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FOREIGN TECHNOLOGY DIVISION

ACADEMY OF SCIENCES OF THE USSR. INSTITUTE OF METALLURGY.
STRUCTURE AND PROPERTIES OF REFRACTORY METALLIC MATERIALS

(SELECTED ARTICLES)

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ABSTRACT

Creep in columbium at high stresses at temperatures ranging from 550 to 1000 degree C was studied. The material studied was remelted in a vacuum arc furnace; it had the following impurity content (wt percent): 0.21 percent Ta, 0.09 percent Ti, 0.08 percent Si, and 0.09 percent Fe. After solidifying, the ingots were forged from a diameter of 34 mm down to 8 mm, and samples were made. One set of samples was tested in the as-forged condition, and another set was vacuum annealed at 1400 degree C for 10 hr and then tested. Creep testing was done in a vacuum at constant stress. The logarithm of the creep rate is given as a function of the reciprocal of the absolute temperature for stresses ranging from 21 to 64 kg/mm (to the second power). A linear dependence occurred for each stress and an activation energy was calculated from the slope of the dependence. By comparing the activation energies at high stresses with the activation energies of vacancy motion, self-diffusion, and vacancy formation, it was concluded that the creep of columbium at high stresses and at the temperatures studied is controlled by the thermally activated motion of vacancies to dislocations which undergo slip. Orig. art. has: 2 figures, 1 table, 3 formulas.

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ABSTRACT

The structure and phase composition of a nickel-chromium alloy containing columbium, molybdenum, titanium and aluminum were studied. Compositions of the carbides and gamma prime-phase are presented for five different heat treatments. After water quenching from 1000 or 1120 degree C, the alloying elements were fixed in solid solution and a small part of the primary NbCN phase occurred. Slow cooling from these temperatures caused a 10-14 percent decomposition of the gamma prime-phase, and aging increased this amount to 20 percent. The phase composition of the gamma prime-phase varied with heat treatment, although each of the component elements were present. The impact energy of EI437B and the nickel-chromium alloy with columbium is given as a function of aging time at temperatures of 600, 650, 700 degree C. The presence of columbium retarded diffusion and consequent coagulation of the gamma prime-phase, as indicated by given phase compositions of the nickel-chromium alloy for different holding times at high temperatures and under stress. Microstructures are shown of the alloy after quenching and aging. Primary carbides and gamma prime-phase particles were distributed in a matrix of solid solution. As aging proceeded, the gamma prime-phase particles thickened and coagulated. Orig. art. has: 2 figures, 2 tables.

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ABSTRACT

A study is made of the resistance to oxidation in air of alloys on a niobium base which have been proposed by various investigators for use at temperatures of 800, 900, 1000, and 1200 C. Of the studied alloys prepared by arc melting, the most resistant at temperatures of 800-1000 C are alloy 21 (Nb, 45 percent Ti, 14 percent W, 4.3 percent Al, 4 percent Cr, 0.7 percent Mn) and alloy 28 (Nb. 41 percent Ti, 4 percent W, 5 percent Al, 3 percent Cr, 3 percent V, 1 percent Mn); the weight increment to these alloys in the indicated temperature range fluctuates from 7 to 36 mg/cm (to the second power) per 100 hrs. At 1200 C all the alloys studied have low resistance to oxidation, except alloy 15 (Nb, 40 percent Ti, 5 percent Mo, 8 percent Cr, 5 percent Al, 1 percent Mn), whose weight increment after 150 hrs. of testing is 91 mg/cm (to the second power). Of the alloys containing niobium the alloys 3 (Nb, 16 percent Ti, 4 percent Zr) and 12 (Nb, 10 percent Ti, 3 percent V, 3.75 percent Mo, 1.25 percent Cr) exhibit more than 70 percent greater resistance to oxidation at 800-1200 C. The effect of a number of elements (tungsten, molybdenum, chromium, aluminum, manganese, carbon) on the oxidation resistance of niobium alloys is demonstrated for multicomponent alloys containing 40-45 percent Ti. Orig art. has: 3 figures and 1 table.

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COLUMBIUM CREEP AT HIGH STRESSES

A. F. Orlov

In some preceding works [1-3] the authors examined the creep of a number of metals and alloys with a face-centered cubic lattice at relatively low temperatures and high stresses. It was established that in the region of high stresses the creep-activation energy at first decreases almost linearly with the growth of stresses, and then ceases to depend on the stresses, reaching a constant maximum value. The numerical evaluations of this value for Al, Pt, Ni, and Ni-Cu alloys support the assumption in [4] that in this region the creep-activation energy in the established stage is determined by the energy of the migration of mobile point defects of the crystalline lattice. The task of the present work consisted of determining the creep characteristics of columbium at high stresses and determining how much does the above mentioned assumption prove to be correct when we pass over to metals with a body-centered lattice.

As the material for the investigation we selected ceramic columbium remelted in a vacuum arc furnace. The content of the principal impurities (in weight percent) is the following: 0.21 Ta, 0.09 Ti, 0.08 Si, 0.09 Fe. After smelting, the rods were forged from a diameter of 34 mm to 8 mm, and then the specimens were machined from them. In this case the external oxide layer of the metal was removed. A part of the specimens was used directly for the tests, the other part was subjected to annealing in a vacuum for 10 hours at a temperature of 1400°C.

The tests were carried out in a VPN-S2 apparatus [6] under a ~ 10^{-4}/mmHg vacuum, under constant stress conditions. The creep of annealed columbium was studied in the 700-1,000°C range, at stresses from 21.0 to 33.6 kg (force)/mm^2. In the case of rapid application of a load corresponding to a stress of 33.6 kg (force/mm^2 the specimens ruptured, even at temperatures near room temperature. However, it was discovered that in the process of slow loading (the stress was increased on the average by 0.25 kg (force/mm^2 per hour), accompanied by brief periodic annealing at temperatures of about 300°C, the specimens were often strengthened and sustained for a long time the maximum load at creep-testing temperatures. Investigation of creep at higher stresses (up to 64 kg (force/mm^2) was carried out on deformed specimens in the temperature range from 550 to 900°C.

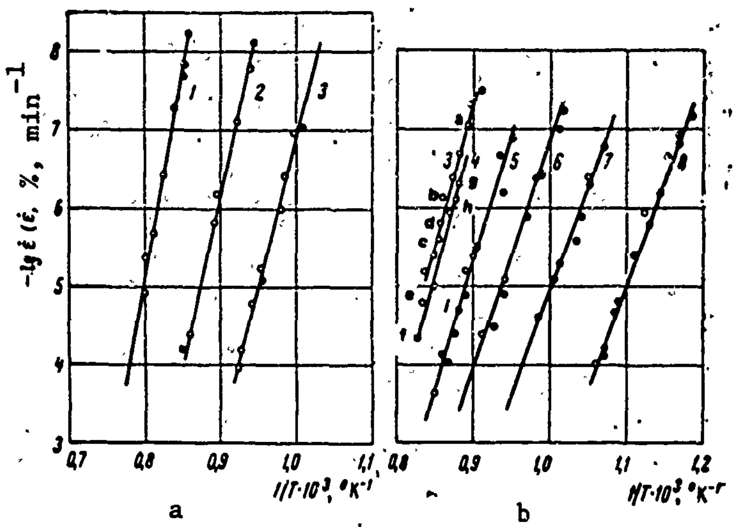

Figure 1. Dependence of the rate of creep of annealed (a) and deformed (b) columbium on temperature at different stresses. 1, 21.0 kg (force)/mm^2; 2, 29.0; 3, 33.6; 4, 33.6; 5, 42.4; 6, 51.4; 7, 56.3; 8, 64.0. Letters a-i signify the ordinal number of the tests on a single specimen.

