

PAPER • OPEN ACCESS

## Study of lamination in beta-phase of Ti-5553 titanium alloy

To cite this article: M Kalienko *et al* 2018 *IOP Conf. Ser.: Mater. Sci. Eng.* **461** 012033

View the [article online](#) for updates and enhancements.

You may also like

- [Application of the SPOTS technique to the measurement of proton tunnelling splittings in beta -phase palladium hydride](#)  
H Avram and R L Armstrong
- [Variational resolution of outflow boundary conditions for incompressible Navier–Stokes](#)  
Michal Bathory and Ulisse Stefanelli
- [ON SOME RESULTS AND PROBLEMS OF THE STABILITY THEORY OF DIFFERENCE SCHEMES](#)  
A V Gulin and A A Samarski



**ECS**  
The  
Electrochemical  
Society  
Advancing solid state &  
electrochemical science & technology

**DISCOVER**  
how sustainability  
intersects with  
electrochemistry & solid  
state science research

## Study of lamination in beta-phase of Ti-5553 titanium alloy

M Kalienko<sup>1,2</sup>, A Volkov<sup>1</sup>, M Leder<sup>1</sup>, A Zhelnina<sup>1,2</sup>, P Panfilov<sup>2</sup>

<sup>1</sup> PSC VSMPO-AVISMA Corporation, Parkovaya st. 1, Verkhnyaya Salda 624760, Russia

<sup>2</sup> Ural Federal University, 19 Mira street, Ekaterinburg 620002, Russia

E-mail: kamak@yandex.ru

**Abstract.** The present study primarily focuses on lamination in beta-phase of Ti-5553 titanium alloy. Microstructure and chemical composition of Ti-5553 alloy after heat-treatment was investigated by electron microscopy (SEM, TEM), x-ray diffraction (XRD) and energy dispersive x-ray analysis (EDX). It was revealed that after annealing and slow cooling from  $\beta$  phase field the  $\beta$  phase separated into  $\beta$ -enriched and  $\beta$ -lean regions. This effect does not effect on precipitation of secondary  $\alpha$  phase.

### 1 Introduction

High-strength titanium alloy Ti-5553 is widely used in the aircraft industry for parts such as the chassis and structural elements. This alloy belongs to the group of metastable  $\beta$  alloys [1]. The microstructure of this type of alloys is very sensitive to thermal treatment. Ti-5553 alloy is used after deformation above beta transus temperature and in  $\alpha+\beta$  fields and the mechanical properties resulting from such treatments are strongly depend of the morphology, volume fraction, size and distribution of the  $\alpha$  phase in the  $\beta$  matrix [2-5].

Decomposition and lamination of the  $\beta$  phase solid solution to  $\beta$  lean and  $\beta$  rich phases was observed by transmission electron microscope in form lamellae with light and dark contrast [6-8]. There has been only a limited number of previous investigations addressing the decomposition and lamination of  $\beta$  phase in titanium alloys. To identify the effect of lamination requires a precision approach, methods such as electron transmission microscopy (HR-TEM, HAADF-STEM), atomic tomography and microanalysis (3DAP Tomography, APT), x-ray analysis. Illarionov A. G. et al. [6] studied structural and phase transformations during cold plastic deformation of VT22 titanium alloy, which was taken as the basis for development of the alloy Ti-5553 [1]. They found the formation of fine lamellae within the layers of the  $\beta$  matrix by TEM. The observed structure in the VT22 alloy according to the electron diffraction and XRD was ascribed to the precipitation of a body-centered-tetragonal  $\tau$  phase via a shear transformation. Zherebtsov S. V. et al. [7] reported that a TEM diffraction pattern from the  $\beta$  phase did not show any other phases, so the presence of “bands” between reflections of the  $\beta$  phase may imply the presence of areas of short-range order with a structure varying somewhat from the matrix. In the work of P. Barriobero-Vila et al. [8], the presence of “bands” in the structure of  $\beta$  phase of the investigated Ti-5553 alloy is associated with the fluctuation of the chemical composition, but does not provide experimental evidence.

