Metal Science and Heat Treatment, Vol. 54, Nos. 11 – 12, March, 2013 (Russian Original Nos. 11 – 12, November – December, 2012)

HEAT TREATMENT

UDC 621.78:669.295.5

EFFECT OF THE METHOD OF HEAT TREATMENT ON FORMATION OF STRUCTURE AND PROPERTIES OF REFRACTORY TITANIUM ALLOYS

A. A. Popov,[1] I. V. Narygina,[1] and M. A. Popova[1]

Translated from *Metallovedenie i Termicheskaya Obrabotka Metallov*, No. 12, pp. 20 – 24, December, 2012.

Special features of formation of structure and properties in pseudo-α-titanium alloys are considered as a function of the time of treatment in the β-range.

Key words: refractory titanium alloys, structure, phase composition, ordering, silicides.

INTRODUCTION

Modern commercial refractory titanium alloys can serve at a temperature exceeding 600°C, which is insufficient for operation in advanced aircraft engines. One of the methods for raising the limiting temperature of the use of such alloys is creation of an $(\alpha + \alpha_2)$ structure in them, which would reduce the residual deformations due to long-term operation of the material. However, an α_2-phase is segregated in commercial alloys in the form of fine particles distributed uniformly over the body of grains, which causes embrittlement of the material and hinders its application in the industry. It has been shown in [1] that an ordered α_2-structure in the form of individual domains with an up to micrometer size arises in some model titanium pseudo-α-alloys after electrocontact heating to the β-range and subsequent cooling to room temperature. Alloys with such a structure possess high enough toughness characteristics. At elevated aging temperatures the authors have observed conventional dispersed segregations of an α_2-phase, and the alloys in such condition were embrittled. The aim of the present work was to study the effect of nonequilibrium composition of α-phase on the forming structures of model titanium alloys with elevated content of aluminum.

[1] Ural Federal University in the Name of the First President of Russia B. N. Eltsyn, Ekaterinburg, Russia (e-mail: a.a.popov@.ustu.ru).

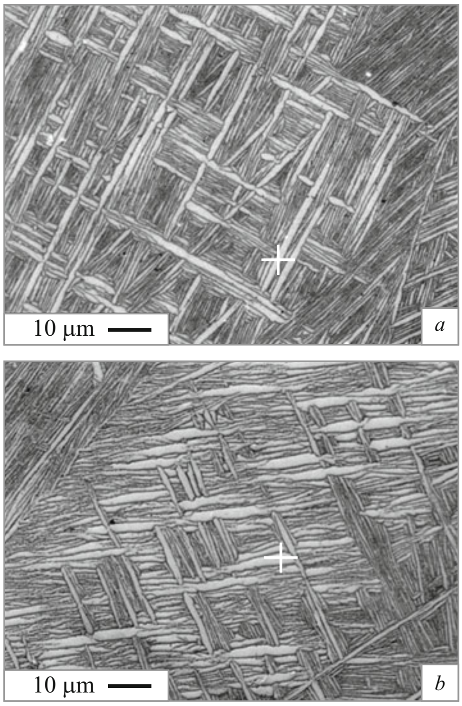
METHODS OF STUDY

We studied experimental alloys of the Ti – Al – Sn – Zr – Mo – Si system with chemical compositions presented in Table 1. Bars 20 mm in diameter with initially lamellar structure were heated to the β-range with a hold of 10 or 60 min and then cooled in air. The aging was conducted at 500 – 700°C at a step of 50°C with a hold of up to 85 h in laboratory muffle furnaces.

The structure was studied on end sections of the bars by the methods of diffraction and scanning electron microscopy with the help of JEM-2100C and Jeol JSM-6490LV microscopes, respectively, with an energy dispersive analyzer of the composition. The x-ray diffraction analysis was performed using a "Brukker D8 Advance" diffractometer in copper K_α radiation. The microhardness was measured using an MHTX CSM Instruments device by the method of Oliver and Farr at a load of 8 N.