The results of the tests are given in Figure 1, a and b in the form of the dependence on the inverse temperature of the logarithm of the rate of creep at the established stage of annealed and deformed Nb. For annealed columbium the slope of the straight lines decreases monotonically with the increase of stress, which corresponds to the decrease of the creep activation energy. In the case of deformed columbium this dependence is observed up to the stress of 56.3 kg (force)/mm^2, and upon a further increase of the applied stress the slope of the straight lines within the bounds of accuracy of the experiment, remains constant. The values of creep activation energy and the pre-exponential coefficient, given in the table, were determined with the conventional equation

$$\dot{\varepsilon} = A \exp(-Q/RT), \quad (1)$$

where ε is the rate of creep at the established stage in %·min^{-1}; Q is the creep-activation energy in cal/gram-atom; R is the gas content; T is the temperature in °K.

Values of creep-activation energy and pre-exponential coefficient

State	σ,kg (force)/mm^2	Q,Kcal/g-atom	ln A
Annealed columbium Deformed columbium	21,0 20,0 33,6 33,6 42,4 51,4 56,3 64,0	102,0 85,7 74,8 72,3 63,5 53,4 53,7 53,4	36,5 32,8 30,9 24,5* 22,5 21,6 21,0 23,5

* Without annealing in the process of testing (see below).

It is characteristic that with a stress of 33.6 kg (force)/mm^2 the creep activation energies of annealed and deformed columbium are approximately equal, whereas their creep rates at different temperatures differ by almost two orders of magnitude. We were not able to verify the correspondence of creep energy activation for annealed and deformed columbium under other stresses. As it had been mentioned earlier, at greater stresses annealed specimens break down even at room temperature. On the other hand, at relatively low stresses an appreciable rate of creep of deformed specimens is observed only at high temperatures, close to the recrystallization temperature. The nature of the change in the rate of creep of one specimen of deformed columbium in successive testing by the temperature-cyclic method (experimental points for σ = 33.6 kg (force/mm^2 in Figure 1,b), apparently indicates that at such temperatures in the process of creep a weakening of the deformed specimens occurs during the steady stage. In this experiment the total creep time, at temperatures corresponding to points e and f, amounted to about six hours.

In Figure 2 we show the dependence of creep activation energy on the value of the stress applied. In the range of stresses between 21 and 33 kg (force)/mm^2, activation energy is a decreasing linear function of the applied stress value. In this range the rate of creep may be described with sufficient accuracy by the following equation

$$\dot{\varepsilon} = A_1 \exp\left(-\frac{Q - \gamma\sigma}{RT}\right), \quad (2)$$

where γ = 2.3 cal·mm^2/gram·gram-atom. With the further increase of stresses the rate of decrease of the activation-energy reduces and, starting at 55 kg/mm^2, the activation energy becomes constant. On the sector with a constant activation-energy value we observe the exponential law of dependence of the rate of creep on the value of applied stress

$$\dot{\varepsilon} \sim \sigma^n. \quad (3)$$

The evaluation of the exponent in this relationship according to the logarithm of the pre-exponential coefficient yields the value of n = 18.7.

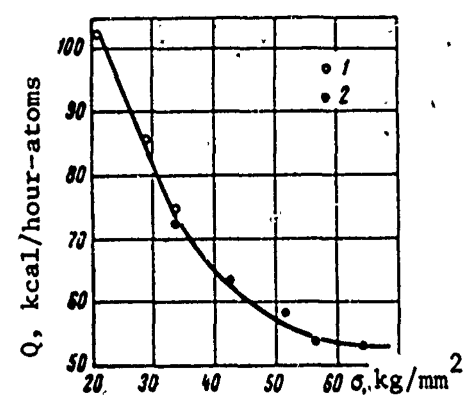

Figure 2. Dependence of creep-activation energy of columbium on the value of the stress applied.
1, annealed samples;
2, deformed samples.

A similar nature of the change in the activation energy in the applied stress function was noted earlier [3,5], and the constant value of activation energy at high stresses was examined as the maximum value characteristic for the given metal. As it had been indicated earlier, this value was compared with the values of the energy parameter q, calculated on the basis of the hypothesis on local melting [5], and with the activation energies of the motion of point defects (vacanci and interstitial atoms) of the crystalline lattice. For columbium q ≈ 46.8 kcal/gram-atom [5] and the activation energy of motion of interstitial atoms E_i = 27.7 kcal/gram-atom [7]. The energy of formation of vacancies $E_{f.v}$ in columbium is equal to 47.0 kcal/gram-atom [8], and the activation energy of self-diffusion $E_{s.d}$ on columbium, according to the data of various authors amounts to 97-106 kcal/gram-atom. Then, according to the $E_{s.d}-E_{f.v}$ difference we find the activation energy of the motion of the vacancies $E_{m.v}$ in columbium, which is equal to 50-59 kcal/gram-atom.

As we see from Figure 2, the maximum value of columbium creep-activation energy at high stresses amounts to about 53 kcal/gram-atom. Such a coincidence gives us grounds for assuming that, just as in the metals with a face-centered cubic lattice, columbium creep at high stresses is controlled by energy-activated motion of point defects in the crystalline lattice to dislocations, which perform the slipping. In this case such flaws apparently are vacancies. Utilizing the results of work [9] we can estimate that for our case (deformation of columbium at the rate of 10^{-2} to $10^{-4}\%\cdot min^{-1}$ at the temperature of 600°C), the concentration of the vacancies originating in the process of deformation should exceed the equilibrium concentration at the given temperature by at least 11-13 orders. The origination of supersaturated concentration of vacancies in columbium in the process of low-temperature plastic deformation was observed experimentally by Foudreaus [10].

It is characteristic that in our experiment a noticeable rate of creep of deformed columbium was observed only at temperatures above 500°C, although according to the data of [11] the strength characteristics of columbium and change little in the range between room temperature at 600°C. At the same time, according to the evaluation of the authors of work [7] we should expect that monovacancies in columbium become mobile in the regions of 350-500°C temperature range. This comparison also indicates the participation of the vacancies in the process of columbium creep in the temperature range investigated at high stresses. At lower temperatures (below 300°C) the deformation point effects in columbium are practically immobile, and the mechanism controlling the rate of creep is the overcoming of Payerls-Nabarro barriers through energy fluctuations [12].

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SPECIFIC FEATURES OF ALLOYING, STRUCTURE, AND PHASE COMPOSITION OF NICKEL-CHROMIUM HEAT-RESISTANT ALLOY WITH COLUMBIUM[1]

K. I. Tereknov

The great majority of multialloyed heat-resistant steels and alloys includes in its composition a large number of alloying elements, included in the solid solution (Ni, Cr, Mn, Mo, Co, W) or those forming the basic strengthening intermetallic γ'-phase (Ti-Al) and partially the carbide phase (Cr, Ti, Nb, C, V).

