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PRODUCTION AND TREATMENT OF NON-FERROUS ALLOYS

- USSR -

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FOREWORD

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PRODUCTION AND TREATMENT OF NON-FERROUS ALLOYS

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Following is a translation of two articles in the Russian-language magazine Tsvetnyye Metally (Non-ferrous Metals), Moscow, Vol. 37, No. 7, July 1964. Complete bibliographic information accompanies each article.

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(SPECIFIC COLD-WORKING PRESSURE FOR TITANIUM ALLOYS)

[Following ② is a translation of an article by V. I. Shilov ① and V. P. Korzh in the Russian-language periodical Tsvetnyye Metally (Nonferrous Metals), Moscow, Vol. 37, No. 7, July 1964, pages 70-72.]

The well-known formula of Hitchcock (1) for calculating the average specific pressure and zone of deformation in which the elastic flattening of rollers is taken into account has been widely disseminated. However, the usefulness of this formula has been disputed on the grounds that it inaccurately reflects the physical essence of the elastic contact of the rollers with the bar since it assumes the stress-strain diagram of the specific pressures in the zone of deformation has a parabolic form (2--4) and therefore gives values of the extent of this zone which are too low.

The available experimental data of M. M. Saf'yan (5) and V. A. Palochkin (6) on the determination of the elastically deformed arc of bite of a bar under extension by rollers nevertheless confirms the practical applicability of Hitchcock's formula for determining the length of the zone of deformation of a bar with rollers where the elastic flattening of the rollers has been taken into account. Therefore, the use of Hitchcock's formula in recent works (7--8) has not been accidental.

Along with this, the calculation of the elastic properties of the metal being deformed, even if also in Hitchcock's formula, is worthy of attention; this has particular value during the cold-rolling of a thin strip of highly-durable metals and alloys which on ordinary rolling equipment are deformed with little reduction per pass. Analysis shows that, in individual cases, the calculation of this factor (i.e., of the effect of the elastic properties of the bar) can overlap the action of the factor which usually inaccurately corresponds to the nature of Hitchcock's formula -- the exaggeration of the specific pressure from the point of view of emergence of metal from the rollers.

A formula for the calculation of specific pressures during rolling which takes into consideration not only the elastic properties of the deforming instrument (the rollers), but those of the bar as well has been presented by A. I. Tselikov (9).

Using Tselikov's equation for the calculation of the length of the zone of deformation, we will define the average specific pressure in terms of the well known total rolling force P_{ob}:

$$\boxed{p = \frac{P_{об}}{\sqrt{BR\left[B\Delta h + 16P_{об}\left(\frac{1-\mu_1^2}{\pi E_1} + \frac{1-\mu_2^2}{\pi E_2}\right)\right]}},}$$

where B is the width of the bar in millimeters; R is the radius of the roller in operation, in millimeters; Δh is the reduction of the strip in millimeters; p is the specific pressure in kg/mm^2; μ_1 and E_1 are Poisson's ratio and the elastic modulus of the roller; and μ_2 and E_2 are Poisson's ratio and the elastic modulus of the bar.

Start [The average specific pressures during cold rolling of thin strips of titanium alloys AT-3, AT-4, and AT-6 were calculated on the basis of an expression obtained from experimental data of work (8) (See Tables 1 and 2). The values for the modulus of elasticity E_2 of these alloys were taken (10) to be 11,200; 12,500; and 12,700 kg/mm^2, respectively; with Poisson's ratios taken as 0.3, 0.33, and 0.33, respectively. For steel rollers, E_1 usually equals 22,000 kg/mm^2 and μ_1 equals 0.3. In the sixth column of Table 2, data are cited on specific pressures obtained from the equation above, but with the condition $\frac{1-\mu_2^2}{\pi E_2} = 0$, i.e., according to the generally-accepted formula of Hitchcock (8). The dependence of average specific pressures on the total coefficient of extension during the cold rolling of strip of alloy AT-4 S (See Table 2), obtained from the calculations of Hitchcock's formula and from the equation above are shown in the diagram.

