vickers hardness was measured on the longitudinal sections by the TP-2 instrument at a load of 10 kgf.

The samples were annealed for 1 h with various degrees of deformation in a vacuum 10^{-5} mm Hg at 900-2000°C.

THE RESULTS OF INVESTIGATION

Softening. The temperature of beginning (t_p^H) and the end (t_p^H) of the recrystallization of the molybdenum with same composition and treatment was determined on the basis of the hardness measurement results and the data of microscopic analysis.

The region of recrystallization temperatures and also of the softening of the forged cermet molybdenum depends on the magnitude of the preceding deformation (Fig. 1, Table 2).

The initial (without extrusion) forged molybdenum is completely recrystallized after annealing at 1600°C. We should indicate that the temperature softening region (1400-1600°C) of the forged (without extrusion) molybdenum used in this study in

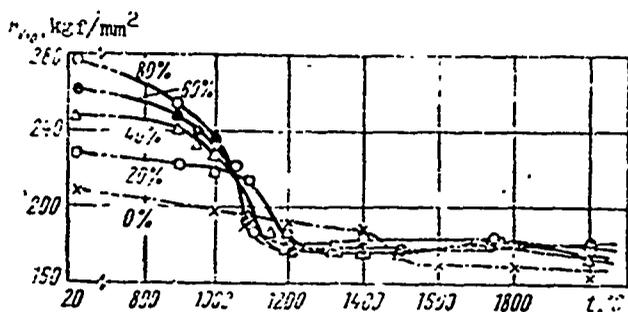


Fig. 1. The effect of the annealing temperature on the hardness of the forged cermet molybdenum subjected to the hydrostatic extrusion with various degrees of deformation (shown on the curves).

Table 2. Temperature of the beginning and the end of the recrystallization of the hydrostatically extruded molybdenum with various composition after the annealing for 1 h.

%, %	Forged cermet molybdenum		Molybdenum annealed (at 2000°C) and then extruded and hot pressed		Hot-pressed molybdenum TsM-2A	
	$t_p^H, ^\circ\text{C}$	$t_p^K, ^\circ\text{C}$	$t_p^H, ^\circ\text{C}$	$t_p^K, ^\circ\text{C}$	$t_p^H, ^\circ\text{C}$	$t_p^K, ^\circ\text{C}$
Initial state	1400	1600	—	—	*	1300—1320
20	1100	1200	1320	1100	—	—
40	1000	1100—1150	1200	1320—1400	1100—1150	1320
60	950—1000	1100	1150—1200	1320—1400	1100	1250—1320
80	950—1020	1050—1100	1150—1200	1250—1320	1050—1100	1250—1320

*A large number of fine recrystallized grains was observed in the initial structure of the hot-pressed molybdenum TsM-2A (without annealing) [4, 13].

the form of rods with 17 mm diameter [4, 5] is significantly different from the published data [6] on the recrystallization (900–1150°C) of the forged molybdenum used for the study in the form of rods with 7 mm diameter, apparently, due to the following reasons. The small magnitude of deformation (about 10–25% per run) during the repeated hot forging and the preparatory heating for forging the large castings (initial size of section 25 × × 25 mm) are accompanied by a partial recovery of properties of the forged material (final size of the semi-finished product – rods

with 17 mm diameter). The recovery process of properties and the changes in structure occur, apparently, as a result of the intense treatment of the polygonization castings (possibly, also due to partial recrystallization), completely active under these conditions. Due to a more complete occurrence of polygonization, the structure of the hot-forged molybdenum (rods with 17 mm diameter) proves to be very resistant to subsequent recrystallization [16, 17], as opposed to the molybdenum structure (rods with 7 mm diameter) which has undergone a stronger work hardening during the rotation forging [6]. The total degree of deformation during forging, their structure, and purity based on the admixtures, are close for both materials. It is possible to assume that the indicated above processes (basically polygonization) are responsible for the softening of the molybdenum (rods with 17 mm diameter) during a subsequent complete recrystallization at high temperatures.