TABLE 1. Chemical Compositions of Studied Alloys

Conventional number of alloy	Content of elements, wt.%							Aluminum equivalent, wt.%
	Al	Sn	Zr	Mo	Nb	Si	Fe	
1	7.0	4.0	7.0	0.5	0.5	0.10	–	10
2	8.3	–	2.2	2.1	–	0.25	0.15	11



Element	wt.%	at.%
Al_{K_α}	8.91	15.12
Si_{K_α}	0.41	0.68
Zr_{L_α}	2.71	1.36
Mo_{L_α}	2.63	1.26
Ti_{K_α}	85.33	81.59
Fe_{K_α}	0.00	0.00

Element	wt.%	at.%
Al_{K_α}	9.46	15.96
Si_{K_α}	0.16	0.26
Zr_{L_α}	2.20	1.10
Mo_{L_α}	2.18	1.03
Ti_{K_α}	85.78	81.47
Fe_{K_α}	0.21	0.17

Fig. 1. Microstructure and local chemical composition in different regions of α-phase (*a*, *b*) of specimens of alloy *2* after heating to the β-range and cooling in air (the crosses mark the places of the analysis).

RESULTS AND DISCUSSION

Preliminary studies have shown that a hold for 10 min in the single-phase range does not cause formation of a homogeneous β-solid solution, and cooling yields a whole spectrum of structures differing in the composition of the phases and in the morphology of the structural components. For example, determination of the concentration of elements in various regions of the α-phase by the method of microscopic x-ray spectrum analysis has shown an up to 1.0% difference in the aluminum concentration (Fig. 1). Increase of the hold in the β-range promotes formation of a more homogeneous solid solution, and the structure of the alloy after cooling is uniform enough. The difference in the homogeneity of the structures results in different values of the microhardness. For example, the microhardness of alloy *2* after a short hold in the β-range and air cooling is 390 *HV*, whereas after a 60-min hold and the same cooling it is 375 *HV*.

The difference in the initial structures results in some special features of aging of the alloys, which is manifested the most vividly in the aging process occurring at a relatively low temperature (500 – 550°C). Complex structures form in the alloys with nonuniform chemical composition of the α-phase (Fig. 2). The plates of the α-phase contain stretched rectilinear dislocations, the content of which increases with the aging time (Fig. 2*a*). In addition, stacking faults are encountered (Fig. 2*c* and *d*). In individual microvolumes we observe dislocations of a paired type and individual segments of dislocation lines, the contrast of which allows us to classify them as elements of anti-phase boundaries (Fig. 2*b*, *d*, and *f*). Microdiffraction analysis of these regions shows the presence of reflections from an α_2-phase, though particles of this phase have not been detected.

In the cases when the aged alloy has a more uniform distribution of alloying elements over the section of primary

