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Influence of annealing on the microstructure of cast TiAl-based alloy

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ABSTRACT

Purpose: The aim of the present work is to study the influence of annealing on the microstructure of cast TiAl-based alloy.

Design/methodology/approach: Effect of annealing on the microstructure of cast Ti-46Al-8Ta (at.%) alloy was evaluated at temperatures ranging from 700 to 800°C. Fine grain convoluted microstructure of heat treated alloy consisted of small lamellar colonies of α2-Ti3Al (crystal structure D019) intermetallic phase within γ-TiAl (crystal structure L10) phase.

Findings: Annealing leads to precipitation of α2 and τ (crystal structure B82) particles at the grain boundaries at the expense of the α2 laths. This process is connected with a depletion of the grain boundaries of Ti and Ta and formation of Al rich γ phase. Lattice parameter a of the γ phase decreases, c increases and aspect ratio c/a increases with increasing time and temperature of ageing.

Research limitations/implications: The annealing leads to an increase of lattice parameters *a* and *c* and decrease of the aspect ratio *c/a* of both α2 and τ phases.

Originality/value: This work present the effect of long-term annealing on microstructure stability and lattice parameters of coexisting phases in this alloy at temperatures ranging from 700 to 800°C.

Keywords: TiAl; Microstructure; Ageing; Lattice parameters

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MATERIALS

1. Introduction

Cast TiAl-based alloys due to their interesting properties like excellent specific strength related to the density are attractive materials for various high-temperature structural applications in the gas turbine and automotive industry [1]. The Al-Ta-Ti system attracted recent interest and a new "airhardenable" Ti-46Al-8Ta (at.%) alloy was designed, since tantalum containing TiAl-based alloys can be grain refined via formation of highly faulted massive γ_M(TiAl) through a diffusionless transformation initiated by

cooling at required rate from single α phase (Ti-based solid solution with hexagonal crystal structure) field [2-7]. Hu et al. [4] showed that elements such as Nb and Ta have low diffusivity in TiAl alloys and favour the massive transformation over the lamellar formation at low cooling rates. Ta has a greater effect than Nb in extending the massive regime to low cooling rate side in TiAl alloys [4]. The microstructure of Ti-46Al-8Ta (at.%) alloy according the ternary Al-Ta-Ti phase diagram proposed by Kubaschewski [8] was expected to contain γ-TiAl (tetragonal crystal structure $L1_0$), α_2-Ti_3Al (hexagonal crystal structure $D0_{19}$) and σ-Ta_2Al phase with tetragonal crystal structure $D8_b$, space group $P4_2/mnm$. Recently modelled new ternary Al-Ta-Ti phase diagram [6] proposed that two-phase α_2 + γ microstructure of the Ti-46Al-8Ta (at.%) alloy should be stable below 1050°C. In contrast to the proposed Al-Ta-Ti phase diagrams, new ternary Ta-rich phase was identified in this alloy after long-term ageing and creep testing at temperatures from 700 to 800ºC [9]. The hexagonal crystal structure of $B8_2$ type and lattice parameters of this new τ - phase were estimated by Lapin et al. [10]. Based on these examinations, the Al-Ta-Ti phase diagram was re-evaluated [5,10]. According to the new thermodynamic calculations, the initial microstructure of the Ti-46Al-8Ta (at.%) alloy is expected to transform to the equilibrium γ + τ type below 870°C during long-term ageing, as shown in Fig. 1. However, this calculated type of microstructure has not been proved experimentally yet. Hence, the study of long-term microstructural stability of this alloy at designed operating temperatures is of great practical interest. Further experiments are still required to refine the position of the boundary between $\alpha_2 + \gamma + \tau$ and $\gamma + \tau$ phase regions. The aim of the present work is to study the effect of long-term annealing on microstructure stability and lattice parameters of coexisting phases in this alloy at temperatures ranging from 700 to 800ºC.

