

Parameters Evolution of Orthorhombic Martensite Lattice Ti-Mo-Al Alloys Studied by in Situ T-XRD

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Abstract. *In-situ* study of heating in an X-ray diffractometer of Ti-8Mo and Ti-3Al-7Mo alloys samples quenched from different temperatures of $\alpha+\beta$ and β -fields was carried out. The characteristics of anisotropy thermal expansion of orthorhombic martensite lattice were investigated. It is shown that there is a critical ratio of lattice parameters $R = \sqrt{3}a/b$ close to 1.11, after which either a reverse martensitic transformation occurs, or R of the martensite lattice decreases upon further heating.

1. Introduction

The Ti-Mo system is the basis for many industrial titanium alloys. The presence of Mo in the composition can significantly increase the mechanical properties of the material. In this case, Mo is an isomorphous β -stabilizer and, according to the data [1], it is possible to produce martensite during the quenching from the β -region in the Mo concentration range up to 11.3% (wt.). According to other data [2], the critical Mo content does not exceed 10% (wt.).

Recently, titanium alloys having a microstructure of orthorhombic martensite obtained either as by direct quenching from high temperatures and as a result of the strain-induced martensitic transformation, became the object of serious study [3]. This is associated with a number of features of the direct and the reverse martensitic transformation, such as the shape memory effect and the low modulus. Previously, a large amount of data on the orthorhombic martensite lattice spacing was accumulated. Its behaviour depends on a chemical composition and parameters of an external impact, such as temperature and deformation. The obtained data was partly structured and classified in works by Moffat and Dobromyslov [2-6]. This study expands the conception of A20 lattice on the example of Ti-Mo and Ti-Al-Mo alloy systems.

2. Research Methodology

The materials of the study were bars of binary and ternary titanium alloys 12 mm in diameter, the chemical compositions of the studied alloys are shown in table 1.

Ingots weighing 30 kg were obtained by the triple remelting. The ingots were homogenized at a temperature of 1200 °C for 6 hours in a vacuum. Ingots were worked to the rods using the hot deformation. The last steps were carried out at heating temperatures below β -transus temperature. The rods were cut and water quenched from both the β - and $(\alpha+\beta)$ - regions.



Table 1. Chemical compositions of the studied alloys.

Name of the alloy	Elements, % wt.									$T_{\beta}, ^\circ\text{C}$
	Ti	Mo	Al	Cr	Ni	Fe	O	N	C	
Ti-8Mo	base	7.6	0.1	0.004	0.03	0.01	0.07	0.003	0.01	790
Ti-3Al-8Mo	base	6.9	2.9	0.005	0.03	0.03	0.05	0.002	0.01	850

The samples quenched from various temperatures were placed in a thermal chamber of an X-ray unit and subjected to heating (T-XRD method, Bruker D8 advance diffractometer with a Cu-K α radiation ($\lambda=0.154178$ nm)) to evaluate the temperature dependence of the change in the periods of the martensite lattice. The phase composition after quenching of the alloys is given in table 2. the T-XRD samples were prepared extremely carefully; grinding was performed without heating in cold fills. The finishing operation was electropolishing in the cooled electrolyte based on mix of acetic and perchloric acids.

Table 2. The phase composition and martensite lattice parameters of quenched samples.

	$T_q, ^\circ\text{C}$	Phase composition	Lattice parameters of α'' -martensite, nm			R ($\sqrt{3}a/b$)
			a	b	c	
Ti-8Mo	750	$\beta+\alpha+\omega$	-	-	-	-
	900	$\alpha''+\beta_{\text{trace}}$	0.3022	0.4987	0.4663	1.049
Ti-3Al-7Mo	800	$\beta+\alpha''$	0.3069	0.4861	0.4616	1.093
	810	$\alpha''+\beta+\alpha$	0.3051	0.4908	0.4632	1.077
	900	α''	0.2978	0.4979	0.4648	1.036

3. Results and Discussion

The microstructure of the alloys quenched from the β -region was typical for the same cases (Fig. 1). The sizes of the equiaxed β -grains were in ranges from 100 to 250 μm . Martensite plates of different orders were present in the structure, the first of which intersected all the grains. A residual β phase was observed in the interplastic layers. Alloys hardened from the ($\alpha+\beta$) - region contained α -phase globules. The matrix was a β -phase with rare martensitic plates difficult to detect by optical metallography.