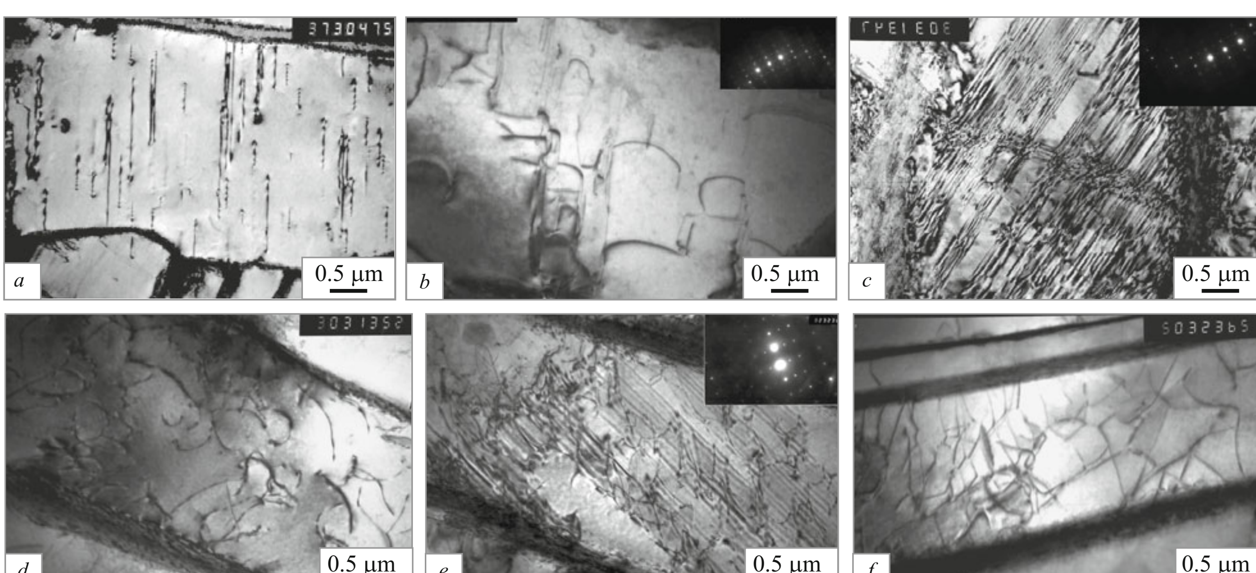


Fig. 2. Microstructure of the alloys after a short hold in the single-phase range and aging at 550°C for: *a*, *b*, *c*) 10 h; *d*, *e*) 25 h; *f*) 85 h.

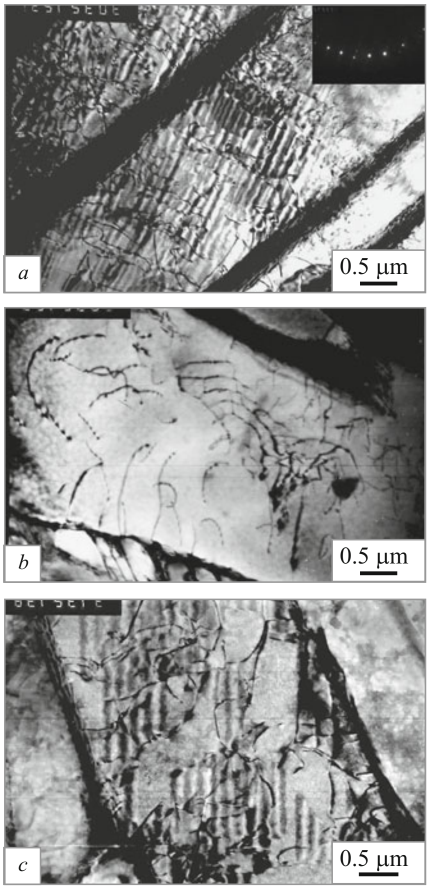


Fig. 3. Microstructure of the alloys after a long hold in the single-phase range and aging at 550°C for: *a*) 10 h; *b*) 85 h.

plates of the α-phase, the forming structure is more homogeneous, elements of anti-phase boundaries are encountered more rarely and after longer holds (Fig. 3*b*). In some microvolumes we observe a striated contrast bounded by dislocation lines (Fig. 3*a* and *c*). Dark-background analysis of such regions in superstructural reflection shows that the contrast changes for an inverse one, which allows us to classify them as individual domains of an ordered structure.

When the aging temperature of the alloys is increased to 650°C, their fine structure changes (Fig. 4). In the first turn this is manifested by segregation of silicide particles over layers of β-phase (Fig. 4*c*). The segregated particles have structural formula $(Ti, Zr)_5Si_3$, and their content is the higher the higher the temperature and the longer the duration of the aging. The segregation is accompanied by fragmentation of β-layers and formation of boundaries perpendicular to their plane surfaces, which disturbs their continuity due to the growth of particles of the primary α-phase in aging. As a result, the initial β-plates break into separate particles (Fig. 4*d*).

It should be noted that the lattice parameter of the β-phase at aging temperatures of 500 – 600°C decreases from 0.3249 to 0.3237 – 0.3239 nm depending on the aging temperature. However, we detected no particles of primary α-phase. This allows us to assume that the β-solid solution decomposes not by nucleation and growth of new particles but due to growth of plates of the primary α-phase.

Aging at the temperatures tested is accompanied by some growth in the hardness of the alloys (Fig. 5). The increase in the hardness of the alloys with initially more homogeneous structure is higher and occurs more rapidly than in the alloys subjected to a short hold in the β-range. The absence of segregations of particles after aging allows us to think that the growth in the hardness is caused primarily by increase in the volume fraction of the stronger α-phase. In addition, the decomposition of the β-solid solution during aging is accompanied by its enrichment with zirconium atoms, the size of which is substantially larger than that of titanium atoms, which contributes in its turn into the solid-solution hardening.