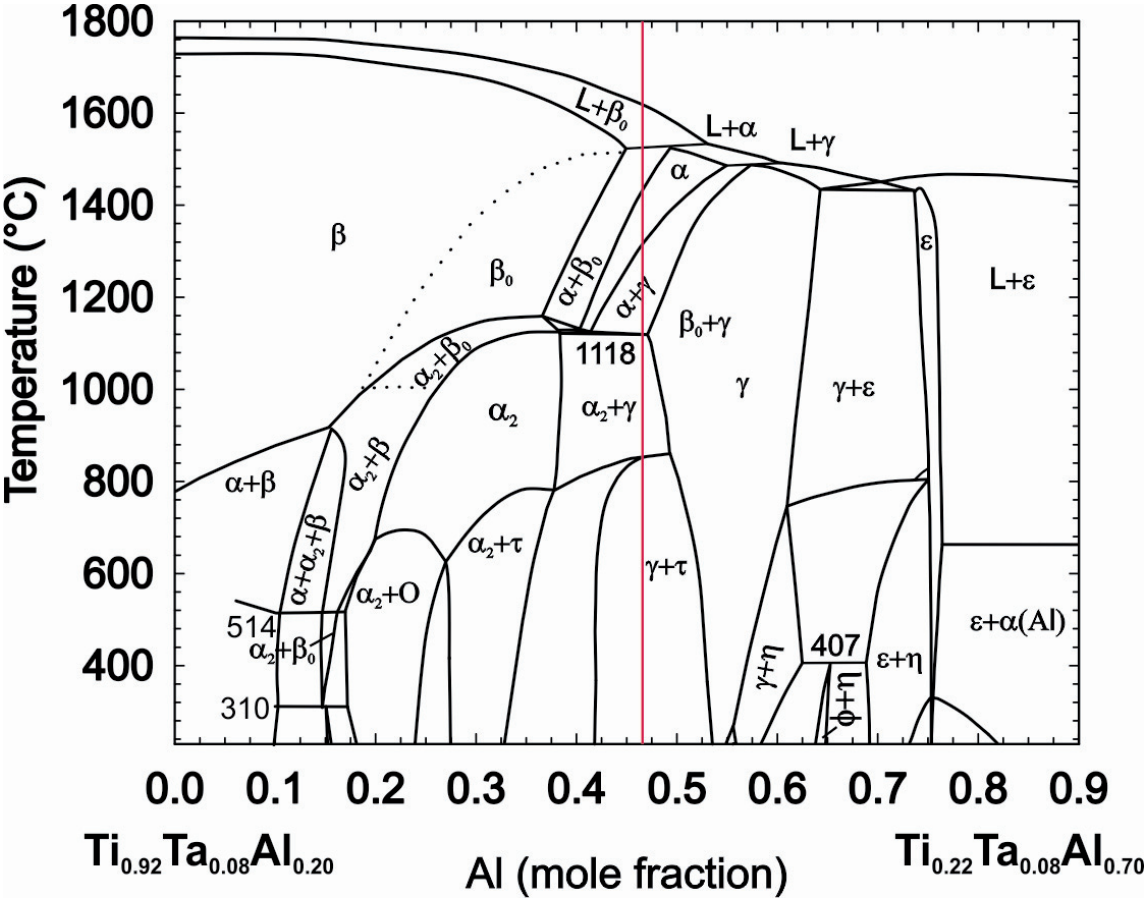

Fig. 1. Calculated isopleth for 8 at.% Ta of the Ti-Al-Ta systems covering the Al composition in the range of 20-70 at.% [5,10]