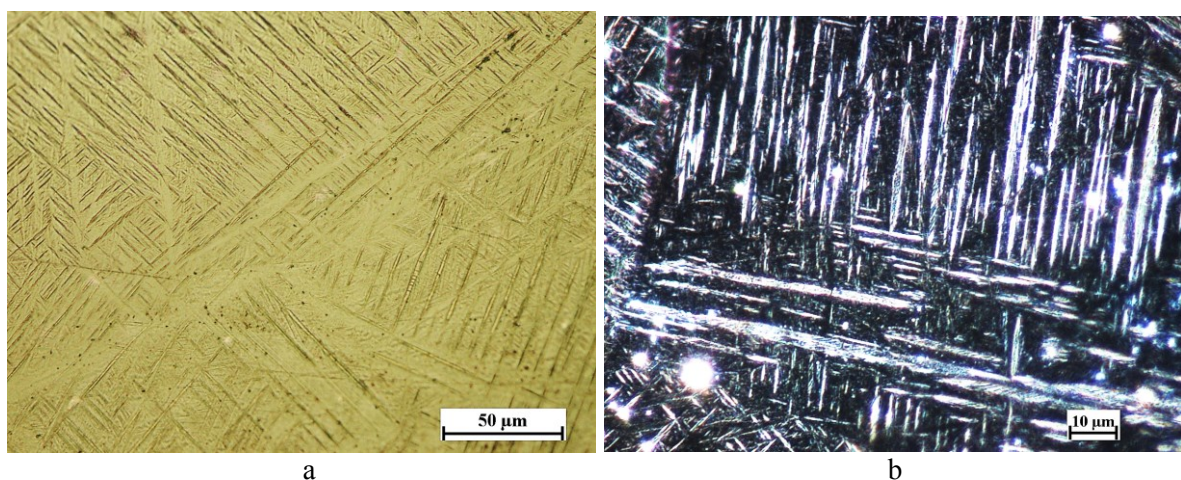


Figure 1. The microstructure of Ti-3Al-7Mo sample $T_q=900$ $^\circ\text{C}$: a – bright field image; b – bright field image

The phase composition of the quenched samples was determined using the X-ray analysis. The parts of the diffraction pattern are shown in Figures (2 a, b). The martensite is present in Ti-8Mo alloy, its lattice parameters are given in table 2. In case of the quenching from a two-phase region, the presence of an α -phase leads to a change in the chemical composition of the β solid solution, accompanied by an increase of its molybdenum concentration. According to the measured data at $T_q = 760^\circ\text{C}$, the Mo content should be about 10 wt. %. Such decrease of the heating temperature led to the suppression of the β - α'' martensitic transformation. Instead, a β - ω transformation is observed, which is confirmed by the diffractogram shown in the figure 2 a. The diffractogram shows the presence of the ω -phase lines. These, containing the ω -phase, samples were not subjected to further in situ heating. It is known [8] that the aluminum alloying leads to suppression of the formation of the ω -phase. Therefore, along with the binary Ti-Mo system, the ternary Ti-Mo-Al system was investigated. The diffractograms of quenched samples of a triple system for different quenching temperatures are demonstrated in the Figure 2 (b). They show the absence of an ω phase in the entire range of quenching temperatures considered. In the samples quenched from the β region, the β -phase lines are absent, almost complete martensitic transformation is observed. According to the diffractogram there is an untransformed β phase in the sample $T_q = 810^\circ\text{C}$. For different quenching temperatures, the martensite lines having identical indices are located at different angles 2θ , that is explained by the change in the alloying of the high-temperature β -solid solution primarily with molybdenum. The changing quenching temperature in 10 degrees led to a significant decrease in the amount of martensite. The intensity of its lines becomes very small when the β -phase lines had high intensity. The quenching temperature of 800°C is close to the critical temperature (T_{cr} is the maximum T_q where only the untransformed solid β solution is fixed).