[The [illegible] contains deformations and force conditions during [illegible] cold rolling [illegible] the titanium alloys.]

end

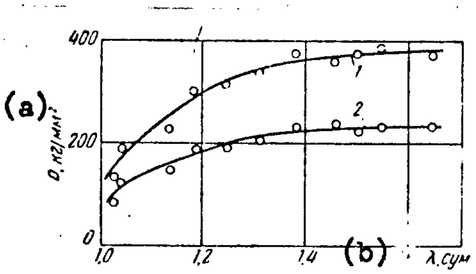
Table 1

Chemical Composition of Titanium Alloys
AT-3, AT-4, and AT-6 according to (10)

(a) Марка сплава	(b) Состав, % (вес.)				
	Al	Cr	Fe	Si	B
AT-3	2,5—3,5	0,4—0,9	0,25—0,60	0,25—0,60	0,01
AT-4	3,5—5,0	0,4—0,9	0,25—0,60	0,25—0,60	0,01
AT-6	5,0—6,5	0,4—0,9	0,25—0,60	0,25—0,60	0,01

Legend: (a) Brand of alloy
(b) Composition, percent by weight

It follows from the data obtained that the effect of the calculation of the elastic properties of thin strip on the magnitude of the average specific pressures during the rolling of type AT titanium alloys is significant; consequently, the calculation of this is expedient, because it gives a fuller and more exact value of the magnitude of the specific pressures.



Dependence of average specific pressure on the total coefficient of elongation which was obtained during the cold rolling of a thin strip of alloy AT-4S with initial thickness 0.255 mm.

1 - calculation using Hitchcock's formula
2 - calculation according to the formula derived here

Legend: (a) p, in kg/mm^2
(b) λ, total

Table 2

Deformations and Force Conditions During the Cold Rolling of Thin Strip of Titanium Alloy, Brands AT-3V, AT-4S, AT-6N. 50- and 75-Millimeter Wide Strip.

(a) Толщина ленты, мм		(b) Усилие прокатки, т P_i	(c) Длина очага деформации, мм l_i'	(d) Удельное давление, кг/мм² p_i	(e) Длина очага деформации, мм	(f) Удельное давление, кг/мм² p_i	(g) Удельное натяжение кг/мм²		$\frac{2l_i'}{h_{i-1}+h_i}$	$\frac{2l_i''}{h_{i-1}+h_i}$	Δp %
h_{i-1}	h_i						t_{i-1}	t_i			
	(h) Сплав АТ-3В				$b = 50$ мм				$d=53,90$ мм		
0,350	0,330	13,9	1,45	192	2,28	122	17,7	11,1	4,27	6,70	36
0,330	0,300	22,0	1,82	243	2,86	154	18,8	17,5	5,77	9,08	37
0,300	0,260	43,5	2,44	356	3,97	219	22,1	13,2	8,72	14,2	38
0,260	0,240	44,3	2,35	376	3,92	226	22,8	22,0	9,40	15,7	41
0,240	0,230	43,6	2,27	382	3,86	226	27,4	12,9	9,66	16,4	41
	Сплав АТ-4С				$b=74$ мм				$d=53,60$ мм		
0,255	0,250	9,00	0,90	134	1,41	86	16,2	6,93	3,59	5,58	34
0,250	0,245	15,8	1,16	186	1,84	116	22,2	13,6	4,67	7,43	38
0,245	0,225	27,4	1,62	229	2,50	148	17,7	17,7	6,90	10,6	35
0,225	0,215	39,7	1,81	296	2,92	184	20,2	15,3	8,25	13,3	38
0,215	0,205	44,6	1,92	315	3,09	195	18,6	24,3	9,14	14,7	38
0,205	0,195	51,1	2,04	339	3,30	209	19,9	19,6	10,2	16,5	38
0,195	0,185	60,2	2,21	369	3,57	228	20,5	25,8	11,6	18,8	38
0,185	0,175	57,7	2,16	361	3,36	232	23,4	22,4	12,0	18,7	37
0,175	0,170	58,9	2,15	371	3,51	227	27,2	28,0	12,4	20,4	39
0,170	0,165	61,4	2,19	379	3,59	231	24.6	23,0	13,1	21,4	39
0,165	0,155	61,7	2,23	374	3,62	230	27,8	23,8	13,9	22,6	38
	Сплав АТ-6Н				$b=76$ мм				$d=53,60$ мм		
0,360	0,350	11,3	1,05	141	1,58	94	12,6	11,8	2,97	4,45	33
0,350	0,330	17,0	1,34	167	1,98	113	15,6	18,5	3,94	5,82	32
0,330	0,310	29,0	1,64	233	2,51	152	18,2	17,7	5,13	7,84	35
0,310	0,285	36,5	1,84	261	2,82	170	18,8	20,9	6,18	9,48	35
0,285	0,265	34,8	1,77	260	2,74	167	21,2	18,8	6,42	9,96	36
0,265	0,255	31,8	1,62	258	2,57	163	20,8	21,5	6,23	9,88	37
0,255	0,240	33,8	1,70	261	2,68	166	23,6	20,1	6,88	10,8	36
0,240	0,235	30,9	1,56	261	2,51	162	23,3	23,9	6,57	10,6	38
0,235	0,220	35,4	1,74	268	2,74	170	25,8	20,0	7,65	12,0	37
0,220	0,210	38,1	1,76	285	2,82	178	25,4	28,8	8,20	13,1	38
0,210	0,200	37,4	1,75	282	2,78	177	31,1	21,4	8,52	13,6	37
0,200	0,190	45,8	1,92	315	3,07	196	27,2	27,1	9,84	15,7	38
0,190	0,175	42,8	1,90	290	3,00	188	28,1	27,7	10,4	16,4	35