The phenomena of solid solution lamination, spinodal decomposition observed in the form of separation in  $\beta$  phase, were found in titanium alloys with high concentration of  $\beta$  stabilizers [9-14]. The theoretical description of the observed phenomena is faced with great difficulties, so new experimental facts describing the variety of possible thermodynamically unstable states are needed to



clarify the theory guidelines. In our work [15] we observed lamination in Ti-5553 alloy with different fraction of the primary  $\alpha$  phase, so the objective of this work is to study the effect of lamination using XRD and TEM analysis

## 2 Materials and methods

The work is carried out on the forged material Ti-5553 alloy, the chemical composition of the alloy is presented in table 1. The  $\beta$  transus temperature of the alloy was confirmed as equal to 823 °C. In order to formation lamellar structure of the alloy, sample was first solution treated in the  $\beta$  field at 870 °C for 1 hour and then furnace cooled to a temperature at 700 °C, kept at this temperature and then cooled in air (the first stage of heat treatment). Then aging was carried out at a temperature of 530 °C for 8 hours (the second stage of heat treatment).

**Table 1.** Chemical composition of Ti-5553 alloy (at. %).

Ti	Al	Mo	V	Cr	Fe	Zr	O ppm	Mo eq.
Bal.	8.55	2.53	4.83	2.64	0.36	<0.0010	142	9.5

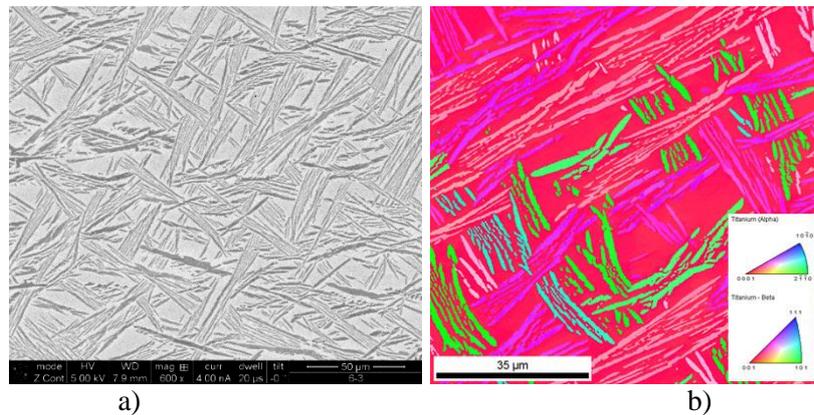
The microstructure and composition of the alloy were characterized using SEM Quanta 3D FEG with EBSD Pegasus XM4 detector and TEM Tecnai G2 20F S-TWIN. The TEM was equipped with both a high angle annular dark field HAADF-STEM detector and energy-dispersive spectroscopy (EDS) capability. EBSD map was taken at a step size of 0.1  $\mu\text{m}$ . The EBSD data was analyzed using the Orientation Imaging Microscopy (OIM 5.31) software. The boundaries with misorientations between 1.5° and 15° were defined as low-angle grain boundaries, and those of misorientations larger than 15° as high-angle grain boundaries.

Specimens for SEM and XRD analysis were prepared by applying the standard mechanical polishing techniques. Thin foils for TEM study were cut by electrical discharge machine, grinding to thickness up to 100 microns, then prepared in TenuPol-5 at a voltage of 30 V and a temperature of -40 °C.

X-ray diffraction measurements were performed on the Bruker D8 Advance diffractometer with a goniometer radius 217.5 mm in Bragg–Brentano diffraction geometry. XRD patterns were recorded at room temperature using Cu-K $\alpha$  radiation with following measurement conditions: tube voltage of 40 kV, tube current of 40 mA, stepscan mode with a step size of 0.02° 2 $\theta$  and counting time of 1 s/step by LynxEye-detector. XRD analysis was carried out using the commercial software TOPAS by “Pawley method” for whole pattern modelling.