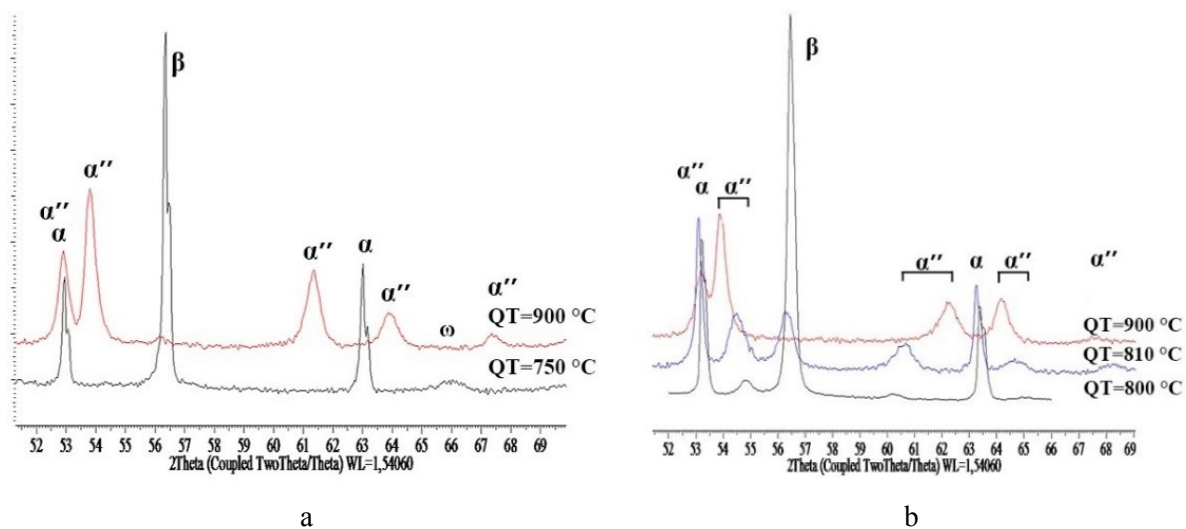


Figure 2. Diffraction patterns of titanium alloys samples quenched from different temperatures Ti-8Mo (a) and Ti-3Al-7Mo (b)

Samples quenched from different temperatures were studied *in situ* during heating on X-ray diffractometer. Diffraction patterns are shown in Figure 2. In all cases at the beginning of heating the line (200) (attributed to a period $\langle a \rangle$) is rapidly was emphasized to shifted toward large angles while the quenching temperature increases. The line (130) remains practically in place and the line (022) shifts towards smaller angles.