2. Experimental procedure

Cylindrical Ti-46Al-8Ta (at.%) samples for annealing experiments with a diameter of 13 mm and thickness of 5 mm were prepared from centrifugally and heat treated bars as described elsewhere [10]. Long-term annealing experiments were performed at temperatures ranging from 700 up to 800ºC for various times from 100 to 30000 h. After each ageing step, the microstructure evaluation was performed by scanning electron microscopy (SEM), backscattered scanning electron microscopy (BSEM), X-ray diffraction (XRD) analysis and energy-dispersive X-ray (EDX) spectroscopy. SEM samples were prepared using standard metallographic techniques and etched in a solution of 150 ml H_2O, 25 ml HNO_3 and 10 ml HF. XRD analysis was carried out using Bruker D8 DISCOVER diffractometer equipped with X-ray tube with rotating Cu anode operating at 12 kW. All measurements were performed in parallel beam geometry with parabolic Goebel mirror in the primary beam. Diffraction patterns were measured within an angular range 20-110° of 2θ with an exposition time of 6 s and step size of 0.01°. The obtained diffraction data were analysed quantitatively using the program EVA 11.0. The further refinement of the patterns to identify the lattice parameters of coexisting phases was performed using TOPAS 3.0. The instrumental line profile was determined from the measurements of polycrystalline texture of free corundum with sufficiently large crystallite size at the same angular range. Volume fractions of coexisting phases and size of lamellar colonies were determined from digitalized BSEM micrographs using a computer image analyser.

3. Results and discussion

3.1. Effect of annealing on microstructure

Figure 2 shows the microstructure of the as-received Ti-46Al-8Ta (at. %) alloy. Convoluted type of microstructure consisting of γ-TiAl (tetragonal crystal structure $L1_0$) and α_2-Ti_3Al (hexagonal crystal structure $D0_{19}$) intermetallic phases is typical for fine grains. α_2 phase with morphology of lamellae much shorter than the individual γ grains precipitated in the form of small lamellar colonies [11]. As shown by Saage et al. [12], this type of microstructure is formed by precipitation of α and/or α_2 phases on four equivalent {111} planes of massively transformed γ_M during the second HIP-ing at 1260°C and cooling from two phase α + γ field. As was shown in [9], the α_2 and γ lamellae maintain the Blackburn crystallographic orientation relationships in a form $(0001)\alpha_2 \parallel \{\bar{1}\,\bar{1}\,1\}\gamma$ and $(11\bar{2}0)\alpha_2 \parallel \langle 1\,\bar{1}\,0\,]\,\gamma$. A mean length and average volume fraction of the α_2 laths were measured to be 8.5 μm and (29.8 ± 2.3) vol.%, respectively. For about 10 vol.% of lamellar colonies retained in this microstructure [11]. Small γ grains and irregular particles of α_2 phase were found on the grain boundaries in the as-received alloy, as shown in BSEM micrograph (Fig. 3a).

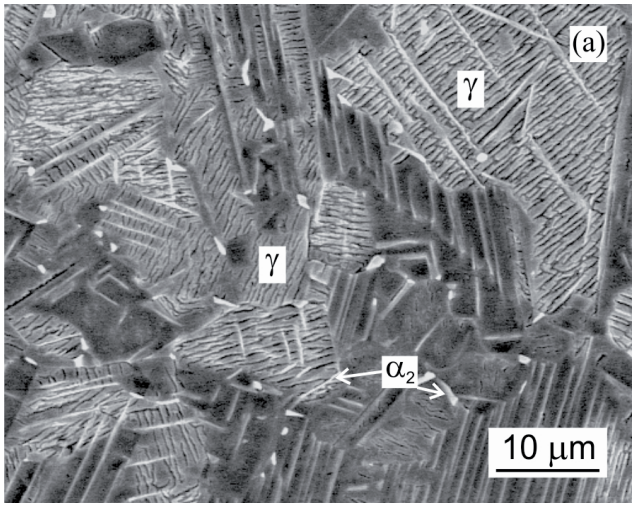

Fig. 2. SEM micrograph showing fine grain structure of as-received Ti-46Al-8Ta (at.%) alloy

Figure 3b shows the typical BSEM microstructure of the alloy after annealing at 700°C for 30000 h. Comparing the as-received microstructure, one can see (i) a lower volume fraction of α_2 lamellae, (ii) precipitation of bright coloured phase predominantly at the grain boundaries and (iii) the dark coloured grain boundaries in the annealed samples. As proposed Kubaschewski [8], ternary Al-Ta-Ti alloy was expected to contain γ-TiAl, α_2-Ti_3Al and σ-Ta_2Al phase during annealing. The evolution XRD patterns of the studied alloy revealed that the initial α_2 + γ microstructure really transforms to the ternary one during annealing, as shown in Fig. 4. Diffraction peaks correspond to the position of the peaks of α_2 and γ phases and new τ-phase with $B8_2$ type crystal structure (space group $P6_3/mmc$, Pearson symbol hP6), which described recently Lapin et al. in [10].