## 3 Results and discussion

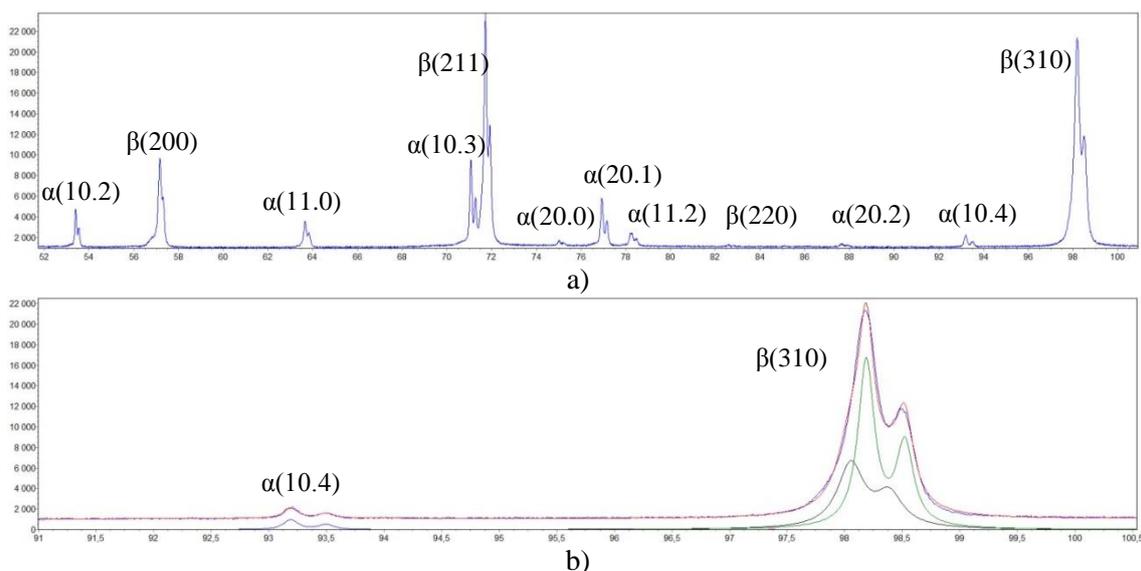
Figure 1a shows backscattered electron micrograph of the Ti-5553 alloy after slow cooling from  $\beta$  phase field. The microstructure of the alloy consisted of lamellar  $\alpha$  colonies within prior  $\beta$  grains. The  $\beta$  grains are equiaxed and the grain size in this condition has been estimated to be 260  $\mu\text{m}$ . The volume fraction of primary  $\alpha$  phase was 33 %, the average thickness of  $\alpha$  lamellae in colonies was 0.4  $\pm$  0.02  $\mu\text{m}$ . After air cooling according to the EBSD method, only the primary  $\alpha$  phase and matrix  $\beta$  phase are present in the alloy structure. Within  $\beta$  grain, the orientation of the crystallographic lattice of the  $\beta$  phase in the study by EBSD remains unchanged, the plates of the primary  $\alpha$  phase have different crystallographic orientation and are oriented in accordance with the crystallographic ratio of Burgers, figure 1b. It should be noted that the calculated disorientation of the angle for the analysis of the subgrain structure of the  $\beta$  phase was taken to be equal to 1.5 degrees. I.e., of the immutability of the orientation of the crystal lattice within a single  $\beta$  grain it is possible to speak with accuracy of 1.5 degrees.



**Figure 1.** The microstructure of Ti-5553 alloy after first stage of heat treatment: (a) SEM image, (b) EBSD orientation map.