(i) В таблице индексы Н, С и В условно обозначают соответственно нижний, средний и верхний преде легирующих элементов в сплаве.

Legend to Table 2:

(a) Thickness of strip in millimeters
(b) Rolling force in metric tons
(c) Length of deforming source in millimeters
(d) Specific pressure in kg/mm^2
(e) Length of deforming source in millimeters
(f) Specific pressure in kg/mm^2
(g) Specific tension in kg/mm^2
(h) Alloys AT-3V, AT-4S, AT-6N
(i) In the table, the indexes N, S, and V [Russian letters H, C, and B] conventionally refer to the lower, average, and upper limits of components of the alloy.

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PRODUCTION OF NIOBIUM-BASE ALLOYS BY CARBOTHERMIC REDUCTION OF THE OXIDES

[Following is an article by O. P. Kolchin, N. P. Chuveleva, and N. V. Sumarokova in the Russian-language periodical Tsvetnye Metally (Nonferrous Metals), Moscow, Vol. 37, No. 7, July 1964, pages 73-76.]

Niobium is used in ever-increasing quantities as a base for heat-resisting and other alloys.

At present, these alloys are prepared basically in one of two ways from the pure metal: 1) smelting of moldings or ingots of niobium with rods, wire, or strip of an alloying metal; 2) pressing into a molding and sintering a mixture of niobium and alloying-metal powders. In the smelting method, the preliminary extraction of the pure metals is necessary and a uniform distribution of alloying additions throughout the volume of the ingot without additional smelting of the ingots is not guaranteed. In the cermet method, the moldings have relatively small dimensions and this limits the productivity of the process. The cost of the alloys obtained by either method is excessively high and is significantly greater than the cost of unalloyed niobium.

These marked deficiencies are eliminated in the process of production of niobium-base alloys described below. This process consists of the joint reduction of niobium and the alloying metals from their oxides by the carbothermic method which is widely used for the reduction of unalloyed niobium in the reduction process of (1, 2). It is possible to introduce the alloying metals into the initial charge as oxides or carbines (4), and also (when indispensable), as pure metals.

Two processes of reduction are studied. In the first, the reduction of a mixture of niobium pentoxide, the oxide of the alloying metal, and niobium carbide is carried out; in the second, a mixture of niobium pentoxide and mixed niobium carbide and alloying metal are used. The latter is obtained preliminarily from a mixture of niobium pentoxide, the oxide of the alloying metal, and soot in a graphite-tubular oven at

1800-1900°C in a hydrogen atmosphere by a method formerly used to obtain niobium carbide (2).

The mixtures obtained by the two processes were pressed into moldings. The reduction of the 10 x 10 mm^2 moldings was carried out at 1700-1750°C in an oven with graphite heaters, at 1900-2100°C in an oven with tungsten heaters; that of the 20 x 20 mm^2 molding took place in vacuo in a furnace with a type OKB-533 graphite heater at 1900° C or in one with type LTs-42 niobium carbide heaters at 2100°C (3). The residual pressure of gases was $1 \cdot 10^{-3}$ mm Hg in this case.

