Effect of Thermomechanical Treatment Parameters on Structure, Phase Composition and Mechanical Properties of Ti-3Al-5Mo-4.5V Titanium Alloy

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Abstract. The structure, phase composition and mechanical properties of (α+β) – titanium alloy solution treated at 850 °C, cold-rolled at the reduction ratio in the range of 0…45%, followed by ageing at 450, 500, 550 °C for 0.5, 1.5, 3 hours was studied using XRD, microindentation and tensile testing. The influence of strain level at cold rolling and time-temperature parameters of ageing on the formation of structure and phase composition of solution treated and water quenched Ti-3Al-5Mo-4.5V alloy was investigated and discussed in terms of tensile properties and microhardness. The parameters of low temperature thermomechanical treatment (LTMT) of the (α+β) – alloy were proposed to obtain a high-strength state.

1. Introduction

The (α+β) – titanium alloy Ti-3Al-5Mo-4.5V (Russian Grade –VT16) was developed to manufacture fasteners in aircraft industry employing cold deformation [1–4] as well as the other high strength product, including medical implants [5]. Strain-induced martensitic transformations and twinning can occur during cold deformation of the solution treated and water quenched alloy depending on the stability of the metastable phases fixed in Ti-3Al-5Mo-4.5V, [1, 6–10]. This is reflected in a staged nature of the decay of metastable phases, which were previously studied during continuous heating in [9, 11–12]. At the same time, strengthening treatment in industrial conditions of include solution treatment and cold deformation with the isothermal ageing as a final stage of the processing [13]. However, data on the development of structure and mechanical properties of the cold-rolled VT16 alloy during isothermal ageing are limited [14–15]. The purpose of the study was to investigate the effect of reduction ratio at cold rolling of the Ti-3Al-5Mo-4.5V on structure and mechanical properties, which are formed during ageing with different time-temperature parameters.

2. Material and Methods

The commercial hot-rolled 12 mm rod of VT16 alloy of the following composition – Ti-2.9Al-4.8Mo-5.1V-0.12O (wt.%) with β-transus of 860 °C was manufactured by VSMPO-AVISMA Corporation. The processing of the rods included solution treatment (ST) at 850 °C followed by water quenching (WQ). The next step was cold rolling (CR) with reduction ratios of 9, 16, 31, 45%. In accordance with [9] a two-phase structure consisting of a small fraction of the primary α-phase in a matrix of α”-orthorhombic martensite obtained as a result of β → α” – transformation is fixed in the alloy during water quenching from 850 °C. An increase in the reduction ratio at CR promotes the development of a strain-induced
α''→ α' + βₐ transformation in the ST alloy. βₐ is a BCC phase with an anomalously high lattice spacing which is detected starting from the reduction ratio of 16%. The ST and CR rods were aged at temperatures of 450, 500, 550 °C for 0.5, 1.5 and 3 hours. The phase composition of the alloy was studied using XRD on a Bruker D8 Advance in Cu Kα-radiation. Vickers microhardness values were determined using an attachment to Neophot-21 microscope at a load of 1 N. Tensile tests were carried out on an Instron 3382 in accordance with ISO 6892.

3. Results and discussion
A comparative analysis of the diffraction patterns of the Ti-3Al-5Mo-4.5V alloy in the ST and aged state revealed that α'' – martensite decomposed according to the α'' → α + β scheme, which was accompanied by the appearance of diffraction peaks of the α and β phases (figure 1a). A similar character of transformations upon heating is characteristic of the alloy, which was ST and CR at reduction ratio of 9%. An increase in the ageing temperature from 450 °C to 550 °C contributes to a deeper development of decomposition in the alloy after ST and CR at 9%. This is evidenced by a decrease in the intensity of martensite peaks and by a corresponding increase in the intensities of the α and β-peaks (figure 1a) with increasing the ageing temperature at the same dwell times. However, the VT16 alloy did not reach a completely equilibrium state. The peaks of the α'' – martensite (figure 1a) preserved in the diffraction patterns at a maximum ageing temperature of 550 °C and dwell time of 3 hours.

![Diffraction patterns of ST alloy](image)

**Figure 1.** Diffraction patterns of ST alloy (a) and after CR at reduction ratio of 45% (b) before and after ageing at 450, 500, 550 °C for 0.5 and 3 hours.