In forged molybdenum with 20% deformation the recrystallization is finished at 1200°C, with deformation of 40% - at 1100-1150°C. The increase in deformation with hydrostatic extrusion up to 80% somewhat lowers the recrystallization temperature. For material with 60-80% deformation, the recrystallization occurs in the 950-1000°C temperature range (see Table 2).

In view of the fact that molybdenum TsM-2A is alloyed with small additions of titanium and zirconium [4, 9, 13], this material has an increased level of the recrystallization temperature (and respectively, the interval of softening), than the molybdenum without alloying [3].

The temperature range of softening of the molybdenum TsM-2A annealed (at 2000°C) and then hydrostatically extruded with various degrees of deformation is determined by the reduction value (Fig. 2, Table 2). The hardness of the completely annealed

(initial) material during reheating is virtually independent of the temperature. In the weakly deformed material ($\psi_1 = 20\%$), the recrystallization is finished at 1400°C . A complete softening of material with 80% deformation starts at 1320°C .

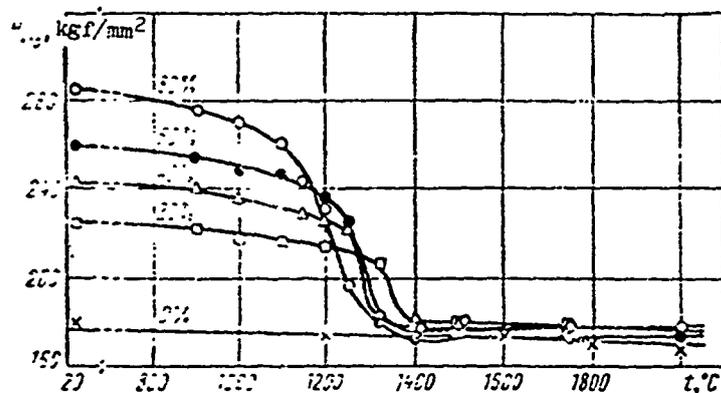


Fig. 2. The temperature effect of annealing on the hardness of the hot-pressed molybdenum TsM-2A, subjected to recrystallization (at 2000°C) and subsequent hydrostatic extrusion with various degrees of deformation (shown on curves).

As shown by the microscopic examination, the formation of coarse-grained structure in the narrow temperature range of recrystallization was observed in the two deformed materials (the molybdenum TsM-2A with 20% reduction and the forged cermet molybdenum with 20% reduction and, it seems, 0%). The process of recrystallization and the coarsening of the material structure with these degrees of deformation occurs both in the mechanism for the selective growth at some initial, less distorted, grains and in the formation mechanism of the new recrystallization centers. The coarsening of grains during recrystallization, divided into these two mechanisms, was revealed in work [18]. Overlapping of the two coarsening of grains processes during annealing, undoubtedly, does occur because the gradient of the degree of deformation and the unhomogeneity of deformation in grains are large at such small reductions of the polycrystalline molybdenum. Within the least deformed grains which are the nuclei of recrystallization, the processes of redistribution of dislocations and formation of the sub-structure of the polygonization, which also leads to softening, occur simultaneously [7].

Characteristic feature during the softening of the hot-pressed molybdenum TsM-2A is that the hot-pressed material with additional plastic deformation during hydrostatic extrusion with 40-80% reduction, just as the original material, is completely recrystallized at about 1300°C (Fig. 3, Table 2). The recrystallization of the hot-pressed extruded molybdenum TsM-2A starts and ends at lower temperatures than in the above described molybdenum TsM-2A of another state (with given and identical content of the alloying additions in the molybdenum).