The conditions of obtaining alloys were basically analogous to the conditions of obtaining pure niobium by the carbothermic method (2). The following metals were used as alloying additions: Al (group III in the periodic table), Ti and Zr (group IV), V and Ta (group V), Mo and W (group VI). These metals, with the exception of aluminum, form unbroken series of solid solutions with niobium (titanium and zirconium in the form of beta phases). Aluminum forms intermetallic compounds with niobium. They originated as a sample of alloying metals from a composition of niobium-base alloys issued by some Amerifirms (4).

The raw materials used to produce the alloys were: niobium pentoxide prepared with the use of the extraction of tributyl phosphate and containing 0.2% TiO_2, 0.1% Fe_2O_3, 0.05% SiO_2, 0.02% Al_2O_3, 0.025% K, 0.015% Na, 0.26% F, less than 0.05% S, less than 0.01% P, less than 0.01% Mn, and less than 0.1% Ca; niobium carbide, produced from this pentoxide and carbon black whose components were 89.2% Nb, 10.6% C, 0.13% N_2; analytical grade vanadium pentoxide; vanadium trioxide obtained by the hydrogen reduction of the pentoxide; sublimated molybdenum trioxide, tungsten trioxide; zirconium dioxide; titanium dioxide; pure tantalum pentoxide and aluminum oxide; and chimney soot.

The following summary reduction reactions were used in the calculation of the charge needed to obtain the alloys:

$$Nb_2O_5 + 5NbC = 7Nb + 5CO.$$

First process

$$Me_xO_y + yNbC = yNb - xMe + yCO;$$

Second process

$$5Me_xC + Nb_2O_5 = 2Nb - 5xMe + 5CO,$$

where Me is W, Mo, Zr, Ti, V, Ta, or Al.

For greater purification from carbon in the process, oxides with 2-5% excess over stoichiometrical quantity were chosen.

As can be seen from the data of Tables 1 and 2, niobium-base alloys with Mo, W, 1% Zr, and 3% V in the described conditions are obtained from the given compositions, inasmuch as the deviations of the analytical results from the predicted compositions lie practically within the limits of error of the analytical methods. The same thing holds true for niobium-tantalum alloys (5). No appreciable difference in the two processes in the production of alloys was noted; however, the first (mixtures of oxides with niobium carbide) is the more expedient for obtaining these alloys. That positive results are obtained during the joint reductions of Nb, W, and Mo is somewhat unexpected, since tungsten and molybdenum oxides are characteristically highly volatile: WO_2 has already significantly sublimed at 1050°C, the pressure of WO_3 vapor is equal to 3.23 mm Hg at 1175°C, and that of MoO_3 is equal to 40 mm Hg at 1165°C. Obviously, in the conditions under which these experiments take place, these oxides basically succeed in being reduced in a region of relatively low temperatures at which evidently, not carbon, but its oxide, becomes a spontaneous reducing agent. This is explained to a significant degree both by the smaller affinity of Mo and W, as opposed to the other metals under study, for oxygen (6), and also by the polymerization of the oxide vapors.

Furthermore, during the investigation, it was noticed that the joint reduction of Nb, W, and Mo in which alloys were formed proceeded more quickly and completely than those in which Nb alone was being reduced, i.e., in the presence of W and Mo, the reduction of Nb was accelerated.

The lowest oxides of V and Ti are characterized as having higher durability than the oxides of W and Mo; thus, it is more difficult to reduce them. At the same time, at the temperature of reduction, these oxides, and also metallic V and Ti, have significant vapor pressure (in contrast to Zr) which leads to important losses when they are in large amounts in the charge. The vapor pressures of Ti and V amount to $1 \cdot 10^{-3}$ mm Hg at 1577 and 1687°C, respectively. When vanadium is first bound into a carbide and niobium mixture and its content in the alloy is 5-6%, no loss of it is observed; but during reduction directly from the oxide with a 10% estimated amount of vanadium, approximately half of it has time to evaporate. In connection with those alloys having greater than 3% V content, it is more rational to use the second process, i.e., the one with the carbide mixture.