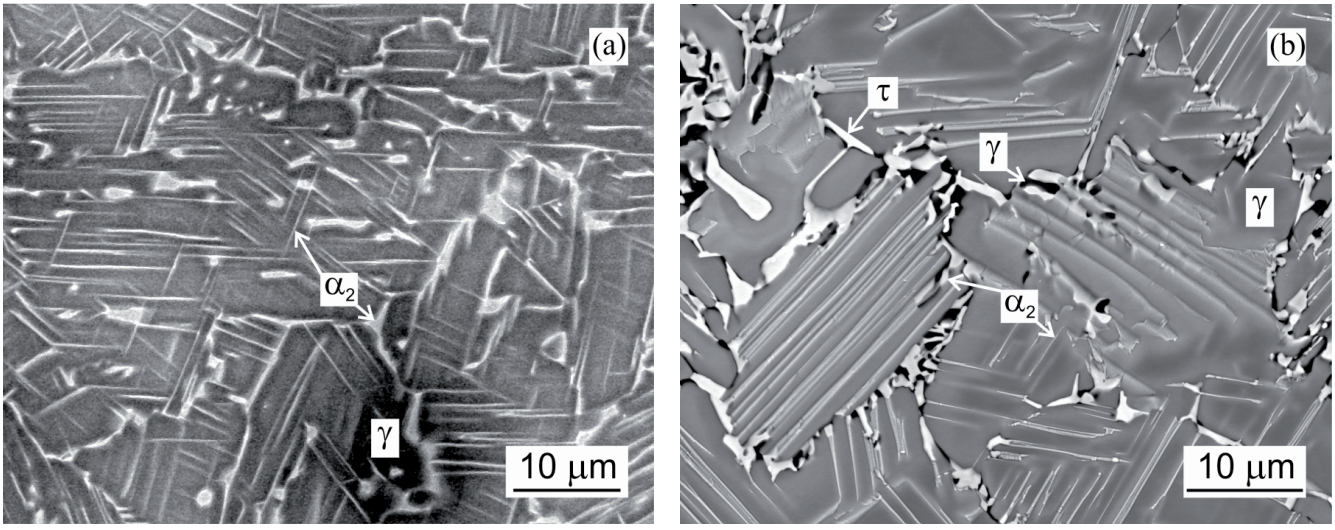

Fig. 3. BSEM micrographs showing the microstructure of Ti-46Al-8Ta (at.%) alloy: (a) as-received alloy composed of α_2 and γ phase, (b) microstructure after annealing at 700°C for 30000 h