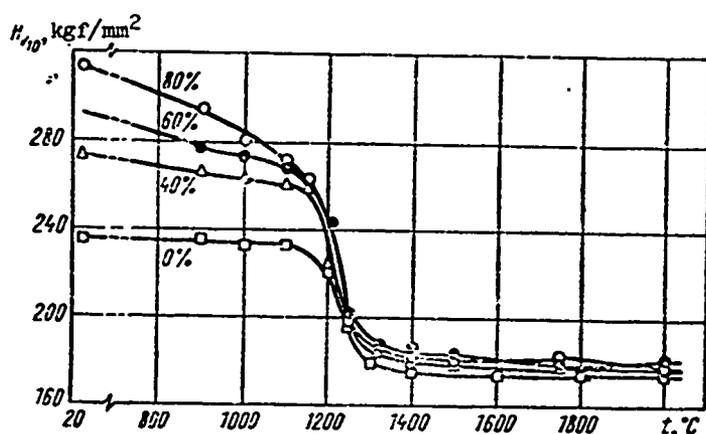


Fig. 3. The temperature effect of annealing on the hardness of the hot-pressed molybdenum TsM-2A subjected to hydrostatic extrusion with various degrees of deformation (shown on curves).

The recrystallization study of the extruded (40-80%) molybdenum with various composition has shown that the new recrystallization nuclei are located basically on the junctions of some of the deformed grains, which is in agreement with the earlier established data [17, 19]. Furthermore, the number of recrystallization nuclei is considerably larger in the extruded (40-80%) hot-pressed molybdenum TsM-2A (see Fig. 3); therefore, the final grain size is considerably less than in the extruded (40-80%) and annealed molybdenum TsM-2A (see Fig. 2). The formation of a small number of nuclei and relatively rather coarse grain is characteristic for the last material TsM-2A. Evidently, the combined effect of hot pressing and subsequent cold hydrostatic

extrusion distorts the lattice in adjacent grains and throughout the volume of the deformed material very strongly and creates a significant density and such distribution nature of dislocations, which, apparently, simultaneously facilitates the formation of an enormous number of nuclei during recrystallization at numerous grain boundaries of the material and, also, somewhat lowers the recrystallization temperature.

Thus, cold deformation under the conditions of high hydrostatic pressure, i.e., hydrostatic extrusion, reduces the recrystallization temperature of technically pure or low-alloy molybdenum, which is in accordance with the established laws of the degree of deformation effect on the nature of recrystallization [17, 19].

The degree of influence of the hydrostatic extrusion on the recrystallization temperature depends on the initial state of the material, i.e., on the preliminary shaping. In this case, only the influence of plastic deformation is examined during the hydrostatic extrusion for the material of certain purity and alloying without considering other factors which change the recrystallization temperature level considerably [3, 17, 19].

The recrystallization diagram of type I. The recrystallization diagram of the extruded forged cermet molybdenum is given in Fig. 4. An increase in the annealing temperature and a decrease in the degree of deformation leads to a gradual change in the average size of the grains. A noticeable growth of grains with increased annealing temperature is observed in the forged molybdenum deformed to 20%. In this work we did not examine the material with small (about 3-15%) "critical" deformations. Nevertheless, as a result of the annealing of material at 2000°C with 20% deformation we have obtained grains

of maximum size (about 220 μm). After holding at 2000°C, the average grain size in initial (without extrusion) forged molybdenum proves to be less than in material with 20% deformation (at the same annealing temperature), although such a magnitude of deformation (20%) is still, apparently, not critical.

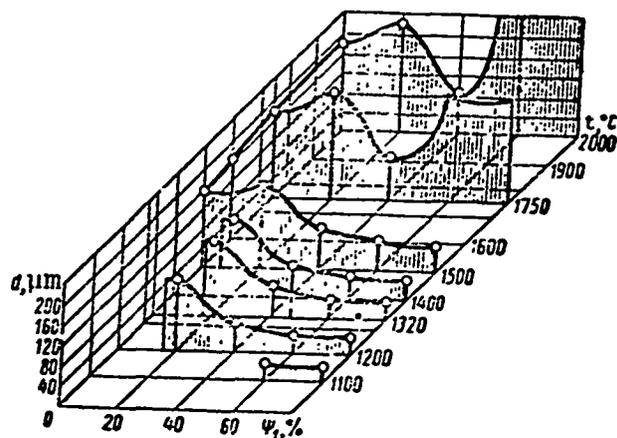


Fig. 4. The recrystallization diagram of type I of the hydrostatically extruded forged cermet molybdenum.

