Ordering Process in Heat-Resistant Titanium Alloys

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The methods of diffraction electron microscopy have shown that long-range order sections are formed at aging temperatures up to 500°C in alloys high in aluminum, and the ordered phase is formed by nucleation and growth mechanism at 700°C aging temperatures. Causes of changing the phase transformation mechanism have been discussed, and the relationship between the structure and properties of alloys, depending on the modes of processing has been analyzed. It is shown that toughness and ductility characteristics are improved due to the realization of homogeneous transformation mechanism, while the formation of particles by the heterogeneous mechanism results in the alloys embrittlement.

Keywords: Heat-resistant titanium alloy, ordering, a2-phase

1. Introduction

The structural investigation of heat-resistant titanium alloys is usually focused on the morphology and behavior of the main phases α and β . However, in these alloys, both different silicide particles can be released1,2), and formation Ti3 Al particles in (phase can be^{3,4)}. All these precipitates can significantly effect on the properties of the alloys. For example, the formation of $(T_i, Z_r)_5 S_{i_3}$ silicides on the interphase α/β boundaries decreases the technological plasticity of the alloys10, and the formation of (Ti, Zr) Si3 particles in the body of α phase decreases the characteristics of heat resistance²⁾. In the cases when the ordering processes occur in the α phase and Ti₃Al particles (α_2 phase) are formed, heat resistance characteristics increase, but plastic properties degrade drammaticaly³⁾. However, the precipitation of α2 phase may occur not as isolated particles, the formation of which is carried out by the mechanism of nucleation and growth, but may result from the second kind phase transformation as a consequence of the ordering processes. In this case, the properties complex of heat-resistant titanium alloys on the basis of α solid solution will be largely determined by the mechanism of the processes leading to the formation of $(\alpha + \alpha_2)$ -structure. In this context, the aim of this paper is to study the mechanisms of α_2 phase domains formation depending on the temperature-time parameters of heat treatment.

2. Materials end Experimental Procedure

Experimental Ti-Al-Sn-Zr-Mo-Si alloys, containing up to 12.5 at. % aluminum were used. Aluminum alloy equivalent ranged from 8 to 10.5, with a β -stability coefficient of 0,7. The deformation of 20 kg ingots was performed using standard technology, which involves a combination of alternating β -deformation and deformation in the $(\alpha + \beta)$ -region. Final deformation of the alloys was carried out by rolling in a two-phase region at temperatures T_{μ} - $(20\text{-}30)^{\circ}$ C. The heat treatment of alloy was performed using an electro-contact technique up to t 1150 °C with 30 seconds time delay

followed by air cooling. Aging was carried out at t 500...700℃ with 1-100 hours time delay in a laboratory muffle furnace.

The structure was studied on samples cut perpendicular to the rolling direction. The main investigation methods were diffraction and scanning electron microscopy, performed on JEM-2100C and JSM6490 with energy analyzers, respectively. X-ray diffraction analysis was performed using a diffractometer (Bruker D8 Advance) in the Cu K^a radiation.

3. Results and Discussion

Electron microprobe analysis of alloys, following electric-heating showed that such rapid heating and short exposure time led to the aligning the body formed β grains having no time to occur, and heterogeneous composition of the material subjected to cooling. For example, in the alloy containing 12. 5 at. % aluminum, the difference in the concentrations of aluminum reached 2 at. % in different regions, formed during α plates cooling. However, a lamellar structure was formed in all cases.

During the TEM observation, heterogeneous lamellar($\alpha+\beta$)-structure was also observed.

The α and β phases plates with a low density of defects and with perfect interfaces within them are visible in separate microvolumes (Figure 1, a, b), while in

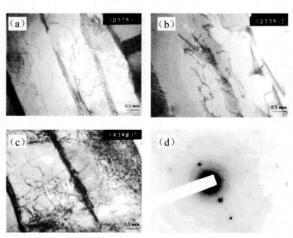


Figure 1. The microstructures of quenched alloys

others, there is an increased density of imperfections in the crystal structure (Figure 1, c). α_2 phase reflections are visible on the electron images, however, individual particles were not detected (Figure 1, d). These reflections can be seen more brightly for alloys having a higher aluminum equivalent.