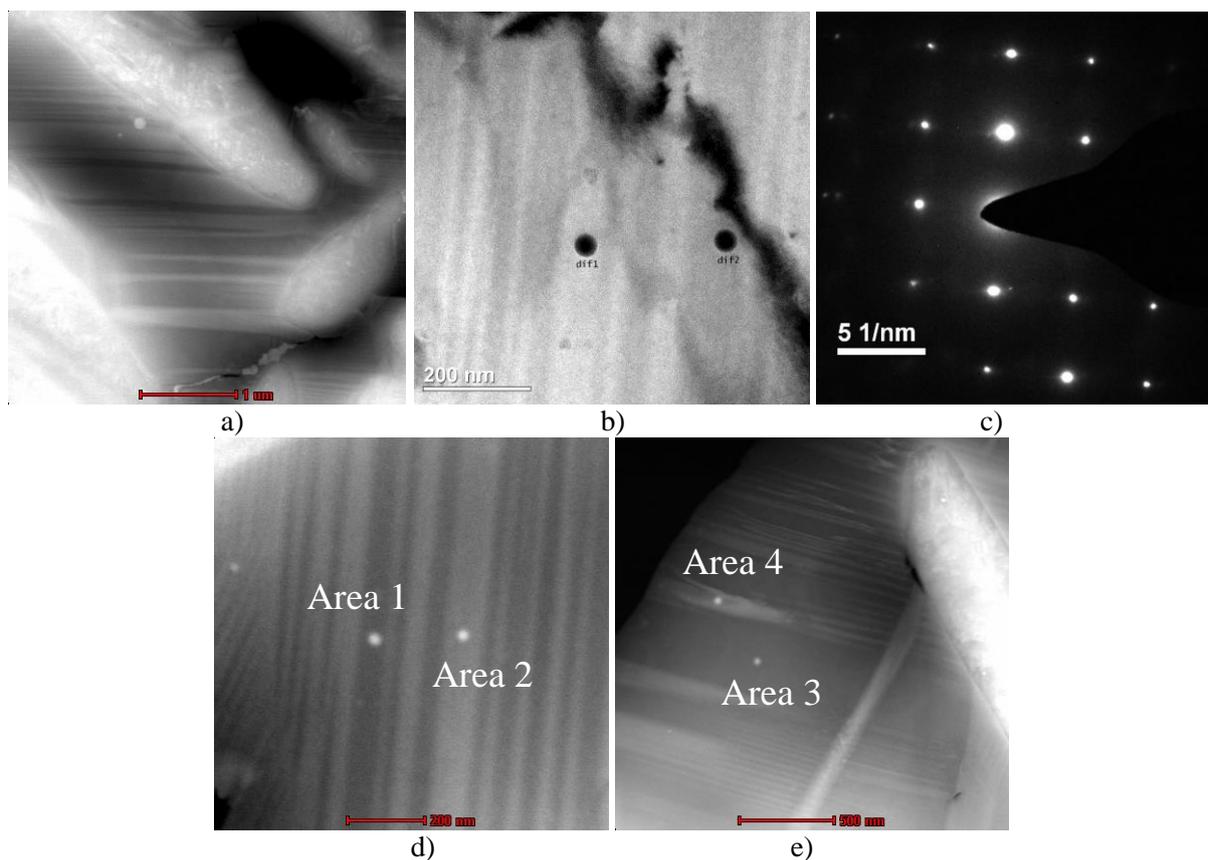
The XRD pattern of the alloy after the first stage of heat treatment is shown in figure 2. The observed asymmetry of peaks of the  $\beta$  phase on the XRD pattern is caused by two adjacent diffraction lines resulting in overlapping peaks appearing as a single asymmetric peak. The two lines caused by one type of crystallographic  $\beta$  phase but with two different lattice parameters. Using the method of full-profile analysis, the diffraction lines of the  $\beta$  phase are well described using a model that includes the presence of several phases of cubic symmetry (BCC,  $Im\bar{3}m$ ), figure 2b. Two different lattice parameters are caused by different chemical composition known as  $\beta$  phase decomposition and known to result in different lattice parameters and different angular position. When the concentration of  $\beta$  stabilizers increase in  $\beta$  phase then its unit volume decreases and parameter of the crystal lattice and the position of the diffraction lines of pattern is modified.