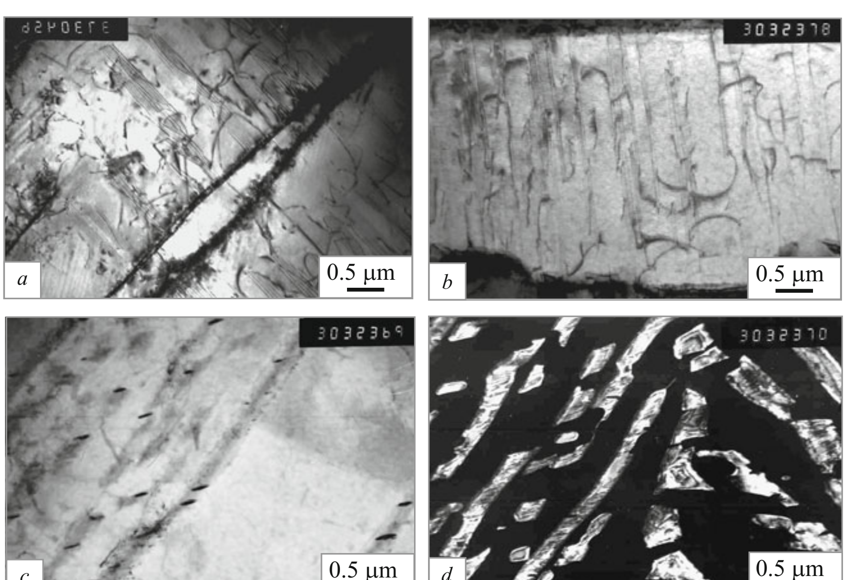


Fig. 4. Microstructure of the alloys after a short hold in the single-phase range and aging at 650°C for: *a*, *b*) 10 h; *c*) 25 h; *d*) 85 h (dark-background image in the light of reflection (220) of the β-phase).

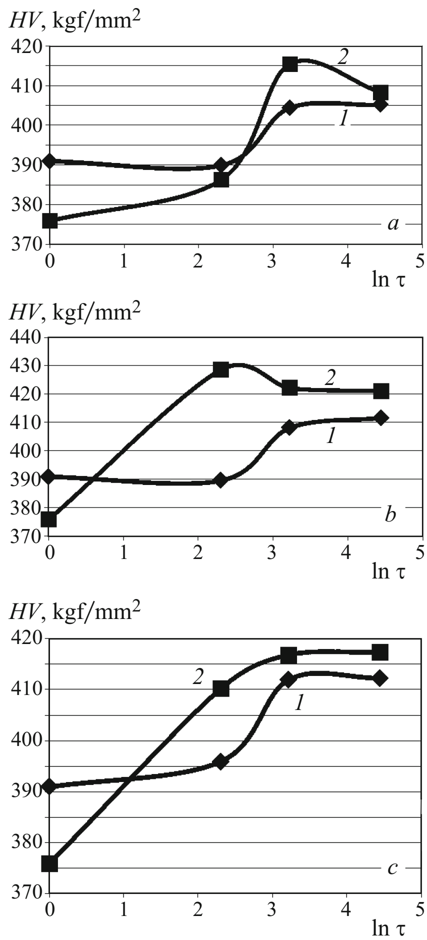


Fig. 5. Microhardness of alloy *2* after aging at 550 (*a*), 600 (*b*), and 700°C (*c*): *1*, *2*) hold in the single-phase range for 10 min and 1 h, respectively.

The structure of the alloys after aging at 700°C differs substantially from the structure formed in the alloys after ag-

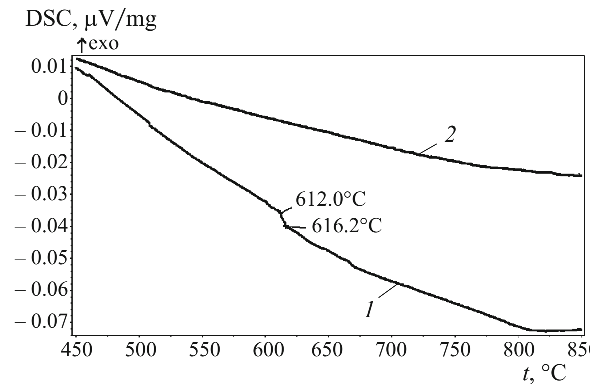


Fig. 7. Parts of the thermograms of cooling of alloy *1* from the β-range after preliminary high-temperature treatment with short-term (*1*) and long-term (*2*) holds.

ing at lower temperatures. The main difference is the absence of regions with anti-phase boundaries and the appearance of a high content of fine segregations of α_2-phase over the body of plates of the α-phase. The segregating particles are distributed uniformly over the body of the β-plates, and even after a 85-h hold at 700°C their size does not exceed 10 – 15 nm (Fig. 6*a* and *c*). In the layers of the α-phase the segregation of silicide particles continues, and their size and content is higher than after aging at lower temperatures (Fig. 6*b* and *d*). Aging at this temperature is not accompanied by a noticeable decrease in the lattice parameter, but the monotonic growth of the hardness continues in the whole of the range studied (see Fig. 5*c*). In our opinion the hardening in the given case is primarily determined by segregation of fine particles of α_2-phase in contrast to the causes considered above.