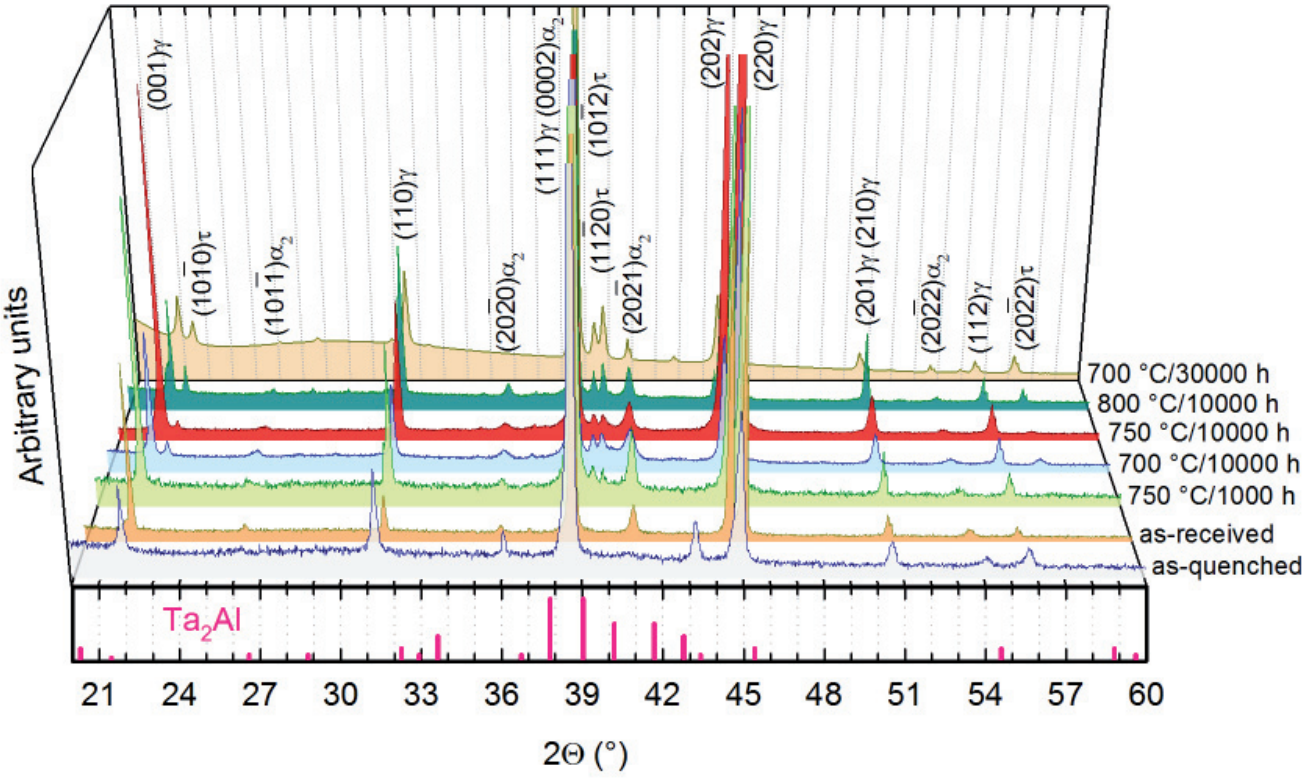

Fig. 4. XRD patterns of the γ_M, as-received and aged Ti-46Al-8Ta (at.%) alloy. The annealing regimes are indicated in the figure

No diffraction peaks correspond to the position of the peaks of the σ-phase (see the position 2Θ diffraction peaks of σ-phase shown in Fig. 4). Clear diffraction peaks of the τ-phase were found in the XRD patterns after annealing for 1000 h at 750°C. Similar ternary Ti_4Al_3Nb phase with hexagonal crystal structure was reported by Witusiewicz et al. [13] for Ti-46Al-8Nb (at.%) alloy. EDX analysis showed that some particles preferentially formed at the grain boundaries in the annealed samples (Fig. 3b) belong to the α_2 phase and other to the new τ phase with the chemical composition close to Ti-(36-40)Al-(12-15)Ta (at.%). In addition, Fig. 4 contains XRD pattern of the as-quenched sample, which was subjected to solution annealing at 1360°C for 1 h and compressed air cooling to form massive γ_M. One can clearly see the shifts between the diffraction peaks between γ_M, γ and α_2 phases. Measured values of volume fraction of the coexisting α_2, γ and τ phases with the ageing time at temperatures from 700 up to 800°C [9] showed that increase of the annealing temperature and time lead to an increase of the volume fractions of the τ and γ phases at the expense of a decrease of the volume fraction of the α_2-phase. Mean length of the α_2 laths was measured to vary between 8.5 and 9.2 mm at all ambient temperatures and annealing times. However, annealing as long as 30000 h at 700°C is still insufficiently long to fully transform the initial $\alpha_2 + \gamma$ microstructure to the $\gamma + \tau$ type that is expected from the new calculated phase diagram at temperatures below 870ºC [5,10], as shown in Fig. 1.