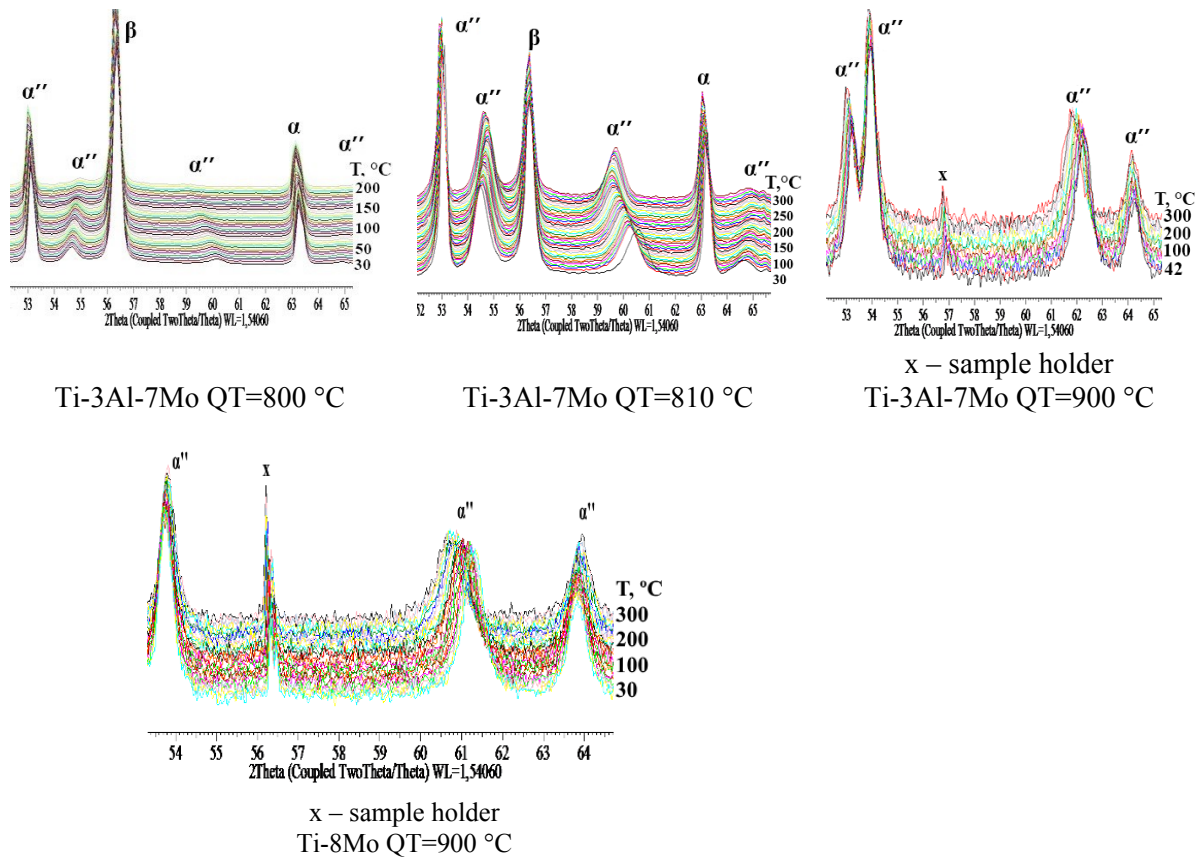


Figure 3. Diffraction patterns of titanium alloys samples quenched from different temperatures during heating

The position of α'' - martensite peaks vary for different crystallographic directions. It proves that the lattice of α'' - martensite behaves anisotropically.

The lattice spacing of orthorhombic martensite were calculated with (200), (130), (112), (022)-peaks. Figure 4 shows the relative change in the period's dependence on the temperature.

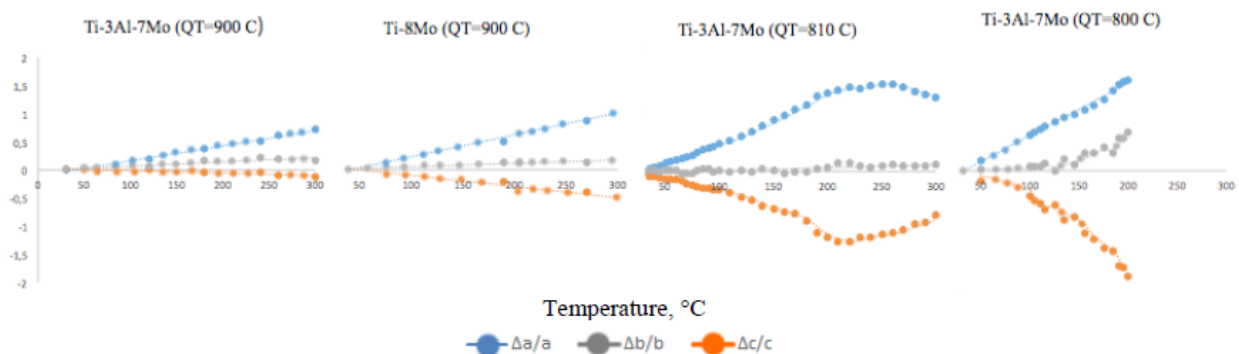


Figure 4. Dependence of QT on martensite lattice spacing during heating

The behaviour of lattice orthorhombic martensite spacing is presented as a combined plots of relative spacing changing for a specific quenching temperature (Fig. 4). Values of spacing at room temperature were taken as the initial values.