It was considered that in the first stage of development of dispersion-hardening heat-resistant nickel-chromium alloys the principal strengthening agent was titanium; then it was discovered that aluminum acts more effectively in this sense and plays a decisive role in the formation of the γ'-phase.[2] Aluminum and nickel form a compound of the berthollide type Ni_3Al, having a lattice similar to a solid solution lattice, but with a greater γ'-phase parameter. Titanium can dissolve in greater amounts in this solution, the parameter of the γ'-phase lattice increasing somewhat.

The significance of such an extremely important element as columbium for stabilization of the solid solution and formation of the principal strengthening phase had been underestimated for a long time, and therefore, the group of heat-resistant steels and alloys, alloyed with columbium, is relatively small both in our country (EI481, EI607) and abroad (Inco-718, Rene-62 (USA); g-18B and G-32B (Britain).

The investigations of a series of heat-resistant alloys containing 2-3% columbium in combination with 2.5-3% of molybdenum and a high aluminum content of 1.3-1.6% in comparison with the EI437 alloy (Al = 0.5-0.90%) performed by us jointly with N. I. Kurkina in 1953-1954,

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1,2, See p. 15.

enabled us to develop an original heat-resistant alloy without cobalt and tungsten with high mechanical and heat-resistant properties (up to 750° C) in the most important working-temperature range of 550-750° C.

With respect to its mechanical and heat-resistant properties our alloy exceeds the best British disk alloy Nimonik-90 with an 18% Co content, and is equivalent to more multialloyed alloys: Rene-41, Waspalloy, etc (USA) containing 10-14% cobalt and 4-10% molybdenum. The number of alloying elements forming the basic strengthening γ'-phase in the alloys compared is about equal: in them the titanium content is 2.5-3.0%, aluminum content is 1.5%, and the base is nickel.

From literature sources we know that with many respects columbium acts similarly to other alloying elements (Mo, W, Co); it has a very effective influence on the strengthening of the solid solution, slows down the diffusion processes and raises greatly the recrystallization temperature, which ensures a substantial rise in the increase of the resistance to plastic deformation at high temperatures.

According to the data of investigations by N. F. Lashko and G. G. Georgiyeva, more than one half of the columbium (≈1%) is included in the composition of the intermetallic γ'-phase; about one third is included in the solid solution, and a part of it (0.2-0.3%) is contained in the primary carbide phase of the Nb(CN) type.

However, the effect of columbium on the structure of heat-resistant steels and alloys proved the most important. Coarse liquation accumulations consist of excessive phases: carbonitrides, titanium oxides, and chromium borides. The principal liquation elements in heat-resistant alloys with the nickel base are titanium and aluminum, and in part boron and carbon.

The presence of 1.5-2% columbium combines the carbon in the early stages of crystallization and in this way changes the physicochemical properties of the mother liquid, creating the conditions for less isolation of the liquating elements, according to the investigation of V. P. Stepanov et al.

The presence of high-melting primary columbium alloys, which are practically insoluble up to 1,250-1,300° C, retards the intensive growth of the grain when the alloy is heated and in this way excludes the formation of coarse zonal variable-grain structure both in the process of hot-pressure working, and when the alloys are heated for quenching.

Study of the Phase Composition of the Nickel-Chromium Alloy

From the data of the phase analysis (Table 1) it follows that immediately after quenching from temperatures of 1,000 and 1,120° C (cooling in water) mainly the solid solution and a small amount of the NbCN carbide phase are fixed, which are not dissolved even upon heating to 1,250° C. Slow cooling in the air from the tempering temperatures results in a considerable separation of the strengthening γ'-phase: after a single tempering the amount of γ'-phase $\approx$ 10%, after double tempering $\approx$ 14.5%.

Subsequent aging at temperatures of 750-775° C for 16 hours results in a further and more copious separation of the strengthening γ'-phase; its total content increases to 20%, i.e., it doubles in comparison with its temperate state.

Table 1. Phase content of a nickel-chromium alloy after various heat-treatment conditions

I Режимы термической обработки	II Содержание легирующих элементов в γ'-фазе, % к весу металла							III Содержание элементов в карбидной фазе, %	
	Ni	Ti	Al	Nb	Cr	Mo	e, %	Ti	Nb
1120°—8 час., воздух IV + 1000°—12 час., вода V . .	0,10	0,04	сл.	сл.	сл.	сл.	0,14	0,05	0,22
1120°—8 час., воздух IV	8,37	0,85	0,4	0,53	0,41	0,10	10,6	0,05	0,11
1120°—8 час., воздух + +1000°—4 час., воздух .	11,8	1,05	0,53	0,60	0,37	0,09	14,4	0,06	0,13
1120°—8 час., воздух + +1000°—4 час., воздух+ 750—775°—16 час., воздух	15,7	1,58	1,05	1,1	0,68	0,18	20,20	0,06	0,18
1120°—8 час., воздух + +1000°—4 час., воздух+ 750°—16 час., воздух + 650°—16 час. VI	18,0	1,62	1,10	1,18	0,72	0,72	22,8	0,06	0,17

Legend I--Heat-treatment conditions; II--alloying element content in the γ'-phase, in % of the weight of the metal; III--element content in the carbide phase, %; IV--hours, air; V--hours, water; VI--hours; VII--traces.

The double aging conditions increase somewhat the total γ'-phase content to 22%, the separation of the γ'-phase occurring in the more finely-dispersive state, which increases substantially the strength characteristics (σ_B; $\sigma_{0.2}$; H_B) and the prolonged strength of the EI-698 alloy in the entire working-temperature range up to 750° C.

From the analysis of the alloying element content in the γ'-phase it follows also that its main components are Ni, Ti, Al, and Nb, the last two elements being included in the γ'-phase in almost equal amounts. Chromium and molybdenum are partially included in the γ'-phase, remaining mainly in the solid solution of the alloy. The amount and composition of the strengthening carbide phase vary relatively little. After double or single tempering its amount is about equal to 0.2%.

According to the data of the X-ray diffraction analysis performed under the direction of N. F. Lashko, two basic phases are present in the alloy: the strengthening intermetallic γ'-phase Ni_3(Al, Nb, Ti, Cr, Mo) and the carbide phase Nb(CN). Very small amounts of titanium nitrides TiN and the boride phase Cr_2B_3 are also fixed.

It is interesting to note that this is the only nickel-base heat-resistant alloy in which we do not find cubic chromium carbides of the $Cr_{23}C_6$ type, located predominantly along the grain boundaries, and which are the principal cause of the sharp drop in the value of the impact ductility of heat-resistant alloys in the process of their heat embrittlement. Apparently, all the carbon in the alloy is absorbed by the columbium and titanium, and is therefore insufficient for the formation of chromium carbides.

According to the data of our investigations and the results of the study of heat embrittlement of the EI-437B alloy, impact ductility values reach 1.5-1.2 kg(force)/cm^2 when the alloy is soaked for a long time, from 50 to 500 hours in the 550-650° C temperature range, and still remain at this level for up to 5,000 hours without undergoing changes. In a heat-resistant alloy with 2% columbium the impact ductility value is not less than 3.5-4 kg(force)/cm^2 even after soaking for 10,000 hours at temperatures of 650, 700 and 750° C (Figure 1).

The presence of columbium retards the diffusion processes and inhibits with especial strength the process of coagulation of the γ'-phase, which in itself proceeds quite slowly in the regions of moderate temperatures, of 600-750° C.