In the alloy having a low content of aluminum (aluminum equivalent of 8), the reflections from α_2 phase is not observed.

A lamellar($\alpha+\beta$)-structure is also observed in the aged state in all alloys. However, in the case of aging at 600-700°C, the globularization processes of (phase are observed (Figure 2), as for example, after aging at t 600°C for 100h. The β phase is a separate globular particles, the formation of which, in our opinion, occurs in accordance with the scheme proposed by Margolin for α plates globularization⁵⁾.

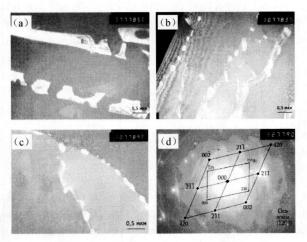


Figure 2. The morphologies of β phase after aging at 600°C

In addition, during aging there is a precipitation of silicide particles, which are mainly located at the interphase α/β boundaries on the part of β phase and have the structural formula of $(Ti, Zr)_5 Si_3$ with hexagonal lattice (Figure 3).

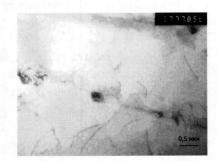


Figure 3. The precipitation of S_1 silicide on the interphase α/β boundary

In the literature, these silicides are denoted as S_1 . Sometimes silicide particles are located inside the separate α phase globules. Then their structural formula correspond to $(Ti,Zr)_6Si_3$, due to the increased zirconium content in these particles. These silicides are deno-

ted S_2 . Allocation of silicides is observed in the alloys aged at 600-700°C. The amount of silicide particles in the alloys aged for 100 hours both at 600, and 700°C is approximately the same. As it was shown in^{2,6)}, the selection of silicide particles on the interphase boundaries (S_1) causes a decrease in the work required for the crack extension.

In alloys, where the aluminum equivalent is more than 9, reflections from the α_2 phase are always clearly observed. Their intensity increases with the aging time but poorly depends on aging temperature (500-700°C). In the structure of alloys with aluminum equivalent of 8. 0-9. 2, aged at t 500°C, there are crystal structure defects, similar to the dislocation (Figure 4, a), and in an alloy with aluminum equivalent of 10.5, antiphase boundaries are visible in separate micro-volumes(Figure 4, b) which clearly indicate the processes of ordering. In our opinion, the defects observed in Figure 4, are also fragments of antiphase boundaries. Microstructures data are indicate of improvements of ordered structure, the formation of which is the result of a homogeneous transformation without the particles being formed by the nucleation and growth mechanism.

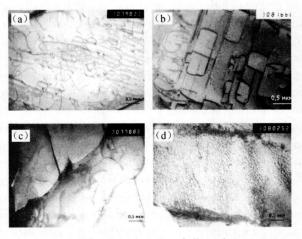


Figure 4. The microstructures of alloys after aging at $500^{\circ}\text{C}(a,b)$, $600^{\circ}\text{C}(c)$ and $700^{\circ}\text{C}(d)$

The formation of such structures does not cause any significant reduction of alloys viscosity.

In the case of aging at 600° C we failed to observe the antiphase boundaries, but in separate micro-volumes pair the dislocation is observed (Figure 4,c). Reflections from the α_2 phase are also observed for all investigated alloys, but their intensity is lower than that after aging at 500° C. Separate dispersed particles were not found. In our opinion, the observed decrease in viscosity is due to the silicides segregation at the interphase interface.

For the alloys aged at 700° C, the precipitation of separate α_2 phase particles was clearly observed. These particles were evenly distributed throughout the body of the grain (Figure 4, d). They were formed by the

mechanism of nucleation and growth, i. e. in a heterogeneous way. As a result of the particles formation there is a disastrous decrease in the toughness characteristics. KCT reduced almost to zero.