For the Ti-5553 alloy after air cooling from 700 °C, the best description of the profile of  $\beta$  phase diffraction lines is used two phases of cubic symmetry in the model. Thus, with a full-profile description of the XRD pattern the model well describes the line  $\beta(310)$  by two phases with parameter of lattice 3.225 and 3.228 Å, figure 2b. Probably, lamination of  $\beta$  phase occurred during cooling from  $\beta$  field and holding at  $\alpha+\beta$  field.



**Figure 2.** XRD pattern of Ti-5553 alloy after first stage of heat treatment (a) and deconvolution of the (310) diffraction peaks of the  $\beta$  phase (b).

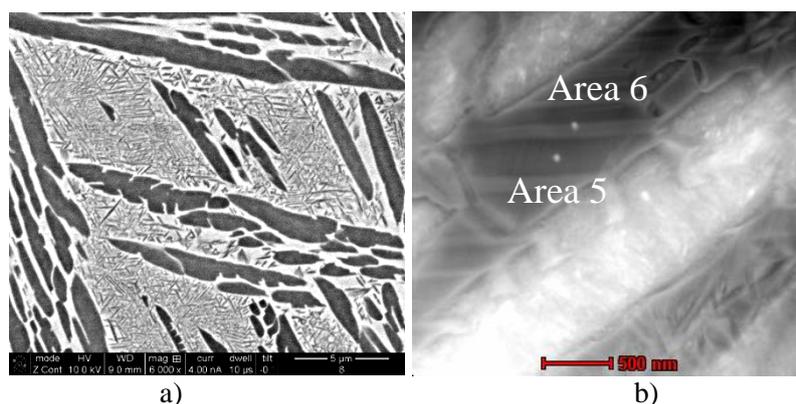
The study of microstructure by transmission electron microscope in the structure of the  $\beta$  phase observed bands that differ in contrast and thickness, figure 3a (HAADF-STEM images). By electron diffraction pattern was compared to the orientation of the crystal lattice of two nearby “bands” of different contrast, the received results is identical for the nearby “bands” and presented figure 3b, c. The obtained result confirms the data obtained in [7]. It is well known that the density of the  $\beta$  phase is greater than the density of the  $\alpha$  phase, since the  $\beta$  phase contains more  $\beta$  stabilizers and less aluminum, therefore, in the images in the reflected electrons, the  $\beta$  phase has a lighter contrast than the  $\alpha$  phase. According to these arguments, the light contrast bands in the  $\beta$  phase should contain more heavy chemical elements. Indeed, according to the results of EDX analysis of the chemical composition of the  $\beta$ -phase regions, a difference in their composition was revealed, table 2. In all studied areas of the  $\beta$  phase with a lighter contrast, the molybdenum equivalent of the chemical composition is higher than in a nearby area with a darker contrast. Molybdenum equivalent was calculated taking into account aluminum [1]. According to the obtained data, the chemical composition of investigated areas of  $\beta$  phase has the maximum difference in the content of aluminum and vanadium, the difference in the content of molybdenum and chromium is less pronounced. The iron content in the studied areas was not estimated due to its small content in the alloy (0.36 at. %) and as a consequence of the large error of its analysis.



**Figure 3.** HAADF-STEM image showing the lamination of  $\beta$  phase in Ti-5553 alloy after first stage of heat treatment. (a). Bright-field TEM images showing the lamination region (b), electron diffraction pattern of the region (c) and areas on HAADF-STEM images for EDX analysis (d-e).