With increased deformation, the average grain size decreases and comprises about 30 μm (degree of deformation - 80%) in the forged molybdenum annealed at 1200°C. The essential feature of the forged extruded cermet molybdenum is in the very insignificant change in the average size of the grains with 60-80% deformation value up to 1600°C annealing temperature (for the material with a given level of admixtures). The weak dependence of the grain size on the annealing temperature of the extruded forged molybdenum [6] is a consequence of the exceptional uniformity of deformation during hydrostatic extrusion, because after the annealing of the extruded cermet molybdenum there forms a fine-grained equiaxial recrystallized structure which is very stable during the annealing over a wide range of temperatures.

The retardation of the aggregating recrystallization (the delay in the growth of the grains with increased annealing temperature) in the extruded unalloyed cermet molybdenum is caused, apparently, also by a certain level of the impurity content in the industrially pure metal (basically, the interstitial impurities).

Another specific feature of recrystallization of the extruded forged cermet molybdenum is the unusually high anomalous growth of grains at the annealing temperatures above 1600°C and degrees of deformation greater than 50%. The effect of treatment with a single cold hydrostatic extrusion is so great and the reserve of the deformation energy is so enormous that, during the subsequent recrystallization of such workhardened material, monocrystals grow over the entire length of the test rod [20]. In works [3, 7-11, 21, 22] in the recrystallization of molybdenum, such an effect of the anomalous growth of grains was not detected, although the phenomenon of monocrystallization of wires when the temperature gradient exists was known earlier [23].

An analogous nature of change in the structure during the annealing is observed in the hydrostatically extruded cermet molybdenum which was not subjected to any shaping prior to the deformation of extrusion (Fig. 5).

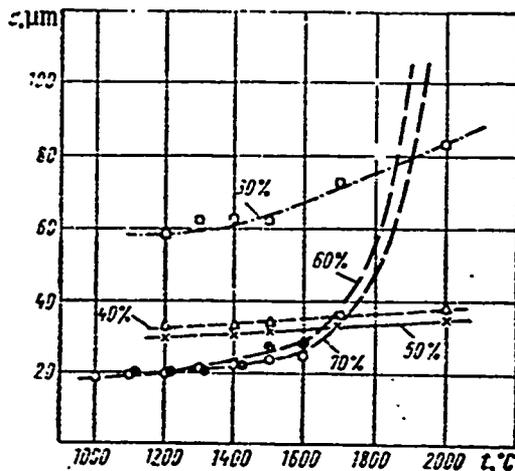


Fig. 5. The temperature effect of annealing on the change in the average grain size of the cermet molybdenum subjected to hydrostatic extrusion with various degrees of deformation (shown on curves).

In molybdenum TsM-2A annealed at 2000°C and then extruded, a coarse-grained structure is formed as a result of heating the original material in the 900-2000°C range without extrusion and with 20% deformation (Fig. 6). For the original material of this state ($\psi_1 = 0\%$) the grain size increases from 80 μm (annealed at 1400°C) to 150-160 μm (at 2000°C). Although 20% deformation is apparently not "critical", nevertheless, the value of the average size grain is large and comprises 180-200 μm for this deformation after being annealed at 2000°C.