Thus, electron-microscopic study of the alloys structure has shown that the results of microdiffraction analysis after all the treatments studied in the alloys a phase is present. However, the relative intensity of the reflections from the α_2 phase increases in the alloys in the aged condition compared with alloys, air cooling from the β -region, while the intensity of the reflections in the alloys with the aluminum equivalent of more than 9. 0 after aging, both after 500°C and after the 700℃, is practically identical. At the same time, isolated α2 phase particles observed in the alloys only after aging at 700°C, and after aging at 500°C there are areas where antiphase boundaries are visible. The number of these areas increases with the aging time and a higher aluminum equivalent, i. e. ordering proceeds by the mechanism of homogeneous transformations.

The results are consistent with the results of 7 , where the mechanism of ordering in binary titaniumaluminum alloys is theoretically considered. Based on the analysis of the change in free energy, depending on the degree of long-range order and temperature, the authors showed that there were two temperatures To and T_s, which characterized the possible mechanisms of transformation. The temperature then, as usual, corresponds to the critical temperature and characterizes the equality of free energies of phases α and α_2 . But unlike the usual representations of T₀, when the curves of the free energies intersect, in this case there is a "detachment" from the curve of the free energy of a disordered alloy, characterizing the ordered phase. Temperature T_s corresponds to the point of inflection on dependence of the free energy on the degree of order and, therefore, defines the region of instability of the ordered phase. The calculations showed that the temperature $T_s < T_o$. For example, Figure 5 shows the effect of

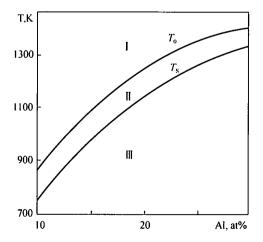


Figure 5. The influence of aluminum concentration on T_0 and T_S temperatures"

concentration on the position of the temperatures T_s and T_o^{7} .

Figure 5 shows that when the alloy is in region 1, then for the nucleation of α_2 phase, the composition fluctuations are required, since $T > T_0$ and the ordered phase has a higher energy. At $T < T_S$ (Region []]), any order degree fluctuation is energetically favorable and the formation of α_2 phase should occur by homogeneous mechanism, which is representative for II kind phase transitions. In the $T_0 - T_S$ interval (Region []) two mechanisms of transformation can occur.

Using the results of? temperatures T_0 and T_S were estimated for the experimental alloys. Alloys were considered as binary titanium-aluminum systems. The temperature T_0 is in the 550... 620°C range depending on the composition, and the temperature T_S -620... 680°C, which agrees with the experimental data presented above. Consequently, the results of? confirm that at 500°C the formation of α_2 structure is the result of ordering by the homogeneous transformation, and for 700°C-the process occurs by the mechanism of nucleation and growth with the formation of dispersed particles of α_2 phase. When the aging temperature is 600°C, both mechanisms of transformation are possible.

4. Conclusions

- (1) It was found that at low aging temperatures (500°C) the formation of α_2 phase occur due to the separate micro volumes ordering by the homogeneous mechanism due to the inhomogeneity of the initial state with the characteristic formation of antiphase boundaries. The increase of the aging temperature to 700°C helps to activate the diffusion processes and contributes to the formation of α_2 phase by the mechanism of nucleation and growth.
- (2) It was confirmed that the structural type S_1 silicides were formed at the interphase α/β boundaries on the part of β phase, and silicides such as S_2 and S_3 were formed in the α phase.
- (3) The influence of the aluminides and silicides allocation on alloys mechanical properties during aging were investigated. It was shown that their formation leads to a slight hardening and a significant decrease in viscosity properties. Plastic characteristics are only moderately changed.
- (4) It was found that in the process of aging within a definite temperature range, the globularization of β phase thin layers was possible.

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