In the structure of the Ti-5553 alloy after the second stage of heat treatment, aging at 530 °C, there is observed of fine secondary  $\alpha$  phase, figure 4a. Precipitation of secondary  $\alpha$  phase is resulted in dispersion hardening of the alloy. The dispersed particles have a large number of interfacial

boundaries, which serve as barriers to the movement of dislocations. In the  $\beta$  phase between the plates of the primary  $\alpha$  phase, the secondary phase is allocated in limited volumes, mainly due to the size limitations of the areas (first factor) and the lower defect density near the interfacial boundaries. During the aging process secondary  $\alpha$  phase first precipitated in the free areas between the colonies, so the concentration of  $\beta$  stabilizers in unit volume of the  $\beta$  phase increases, which is the second limiting factor for the formation of secondary  $\alpha$  phase between the plates in the colonies. The lamination which was observed after annealing at 700 °C was observed also after subsequent aging at 530 °C. Secondary  $\alpha$  phase and contrast “bands” (lamination) in the  $\beta$  phase can be observed between the primary  $\alpha$  phase (large bright particles) in Figure 4b. The density of secondary  $\alpha$  phase formation in the free regions of the matrix  $\beta$  phase is typical. There is no relation between a place of precipitate of the secondary  $\alpha$  phase and the lamination of the  $\beta$  solid solution, figure 4a, b. Analysis of chemical composition of  $\beta$  phase after aging revealed increase content of vanadium, chromium, while the content of molybdenum decreased slightly. It should be noted that the EDX analysis is a semi-quantitative method of calculating the chemical composition, so the results which can be influenced by various factors and the calculation model itself is not ideal. Thus, for example, the calculated molybdenum content depends on the energy line which can be selected for analysis (K or L-line). In this study for analyzed elements were used K-lines of spectrum. The main aim of present work was analyzed the chemical composition of two closely spaced regions of the  $\beta$  phase, so for this analysis the influence of the geometry of experiment conditions is minimal and the obtained data revealed the limitations of the  $\beta$  phase on  $\beta$ -enriched and  $\beta$ -lean regions.



**Figure 4.** SEM (a) and HAADF-STEM (b) micrographs of solution treated and aged specimen. In (b) showing areas for EDX chemical analysis.

**Table 2.** Compositions of  $\beta$  lean and  $\beta$  rich phases regions (at. %).

Area	Al (at. %)	V (at. %)	Cr (at. %)	Mo (at. %)	Mo eq.
№ 1	6.60	5.95	2.14	3.72	11.5
№ 2	<b>5.10</b>	<b>6.30</b>	<b>3.35</b>	<b>3.68</b>	<b>14.5</b>
№ 3	7.52	4.97	3.58	3.40	12.2
№ 4	<b>6.64</b>	<b>6.78</b>	<b>3.60</b>	<b>3.45</b>	<b>14.1</b>
№ 5	6,81	7.77	3.88	2.72	13.8
№ 6	<b>7.30</b>	<b>10.22</b>	<b>3.75</b>	<b>2.91</b>	<b>15.4</b>

#### 4 Conclusions

The decomposition of  $\beta$  phase and subsequent  $\alpha$  precipitation in the Ti-5553 alloy have been investigated by TEM and XRD technics. A new effect for lamination of  $\beta$  phase is revealed. It was found that the  $\beta$  phase separates into periodic “bands” with variation of alloying elements and

represent lean and rich  $\beta$  phase regions. The lamination of  $\beta$  phase does not have impact to homogeneity precipitation of the secondary  $\alpha$  phase.

### Acknowledgements

This article was prepared with the financial support of Ural Federal University Competitiveness Enhancement Program – CEP 3.1.1.1-18.