The decay scheme during ageing changes for the alloy with the reduction ratios of 16, 31, 45%, in which as a result of strain-induced transformation α'' – martensite transforms into α' – martensite and βₐ-phase peaks appear in the diffraction pattern (figure 1b) and becomes the following:

1. \( \alpha' (\alpha'') \rightarrow \alpha' (\alpha'')_{\text{rich}} + \alpha' (\alpha'')_{\text{lean}} \rightarrow \beta + \alpha \)
2. \( \beta_{n} \rightarrow \alpha + \beta \)

where \( \alpha' (\alpha'')_{\text{rich}} \) and \( \alpha' (\alpha'')_{\text{lean}} - \alpha' (\alpha'') \) are phases respectively enriched and depleted with β-stabilizers as a result of diffusion, which takes place during ageing of \( \alpha' (\alpha'') \) – martensite obtained by WQ and
subsequent CR, β – BCC-phase formed of α′(α’’)rich and βa- phases due to their diffusion enrichment with β-stabilizers (Mo, V) as a result of α′(α’’)rich → β and βa→β – transitions.

Analysis of the diffraction patterns of the aged alloy after CR at the reduction ratios in the range of 16–45% demonstrated that the decay of α′(α’’) – martensite starts earlier than of the βa-phase. The decay accelerates with increasing ageing temperature from 450 to 550 °C (figure 1b) similar to ST samples.

Based on XRD data the lattice parameters of α and β-phases (c/a, FWHMα and aβ) formed during ageing (figure 2) were calculated. The influence of the reduction ratio on the intensity of the decay process during ageing was analyzed.

![Graphs](image.png)

**Figure 2.** Lattice parameters c/a (a), FWHM of (100)α diffraction peak (b) and aβ (c) depending on the CR reduction ratio after ageing at 500 (b) and 550 °C (a, c). Microhardness depending on the ageing time of solution treated Ti-3Al-5Mo-4.5V (d) and on the reduction ratio of the cold-rolled alloy aged at 450 °C (e) and 550 °C (f).

It was demonstrated that an increase in the reduction ratio results is the acceleration of decay. This was confirmed by a regular growth c/a of the α-phase with an increase in the reduction ratio upon CR as was shown for ageing at 550 °C (figure 2a). According to [16], the parameter c/a of the α phase
increases with increase in the content of α-stabilizing elements such as Al in VT16 and with a decrease in the concentration of β-stabilizers such as V and Mo.

The development of a more equilibrium state also leads to a regular decrease in the full width at half maximum (FWHM) of the diffraction peaks of α-phase formed during the ageing of the rods with higher reduction ratio (figure 2b). In addition, an increase in the reduction ratio at the same time-temperature parameters of ageing is accompanied by a decrease in the lattice spacing of the β-solid solution (figure 2c) as the result of enrichment with β-stabilizers (Mo, V) which are characterized by a smaller atomic radius than titanium according to [17].

The acceleration of decay processes during ageing with a higher reduction ratio is due to the characteristic increase in a number of crystal structure defects such as dislocations, nonequilibrium point defects such as vacancies and interphase boundaries [9], which serve as the sites for heterogeneous nucleation during decay and accelerate the diffusion [18]. The observed acceleration of decay processes in the ST alloy with an increase in the reduction ratio affects its microhardness in the aged state (figure 2 d, e, f).

Ageing in the temperature range of 450 ... 550 °C for 0.5, 1.5 and 3 hours contributes to an increase in microhardness values both after ST (3100 MPa) and after CR with the average microhardness values: 3300 MPa (ε=9%) 3600-3700 MPa (ε=16, 31%) and 4200 MPa (ε=45%). The influence of the temperature-time parameters of ageing is fundamentally close for ST and CR states, only the level of the values changes. Therefore, the further analysis was based on the ST sample.

The decay in the ST alloy after ageing even for 30 minutes provided an increase in hardness by 1.5...1.6 times up to 4500 MPa (Tα = 450 °C), 4900 MPa (Tα = 500 °C), 4950 MPa (Tα = 550 °C). (figure 2d). The increase in microhardness with an increase in the ageing temperature is attributed to the acceleration of α’-martensite decomposition in accordance with the above-mentioned XRD data (figure 1, 2). An increase in ageing time from 0.5 to 3 hours had a different effect on microhardness for ageing temperatures (figure 2d). A permanent growth of microhardness is characteristic of ageing at 450 °C. Constant values starting from dwell time of 1.5 hours were observed for 500 °C. A decrease in microhardness starting from dwell time of 1.5 hours took place at 550 °C. This is in a good agreement with the XRD data (figure 1), as well as with the general laws of decay of metastable phases during ageing of light alloys [19].