In this respect the results of the phase analysis of the alloy in relation to the temperature and the great aging length of 3,000 to 10,000 hours are given in Table 2. [3]

From the data in Table 2 we can see that even very long soaking, lasting from 3,000 to 10,000 hours at temperatures of 650, 700 and 750° C has no effect on the relationships of the principal alloying elements in the γ'-phase. Its amount increases appreciably in comparison with the initial state, to 19-20% instead of 14.56%.

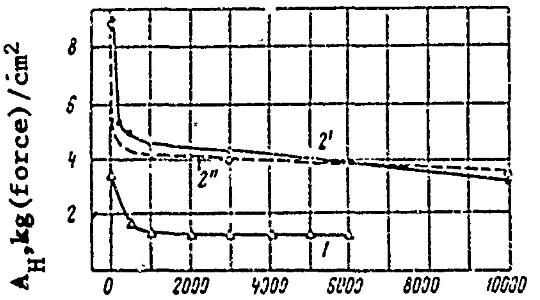

Figure 1. Characteristic of impact ductility in the process of prolonged aging. 1--EI-437B, soaking at 600°C; 2--nickel-chromium alloy, soaking at 650°C (2') and 700° C (2").

The total content of alloying elements in the carbide phase also increased to 0.4-0.43% in comparison with the initial state of ≈0.20%, the increase occurring mainly through columbium. The data on the phase analysis, obtained with the combined action of temperatures and stresses during a relatively long testing time, lasting from 2,000 to 4,000 hours at the temperature of 700° C, are of a substantial interest. The resistance of the specimens under a stress σ = 33 kg(force)/mm^2 at this temperature amounted to more than 4,000 hours, and with σ = 38 kg (force)/mm^2, to about 2,000 hours.

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[3] See p. 15.

Table 2. Phase content of a nickel-chromium alloy after prolonged soaking at high temperature and under a stress

I Температура, время выдержки и напряжение	II Содержание элементов в γ'-фазе, % к весу металла							Содержание элементов в карбидной фазе, % к весу металла III				
	Ni	Ti	Cr	Mo	Al	Nb	ε, %	Ti	Cr	Mo	Nb	ε, %
IV Исходное состояние после термообработки по режиму: V 1120°—8, 1000°—4 час. +800°—16 час. . . .	11,55	1,22	0,30	0,13	0,76	0,60	14,56*	0,08	0,02	0,03	0,24	0,37
VI Тоже+650°—3000 час.	14,90	1,55	0,35	0,16	0,9	0,90	19,76	0,08	0,03	0,03	0,28	0,42
» +700°—3000 час.	15,29	1,60	0,37	0,18	—0,9	0,98	19,40	0,08	0,037	0,03	0,26	0,40
» +700°—10000 час.	14,96	1,54	0,38	0,17	1,02	0,92	19,2	0,08	0,03	0,04	0,24	0,40
» +750°—3000 час.	14,58	1,54	0,37	0,15	1,09	0,95	18,68	0,08	0,03	0,03	0,25	0,39
» +750°——10000 час. VII	14,44	1,52	0,39	—0,16	1,05	0,96	18,52	0,08	0,04	0,04	0,25	0,41
» 700, σ=38 кГ/мм²; 4040 час. V	15,2	1,50	0,40	0,16	1,12	0,94	19,82	0,09	0,06	0,03	0,26	0,44
» 700, σ=38 кГ/мм²; 2000 час: V VII	15,3	1,54	0,40	0,20	1,10	0,95	19,70	0,09	0,05	0,03	0,26	0,43

Legend: --Temperature, soaking time and stress; II--Element content in γ'-phase, in weight percent of the metal; III Element content in the carbide phase, in % of the weight of the metal; IV--Initial state after heat treatment under the conditions: V--hours; VI--same; VII--kg (force)/mm^2

* In this batch the content of Al, Ti and Nb, i.e., of the principal elements forming the γ'-phase was at the low-set temperature (800° C), used previously, caused a certain coagulation of the strengthening γ'-phase.

At the temperature of 650° C and prolonged soakings there is an additional strengthening of the alloy connected with the separation of the strengthening phase in the more fine-despersive stage, which causes a certain increase of the strength characteristics and hardness and an appreciable decrease of plasticity and impact ductility in comparison with the initial state.

The above-mentioned alloying features of the alloy, its structural and phase transformations, as well as the high thermal stability give us grounds to consider that under the conditions of prolonged operation it will prove the most reliable and efficient material for the turbine disks of engines carrying a heavy temperature load and stationary gas turbines.

Metallographic Analysis

The microstructure was studied under an optical and an electron microscope with magnifications of 3,000 and 10,000 respectively. After heat treatment the alloy represents a solid solution plus γ'-phase, plus primary carbides of the Nb (CN) type. The separated particles of the γ'-phase are very fine-dispersive and are not detected at a magnification of 300 after etching (Figure 2, a). After very long aging, with soaking of 3,000 and 10,000 hours, the γ'-phase particle is enlarged and the etching ability of the alloy increases. At 700° C and soaking for 3,000-10,000 hours (Figure 2, b) we noted an enlargement of the γ'-phase (0.1-0.15 micron). At 750° C a coagulation of γ'-phase particles occurs, which is connected with weakening (Figure 2, c).