### References

- [1] J. D. Cotton, R. D. Briggs, R. R. Boyer, et al., State of the art in beta titanium alloys for airframe applications, *JOM* 67 (6) (2015) 1281–1303.
- [2] J.D. Cotton, R.R. Boyer, R.D. Briggs, R.G. Baggerly, C.A. Meyer, M.D. Carter, W. Wood, G. Tewksbury, V. Li, and X. Yao: *Ti-2007: Science and Technology*, M. Ninomi, S. Akiyama, M. Ikeda, M. Hagiwara, and K. Maruyama, eds., The Japan Institute of Metals, Kyoto, 2007, pp. 471–74.
- [3] M. Harper, R. Williams, G.B. Viswanathan, J. Tiley, R. Banerjee, D.J. Evans, and H.L. Fraser: *Titanium 2003: Science and Technology*, G. Lutjering and J. Albrecht, eds., Wiley-VCH, Hamburg, 2003, vol. 3, pp. 1559–66.
- [4] S. Shekhar, R. Sarkar, S. Kumar Kar, A. Bhattacharjee, Effect of solution treatment and aging on microstructure and tensile properties of high strength  $\beta$  titanium alloy, Ti–5Al–5V–5Mo–3Cr, *Materials and Design* 66 (2015) 596–610.
- [5] J.C. Fanning and R.R. Boyer: *Titanium 2003: Science and Technology*, G. Lutjering and J. Albrecht, eds., Wiley-VCH, Hamburg, Germany, 2003, vol. IV, pp. 2635–42.
- [6] A. G. Illarionov, I. V. Narygina, M. S. Karabanalov, et al., Structural and Phase Transformations in a Titanium Alloy of the Transition Class under the Effect of Deformation, *The Physics of Metals and Metallography*, 110 (2010) 279–288.
- [7] S. V. Zharebtsov, M. A. Murzinova, M. V. Klimova, et al., Microstructure evolution during warm working of Ti–5Al–5Mo–5V–1Cr–1Fe at 600 and 800 °C, *Materials Science & Engineering A* 563 (2013) 168–176.
- [8] P. Barriobero-Vila, G. Requena, S. Schwarz, et al., Influence of phase transformation kinetics on the formation of  $\alpha$  in a  $\beta$ -quenched Ti–5Al–5Mo–5V–3Cr–1Zr alloy, *Acta Materialia* 95 (2015) 90–101.
- [9] V. P. Skripov, A. V. Skripov, Spinodal decomposition, *Advances in physical Sciences*, Vol. 128, № 2, 1979, pp. 193–231.
- [10] Z. Fan, A. P. Miodownik, TEM study of metastable  $\beta$ -phase decomposition in rapidly solidified Ti–6Al–4V alloy, *Journal of materials science* 29 (1994) 6403–6412.
- [11] C. R. M. Afonso, P. L. Ferrandini, A. J. Ramirez, R. Caram, High resolution transmission electron microscopy study of the hardening mechanism through phase separation in  $\beta$ -Ti–35Nb–7Zr–5Ta alloy for implant applications, *Acta Biomaterialia* 6 (2010) 1625–1629.
- [12] A. Devaraj, S. Nagand R. Banerjee, Alpha phase precipitation from phase-separated beta phase in a model Ti–Mo–Al alloy studied by direct coupling of transmission electron microscopy and atom probe tomography, *Scripta Materialia* 69 (2013) 513–516.
- [13] C. Ghosh, J. Basu, D. Ramachandran, E. Mohandas, Phase separation and  $\omega$  transformation in binary V–Ti and ternary V–Ti–Cr alloys, *Acta Materialia* 121 (2016) 310–324
- [14] Y. M. Zhu, S. M. Zhu, M. S. Dargusch, J. F. Nie, HAADF-STEM study of phase separation and the subsequent  $\alpha$  phase precipitation in a  $\beta$ -Ti alloy, *Scripta Materialia* 112 (2016) 46–49.
- [15] M.S. Kalienko, A.V. Volkov, V.A. Kropotov, et al., Primary  $\alpha$ -Phase VST5553 Alloy with Lamellar Structure Properties Effect // *Titanium'2011: Science and technology. Proc. 12-th Int. Conf. of Titanium. Beijing, China, 2011.* – pp. 1303–1311.