![Figure 3. Mechanical properties vs. reduction ratio at cold rolling for rods of Ti-3Al-5Mo-4.5V aged at 500 °C for 3 hours.](image)

According to the XRD data (figure 1), the decay of α’’ – martensite during ageing at 450 °C up to a 3 hour dwell did not provide the equilibrium (α + β)-structure. Therefore, hardness increased with increasing exposure time (figure 3d) in accordance with [20]. Structure close to equilibrium was formed at holding for 1.5...3 hours at an ageing temperature of 500 °C and consisted mainly of α and β phases. Therefore, the growth of microhardness stopped and reached a constant level at these dwells. An increase in the ageing temperature up to 550 °C resulted in (α+β) – structure that was close to equilibrium.
starting from 0.5 hour exposure (figure 1). The coagulation of decomposition products and stress relaxation processes took place at exposures for 1.5 and 3 hours, which led to a decrease in microhardness.

An increase in the reduction ratio during CR, accelerated the decomposition of metastable phases and led to higher microhardness values after ageing at 450 °C compared to the ST sample (figure 2e). Active development of softening processes occurred with increasing dwell time during ageing at 550 °C (figure 2f). Therefore, ageing of ST and CR samples at 500 °C for 3 hours was chosen for mechanical testing (figure 3), which provided high strength state along with the equilibrium structure according to the microhardness values (figure 2d) and XRD data (figure 1).

Thus, the LTMT including ST at 850 °C with WQ followed by CR at reduction ratio of 16% and ageing at 500 °C for 3 hours provided the most significant combination of mechanical properties of Ti-3Al-5Mo-4.5V (VT16) titanium alloy (YS > 1550 MPa, UTS > 1560 MPa, EL > 4 %).

References
[1] Moiseev V N 2001 Metal Science and Heat Treatment 43 73–77
[2] Dunaev V V and Shirshov A A 2009 Russian Eng. Research 29 864–870
[3] Xingwu L, Masyue S and Junpeng C 2009 Met. Science and Heat Treatment 51 594–598
[4] Ivanov A, Orlov A and Golubovskii E 2019 Mat. Today: Proc. 19 2163–2166
[5] Illarionov A G, Grib S V and Huppee A V 2019 Materials Science Forum 946 309–314
[6] Moiseev V N 1972 Metal Science and Heat Treatment 14 391–395
[7] Maltsev M 1990 Fiz. Met. Metalloved 12 97–103
[8] Popov A A, Illarionov A G, Stepanov S I and Ivasishin O M 2014 Phys. of Metals and Metallography 115 517–522
[9] Illarionov A G, Demakov S L, Stepanov S I and Illarionova S M 2015 Phys. of Metals and Metallography 116 267–273
[10] Xue Q, Ma Y J, Lei J F, Yang R and Wang C 2018 J. of Mater. Science & Tech 34 2507–2514
[11] Maltsev M V 1976 Fiz. Met. Metalloved 41 1225–31
[12] Maltsev M V 1977 Tsvetn. Met. 68–70
[13] Khorev A I 2011 Russian Eng. Research 31 1227–32
[14] Stepanov S I, Illarionov A G, Demakov S L and Stepanova E D 2016 AIP Conf. Proc. 1785 040084
[15] Grabovetskaya G P, Ratochka I V, Mishin I P, Lykova O N and Zabudchenko O V 2019 Met. Science and Heat Treatment 60 580–588
[16] Zwicker U, 1974 Titan und Titanlegierungen (Berlin: Springer)
[17] Miracle D B and Senkov O N 2017 Acta Mater. 122 448–511
[18] Collings E W 1984 The Physical Metallurgy of Titanium Alloys (Ohio, Metals Park: Am. Soc. for Metals)
[19] Polmear I 2006 Light Alloys: From Traditional Alloys to Nanocrystals (Butterworth-Heinemann: Elsevier)
[20] Kolachev B A, Elagin V I and Livanov V A 2005 Metal Science and Heat Treatment of Non-Ferrous Metals and Alloys (Moscow: MISIS)

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