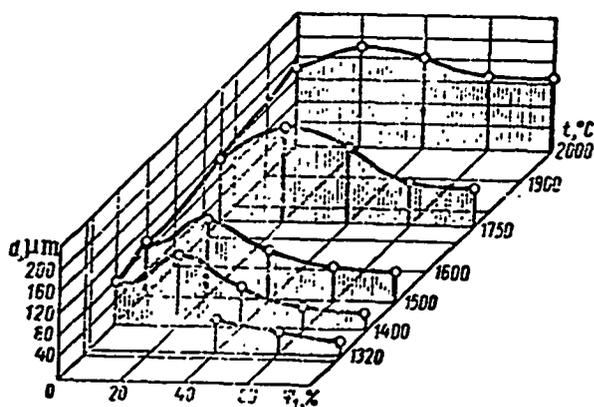


Fig. 6. The recrystallization diagram of type I of the annealed (at 2000°C) and then hydrostatically extruded molybdenum TsM-2A.

For the molybdenum TsM-2A the average grain size is 23-25 μm for the 80% deformation, after being annealed at 1320°C. Nevertheless, even for the deformed (80%) material in this state, after annealing the structure proves to be nonuniform - with relatively coarse and very fine grains, although on the whole the structure is fine-grained. The reason for the nonuniformity in the material structure of this state is apparently connected with the development and growth peculiarities of the recrystallization nuclei.

The noticeable growth in grains is observed in the extruded (40-80%) annealed molybdenum TsM-2A, after being annealed at above 1600°C. The collective recrystallization, leading to the general coarsening of the structure, occurs in the temperature range of 1600-2000°C.

In view of the fact that the hot-pressed molybdenum TsM-2A was not deformed with 5-35% degrees of reduction, therefore, broken lines have been plotted on the isothermal cuts corresponding to this region of deformations (Fig. 7). The structure was fine-grained when the extruded (40-80%) hot-pressed molybdenum TsM-2A was annealed at 1300-1500°C. Thus, the grain size of the extruded (80%) molybdenum annealed at 1320°C is 18-20 μm , and for the original (without extrusion) - about 23-29 μm . Just as for material TsM-2A (see Fig. 6), the grain size increases only after the annealing at temperatures above 1600°C (for any degree of deformation) when there is a collective recrystallization (see Fig. 7).

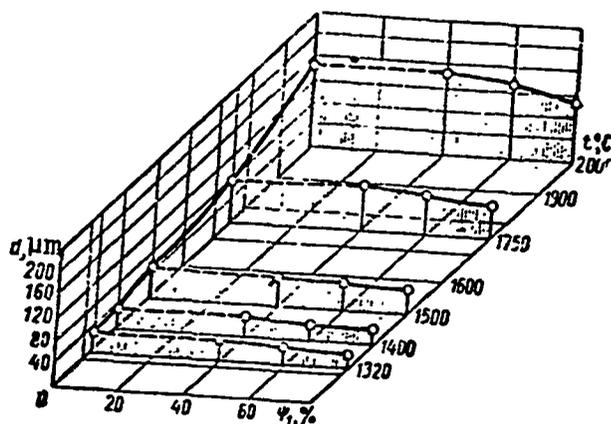


Fig. 7. The recrystallization diagram of type I of the hydrostatically extruded hot-pressed molybdenum TsM-2A.

The recrystallized structure of the extruded (0-80%) hot-pressed molybdenum TsM-2A proves to be more fine-grained than that in the extruded annealed material TsM-2A of the same composition (see Fig. 6), if the comparison is made for the same annealing temperatures and the degrees of deformation.

At high annealing temperature (about 2000°C), the difference in the grain size of material TsM-2A with various degrees of deformation is insignificant (see Figs. 6 and 7).

As follows from the recrystallization diagrams of the extruded molybdenum TsM-2A with various initial state, the effect of the anomalous growth of grains, which occurs in the extruded unalloyed cermet molybdenum, is absent in the TsM-2A material (see Figs. 4 and 5). Apparently, alloying molybdenum with titanium and zirconium stabilizes the structure and decreases the linear growth rate of the grains during collective recrystallization [17, 19].

Thus, the studied recrystallization diagrams of type I of the extruded molybdenum show that a fine-grained structure is formed in the molybdenum of any state as a result of hydrostatic extrusion with 40-80% deformation in the 1100-1600°C temperature range.

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