3.2. Effect of annealing on lattice parameters of coexisting phases

Tables 1, 2 and 3 summarize measured values of lattice parameters of coexisting phases in the studied alloy before and after annealing at temperatures of 700, 750 and 800°C up to 30000 h. Effect of annealing time on lattice parameters was examined for more detail at a temperature of 750ºC. As shown in Tab. 1, lattice parameter a_γ of the γ-phase decreases with increasing annealing time at 750ºC up to 5000 h, for a longer time a_γ remains constant within the experimental error. The lattice parameter c_γ and aspect ratio $(c/a)_\gamma$ increase with increasing annealing time. The decrease of a_γ and increase of both c_γ and $(c/a)_\gamma$ can be attributed to the redistribution of alloying elements during annealing, mainly to an increase of Al content in the γ-phase [14].

Table 2 shows evolution of the measured values of the lattice parameters of the α_2 phase on the annealing time. Both lattice parameters $a_{\alpha 2}$ and $c_{\alpha 2}$ increase and the aspect ratio $(c/a)_{\alpha 2}$ linearly decreases with increasing annealing time.

Table 1.
Lattice parameters for γ phase with $L1_0$ crystal structure, P4/mmm

Annealing, °C/h	a, 10^{-10} m	c, 10^{-10} m	c/a	Notes
Standard TiAl	*4.0050*	*4.0700*	*1.0162*	*XRD data*
As cast alloy	4.0199±0.0007	4.0628±0.0006	1.0106±0.0004	fitted using TOPAS
700/10012	4.0180±0.0005	4.0638±0.0006	1.0113±0.0003	fitted using TOPAS
700/30000	4.0178±0.0005	4.0641±0.0006	1.0115±0.0004	fitted using TOPAS
750/500	4.0199±0.0004	4.0629±0.0004	1.0106±0.0003	fitted using TOPAS
750/2000	4.0194±0.0005	4.0631±0.0006	1.0108±0.0004	fitted using TOPAS
750/5000	4.0180±0.0005	4.0632±0.0004	1.0112±0.0003	fitted using TOPAS
750/8000	4.0179±0.0006	4.0634±0.0006	1.0113±0.0002	fitted using TOPAS
750/10000	4.0180±0.0005	4.0640±0.0006	1.0114±0.0002	fitted using TOPAS
800/10030	4.0179±0.0004	4.0640±0.0004	1.0114±0.0003	fitted using TOPAS

Table 2.
Lattice parameters for α_2 phase with $D0_{19}$ crystal structure, $P6_3/mmc$

Annealing, °C/h	a, 10^{-10} m	c, 10^{-10} m	c/a	Notes
Standard Ti_3Al	*5.7930*	*4.6490*	*0.8025*	*XRD data*
As cast alloy	5.7759±0.0004	4.6556±0.0006	0.8060±0.0004	fitted using TOPAS
700/10012	5.7905±0.0002	4.6575±0.0003	0.8043±0.0003	fitted using TOPAS
700/30000	5.7990±0.0002	4.6586±0.0004	0.8033±0.0003	fitted using TOPAS
750/500	5.7778±0.0004	4.6561±0.0006	0.8058±0.0003	fitted using TOPAS
750/2000	5.7817±0.0004	4.6570±0.0005	0.8054±0.0002	fitted using TOPAS
750/5000	5.7840±0.0004	4.6575±0.0006	0.8052±0.0002	fitted using TOPAS
750/8000	5.7900±0.0004	4.6586±0.0006	0.8045±0.0003	fitted using TOPAS
750/10000	5.7921±0.0004	4.6592±0.0006	0.8044±0.0003	fitted using TOPAS
800/10030	5.7922±0.0004	4.6589±0.0006	0.8043±0.0004	fitted using TOPAS