Analysis of the above relationships showed that spacing «a» and «c» increase naturally but spacing «b» decreases during heating. In the first approximation, curves can be considered as straight lines; furthermore, a slope angle for different quenching temperature is different. The increase in spacing «a» with an appropriate decrease in spacing «b» transforms orthorhombic lattice towards the bcc lattice. For the quenching temperature of 800 °C, the reverse transformation took place accompanied by increased β -phase lines intensity [6, 7]. For other temperatures of quenching above 800 °C the reverse transformation did not occur.

For the generalization of the data a dependence of the change in the rhombic degree R as a function of temperature was constructed. The rhombic degree is the ratio of $\sqrt{3}a/b$. In the indexes of orthorhombic lattice R of hcp is 1, for a bcc lattice $\sqrt{3}/\sqrt{2}$. In the case of an orthorhombic lattice the R has an intermediate value with respect to the above. The change of the angle of inclination depending on the initial rhombic degree is well traced. The sample of the alloy Ti-3Al-7Mo quenched from the β -region having the lowest initial rhombic degree showed the greatest inclination angle. Conversely the smallest inclination angle showed a sample of the same alloy quenched from 800°C having the largest initial rhombic degree. In the sample Ti-3Al-7Mo $T_i = 810$ °C, the slope angle gradually decreases as the heating proceeds and becomes approximately equal to the inclination of the sample curve with $T_q = 800$ °C. When the rhombic degree of 1.11 is reached a bend is observed in the course of the curves but their behaviour is significantly different. If the curve (Ti-3Al-7Mo $T_q = 810$ °C) goes in the opposite direction, the rhombic degree in a certain temperature range remains constant, and then goes back. In the case where the reverse martensitic transformation takes place (Ti-3Al-7Mo $T_q = 800$ °C), the direction of the curve does not change and the angle of inclination continues to increase until the phase disappears completely.

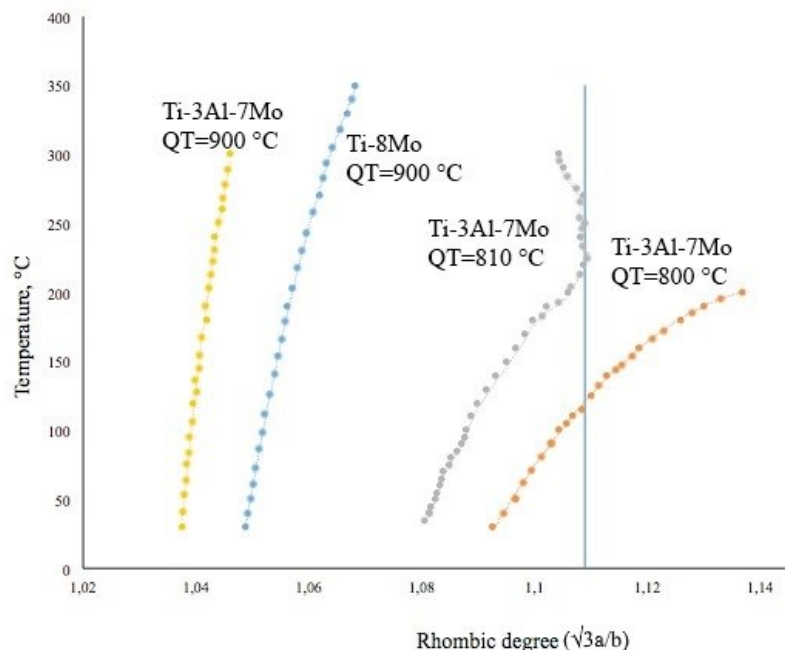


Figure 5. Dependence of the rhombic degree on martensite lattice spacing during heating of Ti-8Mo and Ti-3Al-7Mo alloy samples

4. Conclusion

As a result of the study, the anisotropy characteristics of the thermal expansion of the titanium orthorhombic martensite lattice are determined. It is established that either in the Ti-Al-Mo system there is a critical value of the relative parameter R ($\sqrt{3}a/b$) close to 1.11, after which a reverse martensitic transformation occurs or a decrease of the rhombic degree of the martensite lattice upon further heating. It is possible that the difference is due to the temperature at which the critical value R is reached.

5. Acknowledgements

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6. References

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