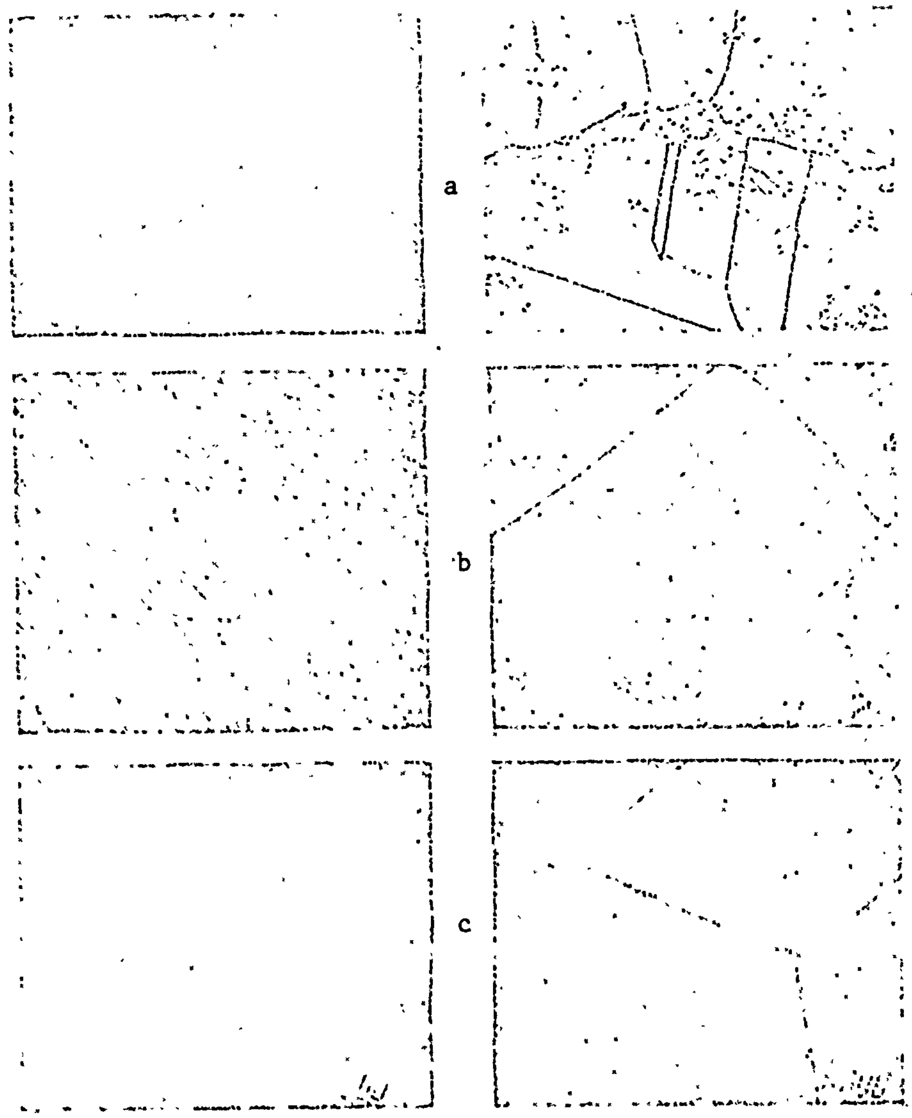

Figure 2. Microsturucture of the nickel-chromium heat-resistant alloy. a--tempering and aging, ×300, ×1000; b--tempering + aging 700°C, 10,000 hours, ×300, and ×1000; c--tempering + aging 750°C, 10,000 hours, ×300, and ×1000.

Footnotes

1. To p. 8. The experimental part of the investigation was performed with the participation of N. I. Kurkina, A. P. Ozerova, G. G. Georgiyeva, and E. V. Polyak.

2. To p. 8. According to the data of S. T. Kishkin, N. F. Lashko, et al.

3. To p. 12. The specimens were aged and tested for their prolonged strength by B. Ye. Levin and I. G. Taubina.

COMPARATIVE STUDY OF OXIDABILITY AND HEAT RESISTANCE OF COLUMBIUM-BASE ALLOYS

O. S. Ivanov, I. I. Rayevskiy, M. A. Skryabina

Columbium is a prominent metal with respect to its physicomechanical properties: its high melting point (2,415°C) assures its use as the base for heat-resistant alloys, its relatively low specific gravity (8.4 grams/ cm^3) gives it an advantage in application in flight machinery, and some other instances, and its high plasticity makes it possible to work columbium easier than such metals as tantallum, molybdenum, and tungsten. All these circumstances attracted in the last decade a great attention to the problem of practical uses of columbium and of developing alloys based on it [1-6].

In the A. A. Baykov Institute of Metallurgy of the Academy of Sciences USSR a number of investigations were carried out on the study of the structure and properties of columbium alloys. Thus, a study was made of the effect of two to ten atomic percent of Ti, V,Cr, and Mo on the kinetics of oxidation and gas saturation of the surface layer of columbium alloys with 10 and 30 atomic % of zirconium at 500 and 1,000°C in the air atmosphere [6]. An examination was made of the microstructure, malleability at 1,100°C, hardness at 1,000°C, recrystallization temperature, deformability in the cold state, and heat resistance of alloys (made by arc- and electron-beam smelting) of the columbium corner of the Nb-W-V system with tungsten and vanadium content up to 40 weight %. The oxidative ability in air and the depth of penetration of gases for alloys of columbium with 0.4-3 weight % of Cr + Ti were determined. An investigation was made of the microstructure, malleability at 1,100°C, recrystallization, deformability in the cold state, hardness at 400-1,000°C, and corrosion-resistance in water and carbon dioxide of high parameters binary alloys Nb-Ta (up to 35 weight % Ta), Nb-Mo (up to 20 weight % Mo), ternary columbium alloys of the Nb-Ta-W systems (up to 35 weight % of Ta + W) and Nb-Mo-W (up to 20 weight % Mo + W) smelted in an argon-arc furnace. An examination was made of the microstructure, malleability at 1,000°C, deformability in the cold state during rolling, recrystallization, tensile strength at 800°C, oxidability in air at 800°C, and corrosion in water at high parameters of alloys of the columbium corner of the ternary system Nb-Ti-Zr (up to 30 weight % Ti + Zr) smelted in an argon-arc furnace. In a similar way we investigated the alloys and plotted the isothermic cross sections of the constitution diagram of the ternary system Nb-Zr-Cr (up to 30 weight % Zr + Cr). We studied the oxidibility in air at 500-1,000°C and in water and carbon dioxide at high parameters, of columbium alloys of the ternary system Nb-V-Zr including those additionally alloyed with Mo, Ti, Cr, and W and also alloys of the Nb-Cr-Mo system additionally alloyed with Be, Ce, V, and Ti. In all the investigations the alloys optimum with respect to their properties were determined.

Taking into account the large number of columbium alloys offered for practical use, it appeared more advisable to carry out comparative testing thereof with respect to the combination of workability, heat resistance, and strength at high heat. Below we show the result of the first stage of this comparative study.

Table 1. Compositions of the alloys studied

Alloy Number	Composition, weight %	Sources
1	Nb—4Ti+1Zr	
2	Nb+8Ti+2Zr	
3	Nb+16Ti+4Zr	
4	Nb+3,3Mo+1,7W	
5	Nb+1,7Mo+3,3W	
6	Nb+7,5W+2,5Ta	Experimental specimens
7	Nb+5Mo	
8	Nb+5V	
9	Nb+3.3Mo+3,1Al	
10	Nb+3,1Mo+4,9Zr	
11	Nb+3,2Mo+1,5Al	
12	Nb+10Ti+3V+3,75Mo+1,25Cr	
13	Nb+7Ti+20W+3Mo+1Zr	(8)
14	Nb+7Ti+20W+3Mo+1Zr+1Mn	
15	Nb+40Ti+5Mo+8Cr+5Al+1Mn	
16	Nb+40Ti+10W+8Cr+5Al+1Mn	Experimental specimens
17	Nb+49Ti+5Mo+10Cr+5Al+1Mn	
18	Nb+44Ti+10W+10Cr+5Al+1Mn	
19	Nb+20Ti+10Mo+1Zr	
20	*FS*-85(9), 26—*F*-50(10), 27—*F*-48(4)	
21	Nb+45Ti+14W+4,3Al+4Cr+0,7Mn	
22	Nb+44,7Ti+14W+4,3Al+4,0Cr+0,7Mn+0,3C	
23	Nb+40Ti+5Mo+8Cr+1Mn	Experimental specimens
24	Nb+40Ti+5Mo+5Al+1Mn	
25	Nb+40Ti+5Mo+1Mn	
28	Nb+41Ti+3Cr+3V+5Al+4W+1Mn	
29	Nb+4Mo	
30	*Cb*-74(9), 32—*D*-43(11)	
31	BH-2	

In Table 1 we give the composition of the alloys investigated. Alloys 1-12 are taken from the works mentioned above. Some alloys are known from foreign works or are variations of alloys proposed by the authors[1].