Figure 7 presents the data of differential scanning calorimetry on variation of the heat content obtained in cooling

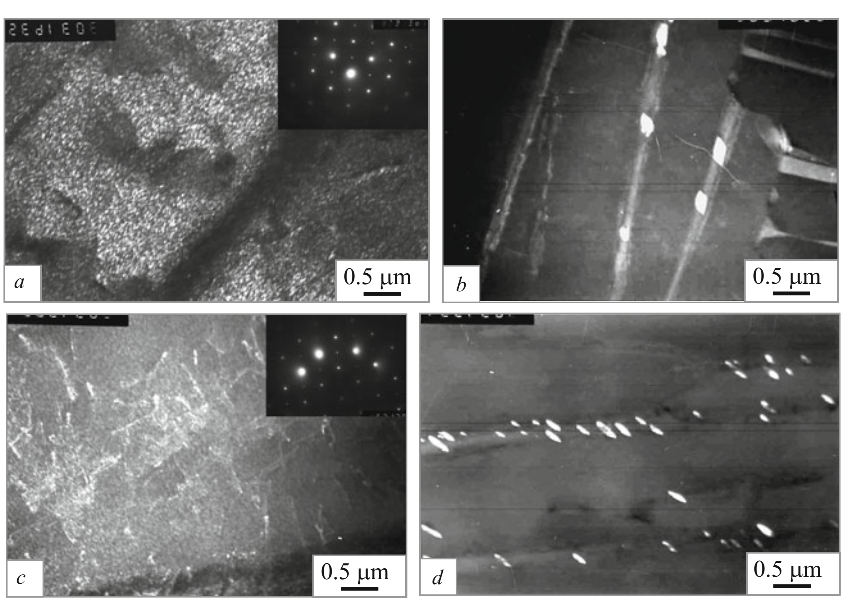


Fig. 6. Microstructure of the alloys after a short hold in the single-phase range and aging at 700°C for: *a*, *b*) 25 h; *c*, *d*) 85 h; dark-background images in the light of reflections (100) of the α_2-phase (*a*), $(4\bar{3}2)$ of the S_1-phase (*b*), (110) of the α_2-phase (*c*), and (101) of the S_1-phase (*d*).

of alloy *1* from the β-range. It can be seen that in the range of 800 – 450°C the alloy treated from the β-range with a hold of 1 h does not exhibit endo- and exothermic effects, whereas the alloy held in the β-range for 10 min exhibits a marked inflection at a temperature of 620°C, which is typical for phase transformations of the second kind. These results confirm indirectly the fact of ordering of an alloy with nonequilibrium structure in cooling [2].

CONCLUSIONS

It has been proved that the aging-induced transformations in the supersaturated α-solid solution of titanium pseudo-alpha-alloys with a high aluminum equivalent occur by different mechanisms depending on the aging temperature. At a low temperature the transformation is homogeneous and yields ordered relatively coarse regions of α_2-phase, whereas increase of the aging temperature to 700°C changes the mechanism of the transformation and the α_2-phase forms by the mechanism of nucleation and growth. This is the most obvious for the alloys where a more nonequilibrium structure forms due to a preliminary treatment in the β-range.

The work has been performed with support of the Russian Foundation for Basic Research, Grant RFFI-Ural No. 10003-96073.

REFERENCES

1. A. A. Popov, N. G. Rossina, M. A. Popova, and A. V. Volkov, “Processes of ordering in high-temperature titanium alloys,” *Titan*, No. 1, 36 – 42 (2001).
2. B. G. Livshits, V. S. Kraposhin, and Ya. L. Linetskii, *Physical Properties of Metals and Alloys* [in Russian], Metallurgiya, Moscow (1980), 320 p.