Titanium evaporated during the production of alloys in both processes. It is possible to produce alloys with 2-4% Ti by the first process if an excess of titanium dioxide is introduced into the initial charge. It is possible to obtain alloys having approximately 1% Zr by this same process by introducing an excess of zirconium oxide when its content in the alloy is increased.

The attempt to obtain aluminum alloys under the conditions of the experiments ended in failure due to the very high vapor pressure of aluminum ($1 \cdot 10^{-1}$ mm Hg even at 1374°C).

As was shown above, all the alloys were prepared in conditions for obtaining unalloyed niobium. Perhaps, in the case of alloys of Nb and Ti and great amounts of Zr and V, these conditions are not optimal and their more precise definition will allow alloys of these metals to be produced closer to a preset composition.

The data of Tables 1 and 2 show that higher quality alloys, stable with respect to O, N, and C content, are obtained in the case of reduction at 2100°C.

Niobium-base alloys which are reducible by the carbothermic method usually have porosity in the range of 40-45%. It is expedient to remelt them in electron-beam furnaces in order to shape and more conveniently to pressure-treat and additionally refine them. As Table 3 shows, the content of the impurities O, N, and, in some cases, C is essentially lowered in this case, the amount of the alloying metals W and Mo is practically unchanged, and the amount of Zr with its small (approximately 1%) content, as well. When the Zr content is greater than 1.5%, part of it evaporates during melting -- this must be taken into consideration by increasing the amount of zirconium dioxide in a charge to be reduced. When necessary, the reduced alloys may be reprocessed by the well-known method of unalloyed Nb, including hydrogenation of alloys, pulverization, dehydrogenation, pressing of moldings and vacuum sintering conforming to the conditions for the sintering of Nb, but at temperatures 150-200°C below the melting point of the alloys.

The work which has been conducted testifies to the outlook for the process of producing niobium-base alloys by joint reduction using the carbothermic method: this method allows the stages of first producing the pure metals and then their alloys to be eliminated and ensures the uniformity of distribution of the alloying metals and the production of ingots of high cleanness during the first remelting in the electron-beam furnace. At the same time, the cost of niobium-base alloys is not increased by this method but, conversely, even drops in comparison with the method using unalloyed niobium when a significant amount of cheaper alloying metals is used.

Table 1

Conditions for Carrying Out and Results of the Experiments (1st Process)

(a) Номинальный состав сплава, % (ниобий—основа)		(b) Температура восстановления, °C	(c) Содержание, % (вес.)			
			(d) легирующий металл	C	N	O
Mo	4,5	1900	4,8	0,03	0,02	0,02
	4,5	1920	4,5	0,04	0,05	0,08
	4,5	2100	4,8	0,06	0,01	0,01
	5	1900	4,8	0,02	—	0,42
	6	2000	5,5	0,02	—	0,2
Mo	4,5	—	4,25	—	—	—
	—	1900	—	0,14	0,17	0,08
Zr	0,8	—	—	0,7	—	—
Mo	4,5	—	4,9	—	—	—
	—	1900	—	0,03	—	0,01
Zr	2,5	—	1,2	—	—	—
Mo	5	—	4,9	—	—	—
	—	1900	—	0,04	0,09	0,04
Zr	3	—	1,85	—	—	—
Mo	4,5	—	4,4	—	—	—
	—	2000	—	0,04	0,06	0,04
Zr	2,5	—	1,35	—	—	—
Mo	4,5	—	4,6	—	—	—
	—	2100	—	0,03	0,05	0,02
Zr	2,5	—	0,87	—	—	—
W	6	1900	5,7	0,02	0,07	0,10
	15	1900	13,3	0,04	0,09	0,11
	17	1930	15,4	0,05	0,04	0,13
	15	1960	15,0	0,02	0,05	0,19
	15	2100	13,5	0,05	0,02	0,01
	177	2100	15,4	0,02	0,02	0,03
	20	2100	19,0	0,03	0,04	0,01—0,08

(Continued)

(Continuation)