Preparation of Alloys and Investigation Procedures

Preparation of Alloys

The alloys were smelted in an arc furnace with an unconsumed tungsten electrode in an argon atmosphere. Each ingot weighed 100 grams. For better mixing of the components the alloys of every composition were first smelted in the form of 25 gram ingots. The ingots remelted 4-5 times, were thereupon successively fused with one another. Alloys containing additives of such components as Cr, Al, V, and Mn were made in the arc furnace under a high x-argon pressure (400-500 mm Hg), which made it possible to reduce their volatilization during smelting. For composition control the alloys were weighed after smelting, and a part of the alloys was subjected to selective chemical analysis. Cast alloys containing much titanium (45%) and tungsten (14-20%) exhibited the existence of strong liquation. After homogenization, to which we subjected all the alloys without exception, liquation disappeared, which was established by microstructure analysis. Homogenization was carried out in a TVV-4 furnace at a 1,500°C temperature and under a 10^{-4} mm/Hg vacuum, for 3-5 hours. The alloys were not subjected to plastic deformations.

Method of Evaluating the Resistance to Heat of Alloys

The specimens for investigating the heat resistance of alloys were prepared in the form of cylinders about 10 mm high and 5-6 mm in diameter. The surface of the specimens, after they were made, was subjected to electrolytic polishing. The resistance of the alloys against oxidation in air was determined by the method of periodic weighing and the evaluation was made according to the increase in weight per unit of area of the specimens' surface. The alloys were oxidized in a celite furnace in Al_2O_3 crucibles previously calcined to constant weight. The temperature of the furnace was maintained with an accuracy of ±10°C and controlled with a platinum-platinorhodium thermocouple.

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[1] See p. 26.

Evaluation of the Strength of Alloys at High Temperatures

The strength of alloys at high temperatures was determined on the basis of measurement of hardness at temperatures of 800, 900, 1,000, 1,100, 1,200, 1,300, and 1,400°C. Hardness was measured on a machine designed and built by the Institute of Metallurgy, in a 10^{-4} mm Hg vacuum. The specimens for hardness measurement had a cylindrical shape with a diameter of about 14 mm and height of 4-5 mm. The end of the specimens, on which the testing was performed, was polished. As the indenter we used a sapphire crystal ground in the form of a four-faced pyramid with an apex angle of 136°. The load on the indenter amounted to 2.5 kg (force). The diagonals of the imprints produced were measured under a microscope after the specimens were cooled and hardness was calculated according to the data of these measurements. The temperature of the specimen was measured by a W-(Mo + Al) thermocouple in a set with a PP-type potentiometer. The hot junction of the thermocouple was placed in an indentation made in the lower end of the specimen.

Investigation Results and Their Discussion

All the values given here on oxidation of the alloys and on their hardness at high temperatures are averages of the results of measurements of two or three parallel specimens made from an ingot of the same batch.

Resistance of Alloys to Oxidation at 800, 900, 1,000, 1,200°C

In Figure 1, a, we show the resistance of alloys to oxidation at 800°C. At this temperature the best alloys (21 and 22) are of the following compositions: Nb + 45 Ti + 14 W + 4.3 Al + 4 Cr + 0.7 Mn, Nb + 44.7 Ti + 14 W + 4.3 Al + 4 Cr + 0.7 Mn + 0.3 C. These alloys have a high titanium content in the presence of W, Al, and Cr and differ from one another only by the presence of 0.3% C in one of them (alloy 22). Their gain in weight after 100 hours amounts to 7.5 and 22 mg/cm^2. The somewhat inferior resistance of alloy 22 should be fully attributed to the presence of carbon in it. Good resistance is also exhibited by alloy 28 with the composition: Nb + 41 Ti + 3 Cr + 3V + 5 Al + 4W + 1 Mn and binary alloy 8 with 5% V. However, the latter at higher temperatures has little resistance to oxidation. A good oxidation resistance is exhibited also by alloys 15 and 19, which contain 40 and 20% Ti in combination with Mo, Zr, Al, Cr, and Mn. Their gain in weight after 100 hours amounts to 84 and 50 mg/cm^2 respectively. Alloy

6, containing 7.5 W and 2.5 Ta has little oxidation resistance at 800°C. Its gain in weight after 20 hours amounts to 289 mg/cm^2. Very poor oxidation resistance was exhibited by the well-known high-strength alloys FS-85 (alloy 20), F-50 (alloy 26) and F-48 (alloy27). Alloy FS-85 oxidizes more than unalloyed columbium.

The best at 900°C are multicomponent alloys (alloys 16, 18, 21, 22, and 28) containing (in %) 40-45 Ti, 4-14 W, and additives of 3-10 Cr, 4-5 Al, 0.7-1 Mn, and 3 V. Not one of the alloys among the most oxidation-resistant at 900°C, contains any molybdenum. For example, by comparing the oxidability of alloys 15 and 16 we can see that replacement of 10 weight % W with 5 weight % Mo (in atomic percent, about equal amounts of tungsten and molybdenum) results in a deterioration of heat resistance. Alloys 16 and 18 containing in addition to chromium, aluminum, and manganese, 10% tungsten, show a gain in weight after 150- hour tests of 15 and 21 mg/cm^2 respectively. Alloys 21 and 22 with the highest titanium and tungsten content which were the best at 800°C, remain the best at 900°C as well. The most heat-resistant is alloy 21 (Nb + 45 Ti + 14 W + 4.3 Al + 4 Cr + 0.7 Mn): its weight increase after 150-hour tests amounts to 11 mg/cm^2. Alloy 17, of a similar composition, containing molybdenum instead of tungsten, is much less heat resistant.

In Figure 1, b, we give data on oxidability of alloys at 1,000°C. At this temperature the best are alloys 28 (Nb + 41 Ti + 3Cr + 3V + 5Al + 4W + 1 Mn) and 21 (Nb + 45 Ti + 14W + 4.3 Al + 4 Cr + 0.7 Mn). The gain in weight of these alloys in 100 hours amounts to 26 and 37 mg/cm^2 respectively. It is interesting to note that with respect to oxidation resistance at 1,000°C, the group of multicomponent alloys, containing 40 and more % Ti, is arranged in the following sequence in order of decrease of heat resistance: alloy 28 (41 Ti - 4 W), 21 (45 Ti - 14 W), 18 (44 Ti - 10 W), 16 (40 Ti - 10 W, 15 (40 Ti - 5 Mo),and 17 (49 Ti - 5 Mo). Replacement of tungsten by molybdenum again decreases sharply the oxidation resistance of the alloys. The most heat-resistant of the alloys containing over 70% Nb is alloy 3 (Nb + 16 Ti + 4 Zr). The worst oxidation resistance at 1,000°C is exhibited by Cb-47 (alloy 30), which at this temperature is less heat resistant than unalloyed columbium, and the Nb + 7.5 W + 2.5 Ta alloy.