Table 3.
Lattice parameters for τ phase with $B8_2$ crystal structure, $P6_3/mmc$

Annealing, °C/h	a, 10^{-10} m	c, 10^{-10} m	c/a	Notes
700/10012	4.5767±0.0006	5.5175±0.0006	1.2056±0.0003	fitted using TOPAS
700/30000	4.5814±0.0007	5.5233±0.0009	1.2056±0.0003	fitted using TOPAS
750/5000	4.5660±0.0018	5.5162±0.0007	1.2081±0.0004	fitted using TOPAS
750/8000	4.5692±0.0019	5.5168±0.0006	1.2074±0.0002	fitted using TOPAS
750/10000	4.5771±0.0016	5.5181±0.0007	1.2056±0.0003	fitted using TOPAS
800/10030	4.5772±0.0006	5.5183±0.0006	1.2056±0.0004	fitted using TOPAS

Table 3 shows measured lattice parameters of the τ-phase. The calculated lattice parameters a_τ and c_τ increase and $(c/a)_\tau$ ratio decreases with increasing annealing time. The change of the lattice parameters of the τ-phase is connected with redistribution of alloying elements among the coexisting α_2, γ and τ phases during annealing.

Effect of annealing temperature on lattice parameters of the coexisting α_2, γ and τ phases can be illustrated on the samples annealed for 10000 h (Tabs. 1-3). Since the microstructure of the studied alloy evolves from the metastable massive γ_M, the measurements of lattice parameters of the γ_M-phase have been also performed on samples subjected to solution annealing at 1360ºC for 1 h and air cooling to room temperature. The quantitative analysis of XRD diffraction data (shown in Fig. 4) of samples with the γ_M microstructure results in $a_{\gamma M}$ = (0.4033 ± 0.0004) nm, $c_{\gamma M}$ = (0.4049 ± 0.0004) nm and aspect ratio of $(c/a)_{\gamma M}$ = (1.0040 ± 0.0002). When compared with those for the as-received alloy, the lattice parameter a_γ of γ-phase decreases and c_γ increases after 10000 h annealing at 700-800ºC. The change of the γ_M and γ phases during the precipitation of α/α_2 laths and subsequent long-term annealing can be attributed to the redistribution of alloying elements during annealing, mainly to an increase of

Al content in the γ-phase [14]. The lattice parameters $a_{\alpha 2}$ and $c_{\alpha 2}$ of the α_2-phase increase with annealing temperature. Applied annealing temperatures from 700 to 800ºC have no serious effect on the average values of the lattice parameters *a* and *c* of both γ and α_2 phases nor on lattice parameters a_τ and c_τ of the τ phase which remain statistically the same during annealing. It should be noted that both the lattice parameters of the τ phase increased with increasing time up to 10000 h during annealing at 750ºC.

4. Conclusions

The investigation of the development of microstructure and lattice parameters of coexisting intermetallic phases in Ti-46Al-8Ta (at.%) alloy during annealing at 700, 750 and 800ºC suggests the next conclusions:

1. The thermodynamically unstable $\alpha_2 + \gamma$ microstructure transforms to the $\alpha_2 + \gamma + \tau$ type during long-term annealing. Particles of the τ-phase are preferentially formed along the grain and lamellar colony boundaries at the expense of the α_2 laths, which partially transform to the γ matrix and τ particles.
2. The measured lattice parameters of the γ, α_2 and τ phases change with the annealing time. The lattice parameter *a* decreases and the lattice parameter *c* and the aspect ratio *c/a* increase with increasing annealing time for the γ-phase. The lattice parameters *a* and *c* increase and the aspect ratio *c/a* decreases with increasing annealing time for both τ and α_2 phases.
3. The lattice parameters of the γ and α_2 phases change during annealing compared with those of the as-received alloy. The annealing temperature has no measurable effect on the lattice parameters of coexisting γ, α_2 and τ phases after 10000 h annealing.

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