(a) Номинальный состав сплава, % (ниобий-основа)		(b) Температура восстановления, °C	(c) Содержание, % (вес.)			
			(d) легирующий металл	C	N	O
W	15	—	14,1—14,8	—	—	—
Mo	5	1900	5,0—5,2	0,14	0,03	0,01
Zr	1	—	1,0—0,93	—	—	—
W	15	—	14,05	—	—	—
Mo	5	1900	5,0	0,02	0,04	0,32
Zr	1	—	1,1	—	—	—
Ti	5	1900	2,2	0,02	—	0,02
W	28	—	24,0	—	—	—
	—	1960	—	0,03	0,08	0,08
Ti	7	—	4,5	—	—	—
W	28	—	26,4	—	—	—
	—	2100	—	0,02	0,02	0,06
Ti	7	—	3,0	—	—	—
V	10	1930	5,3	0,05	—	—
	10	2100	4,3	0,02	0,03	0,02
V	3	—	3,0	—	—	—
	—	1900	—	0,01	0,07	0,05
Al	3	—	0,5	—	—	—
V	3	—	3,0	—	—	—
	—	2050	—	0,05	0,03	0,01
Al	3	—	0,5	—	—	—

Legend:
(a) Nominal composition of alloy, % (niobium-base)
(b) Reduction temperature, °C
(c) Content, weight percent
(d) Alloying metal

Table 2

Conditions for Carrying Out and Results of the Experiments (2nd Process)

(a) Номинальный состав сплава, % (основа—ниобий)		(b) Температура восстановления, °C	(c) Содержание, %			
			(d) легирующий металл	C	N	O
Mo	4,5	1900	5,0	0,03	0,03	0,05
	4,5	2100	4,0	0,03	—	0,08
	5	1950	4,9	0,01	0,02	0,08
	6	2000	5,4	0,03	0,03	0,43
	6	2100	6,2	0,10	0,02	0,02
Mo Zr	5 — 1	— 1900 —	4,8 — 0,97	— 0,02 —	— 0,03 —	— 0,32 —
Mo Zr	5 — 1	— 2000 —	4,9 — 1,0	— 0,01 —	— 0,01 —	— 0,27 —
W	6	1900	5,8	0,04	—	0,17
	6	2000	5,9	0,02	0,05	0,25
	15	2100	14,0	0,04	0,07	0,03
	17	2000	16,3	0,01	0,02	0,09
	25	2000	24,6	0,02	0,02	0,2
W Ti	15 — 7	— 1900 —	17,0 — 2,07	— 0,05 —	— — —	— — —

(Continued)

(Continuation)

(a)		(b)	(c)			
			(d)	C	N	O
W Ti	15 — 7	— 2100 —	15,75 — —	— 0,02 —	— 0,01 —	— 0,06 —
W Ti	25 — 7	— 1900 —	20,1 — 3,8	— — —	— — —	— 0,01 —
W Ti	25 — 7	— 2000 —	21,8 — 3,45	— — —	— 0,11 —	— 0,01 —
W Ta	5 — 2	— 2100 —	5,0 — 2,0	— 0,04 —	— 0,06 —	— 0,01 —
V	5	1900	4,8	0,01	—	0,32
	5	2000	4,8	0,01	—	0,03
	6	2000	5,8	0,01	—	0,40
Al V	3 — 3	— 1800 —	0,45 — 3.5	— — —	— — —	— — —
Al V	3 — 3	— 1900 —	0,4 — 3,5	— 0,01 —	— — —	— 0,06 —

Legend:
(a) Nominal composition of alloy, % (base-niobium)
(b) Reduction temperature, °C
(c) Content, %
(d) Alloying metal

Table 3

The Behavior of Alloying Metals and the Refining of Carbothermic Alloys During an Electron-Beam Melt *

(a) Содержание в восстановленном сплаве, %						(b) Содержание в слитке, %					
W	Mo	Zr	C	N	O	W	Mo	Zr	C	N	O
14,5	—	—	0,04	0,09	0,11	14,4	—	—	0,01	0,01	0,01
—	4,6	—	0,04	0,10	0,12	—	4,6	—	0,01	0,01	0,01
—	4,9	1,8	0,04	0,09	0,04	—	4,16	1,2	0,02	0,01	0,03
—	—	1,3	0,03	0,04	0,03	—	4,2	1,1	0,01	0,01	0,03
—	4,4	1,2	0,02	—	0,14	—	3,9	1,1	0,02	—	0,04
—	4,9	1,2	0,03	—	0,10	—	4,9	1,1	0,03	0,02	0,06

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* Melt experiments were conducted in the laboratory furnace by A. V. Yelyutin.

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