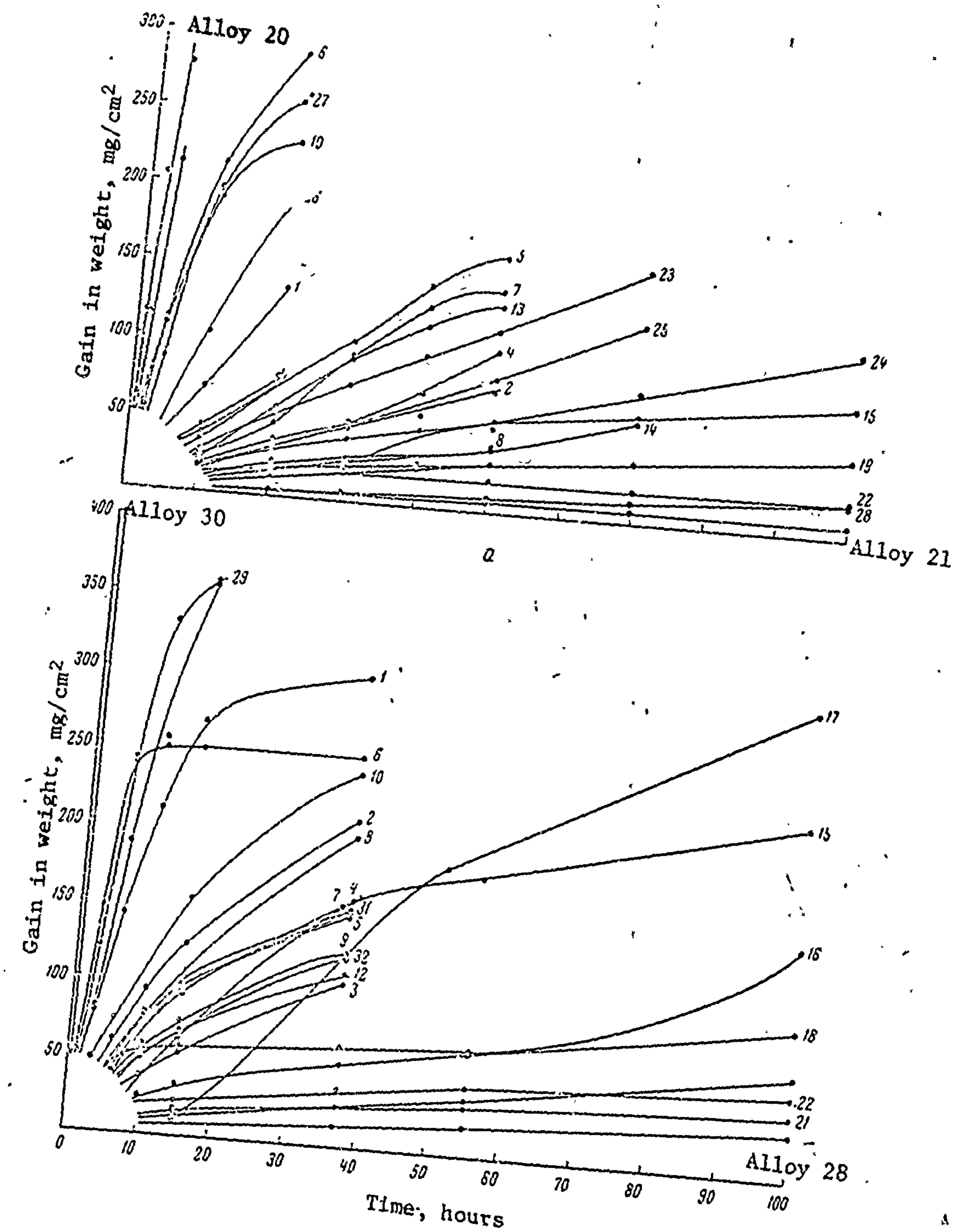

Figure 1. Oxidation resistance of alloys.

Almost all alloys are little oxidation-resistant at 1,200°C and are destroyed already after 10 to 20-hour tests. The exception are only alloys 15 (Nb + 40 Ti + 5 Mo + 8 Cr + 5 Al + 1 Mn) and 17 (Nb + 49 Ti + 5 Mo + 10 Cr + 5 Al + 1 Mn), the weight gain of which after 150-hour tests, amounts to 91 and 149 mg/cm^2. Alloys 21 and 22 with 45% Ti, 14% W, and additions of aluminum, chromium, and manganese which were the most heat resistant at 800 and 1,000°C, disintegrate rapidly even in the first hours of oxidation at 1,200°C. Of the alloys containing over 70% Nb, the best at the temperature of 1,200°C is the alloy with the composition of Nb + 10 Ti + 3V + 3.75 Mo + 1.25 Cr.

The results of the heat-resistant tests of alloys in air at temperatures of 800-1,200°C bring out a group of the most heat-resistant alloys with a gain in weight of up to 100 mg/cm^2. Alloys of this group are divided into two subgroups, one of which contains tungsten and the other, molybdenum. These multicomponent alloys containing a large amount of titanium, in the presence of tungsten in them, prove more resistant to oxidation at temperatures of 800-1,000°C than alloys of a similar composition containing molybdenum. At 1,200°C the picture changes, and alloys containing molybdenum are more resistant.

Our results on oxidation resistance at 800-1,000°C temperatures cannot be compared to the data obtained in the works of Sims and Klopp [7], in which the harmful influence of tungsten on the oxidation resistance of columbium at 1,000°C was established.

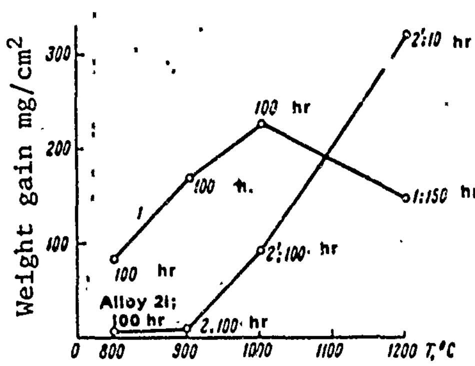

Figure 2. Effect of additions of molybdenum (1) and tungsten (2) on oxidation resistance of alloys at 800-1,200°C temperatures. 1, Alloy 15 (Nb + 40 Ti + 8 Cr + 5 Al + 1 Mn + 5 Mo); 2, Alloy 21 (Nb + 45 Ti + 4 Cr + 4.3 Al + 0.7 Mn + 14 W); 2', Alloy 2' (Nb + 44 Ti + 10 Cr + 5 Al + 1 Mn + 10 W).

In Figure 2 we show that our data also support the sharp increase of oxidability of the alloys containing tungsten upon passing from 900° to 1,000°C, by almost one order. The favorable influence of molybdenum on heat resistance at high temperatures was noted in the work of Argent and Phelps [12], in which this is attributed to sintering of the Mo (MoO_3) oxide.

Additions of 8-10% chromium are less effective than additions of 3-5% aluminum. This corresponds to the data in the work by Sims and Klopp [7], who explain the low effectiveness of additions of chromium to columbium in the amount of less than 20% by the fact that chromium has about the same affinity to oxygen as columbium. According to our data, heat resistance is increased the most by the joint addition of chromium and aluminum in the total amount of 10-12%.

Using the example of alloy 14, which has the composition of Nb - 7 Ti - 20 W - 3 Mo - 1 Zr - 1 Mn and differs from alloy 13 only by the presence of 1% Mn, we can speak about the favorable effect of the latter on the heat resistance of the alloy at 800°C, inasmuch as the introduction of 1% of manganese almost doubles the heat resistance in this case.

Strength of Alloys at High Temperatures

The analysis of the relationship between the hardness of alloys and the 800-1,400°C. temperature shows that all the alloys containing a large amount of titanium have low hardness at high temperatures. Beginning with 50 kg(force)/mm^2 at 800°C the hardness drops to 1-2 kg(force)/mm^2 at 1,300-1,400°C. Alloys containing over 70% Nb and additions of tungsten, molybdenum, tantalum and chromium, lose their hardness to a lesser extent. The strongest ones are alloys F-48 (Nb - 15 W - 5 Mo - 1 Zr - 0.1 C), F-50 (Nb - 15 W - 5 Mo - 5 Ti - 1 Zr - 0.1 C), and also alloy 14 (Nb - 7 Ti - 20 W - 3 Mo - 1 Zr - 1 Mn). Their hardness at 1,400°C lies within the range of 40-50 kg(force)/mm^2. Introduction of 1% Mn into the Nb - 7 Ti - 20 W - 3 Mo - 1 Zr alloy makes it somewhat harder at temperatures up to 1,200-1,300°C, however, this effect is not observed at 1,400°C.

In Figure 3 we present a diagram on which we give the data on heat resistance and hot hardness of most of the alloys studied by us. The best combination of heat-resistance and hot hardness at temperatures of 1,200-1,400°C is possessed by the alloy of the following composition: Nb - 7 Ti - 20 W - 3 Mo - 1 Zr - 1 Mn.

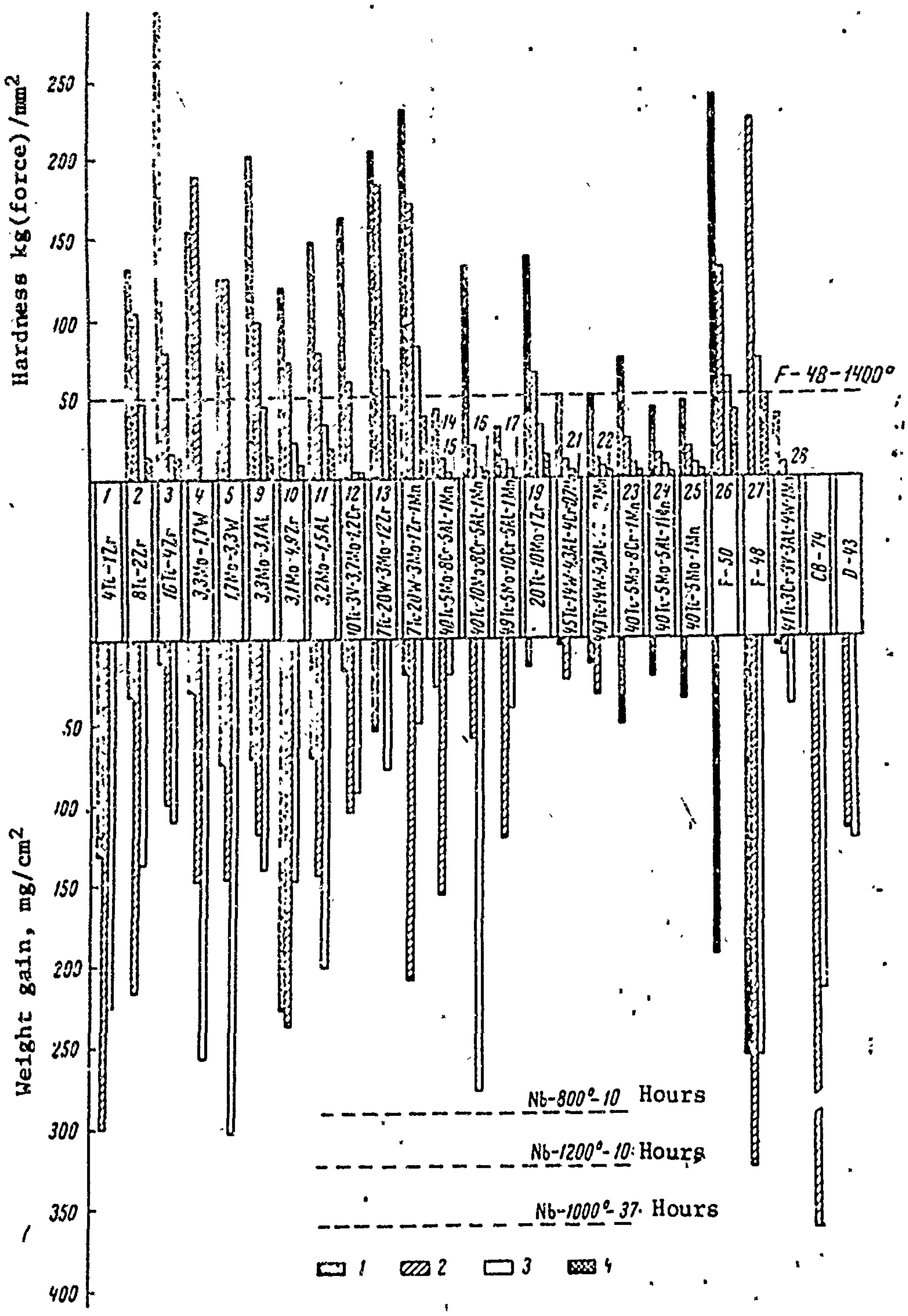

Figure 3. Heat resistance and hardness of alloys at temperatures of 800-1,400°C. 1--800°C, 2--1,000°C, 3--1,200°C, 4--1,400°C.

Conclusions

1. A study was made of the oxidation resistance in the air of columbium-base alloys, offered by various researchers, at temperatures of 800, 900, 1,000 and 1,200°C. Among the alloys studied, prepared by means of arc smelting, the most resistant at temperatures of 800-1,000°C. are: Nb - 45 Ti - 14W - 4.3 Al - 4 Cr - 0.7 Mn (alloy 21) and Nb - 41 Ti - 4 W - 5 Al - 3 Cr - 3V - 1 Mn (alloy 28). The gain in weight of these alloys in the temperature range mentioned fluctuates between 7 and 36 mg/cm^2 in 100 hours. At 1,200°C, all the alloys studied are little-resistant against oxidation, with the exception of the alloy Nb - 40 Ti - 5 Mo - 8 Cr - 5 Al - 1 Mn (alloy 15), its weight gain after 150-hour testing amounting to 91 mg/cm^2. Among the alloys containing over 70 weights % of columbium the best oxidation resistance at 800-1,200°C. temperatures is exhibited by alloys Nb-16Ti-4Zr (alloy 3) and Nb - 10 Ti - 3V - 3.75 Mo - 1.25 Cr (alloy 12).

We determined the effect of a number of elements (tungsten, molybdenum, chromium, aluminum, manganese, and carbon) on the oxidation resistance of columbium alloys. For multicomponent alloys containing 40-45 % Ti at temperatures of 800-1,000°C additions of tungsten are more favorable than additions of molybdenum. At 1,200°C, to the contrary, molybdenum additions are more favorable.

2. We evaluated the strength at high temperatures of certain alloys made by means of arc smelting on the basis of the study of hardness at 800-1,400°C temperatures. It was established that the strongest at high temperatrues are alloys F-50 and F-48, as well as the alloy Nb - 7 Ti - 20 W - 3 Mo - 1 Zr - 1 Mn (alloy 14). Their hardness at 1,400°C lies within the range of 40-50 kg(force)/mm^2.

3. Among the columbium-base alloys studied we should note the alloy Nb - 7 Ti - 20 W - 3 Mo - 1 Zr - 1 Mn (alloy 14) is the optimum with respect to the combination of oxidation resistance and strength at high temperature, whereas such high-strength alloys as F-48 and F-50 differ little from unalloyed Nb with respect to oxidation resistance.

Footnotes

1. To p. 18. Some of the alloy variations (alloys 15-18, 21, 22, and 28) were proposed for study by L. I. Gomozov. Alloy 31 was received from the All-Union Scientific-Research Institute of Aviation Metal Research.

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