Phase transformation sequence of Ti-6Al-4V as a function of the cooling rate

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Abstract. The growth kinetics of allotriomorphic α along the prior β grain boundaries and of globular primary α in Ti-6Al-4V during continuous cooling is described. A physical model is developed based on classical nucleation and growth of platelets for the allotriomorphic α . The growth of the primary α is modelled based on the growth of spherical particle immersed on a supersaturated β -matrix. Continuous cooling tests at two different holding temperatures in the $\alpha+\beta$ field, 930°C and 960°C, and five different cooling rates, 10, 30, 40, 100 and 300°C/min, are conducted to validate the proposed models and elucidate the growth sequence of those α morphologies. Additionally, interrupted tests at different temperatures are conducted to determine the progress of growth of primary α and formation allotriomorphic α during cooling. The size of primary α increases while its size distribution broadens with a decrease in cooling rate. Area fractions of primary α decrease with increasing cooling rate and increasing holding temperature. Moreover, the lower the cooling rate, the thicker the plates of allotriomorphic α . At the beginning of the cooling, growth of primary α , as well as formation of allotriomorphic α plates is observed. The experimental and modelled results show good agreement.

Introduction

The mechanical properties of Ti-6Al-4V, especially fatigue resistance, toughness and ductility, are strictly correlated with its microstructure formed during the industrial thermo-mechanical treatments [1]. Different microstructures can be achieved, i.e. martensitic, lamellar, equiaxed or bimodal, and they are mainly related to the cooling rate during thermo-mechanical treatment. A bimodal microstructure consisting of lamellar α and globular primary α (α_p) is normally desired owing to the combination of high ductility and high toughness [1]. The globular α_p phase grows during cooling, and the allotriomorphic α phase is formed along the grain boundaries.

The growth of α_p phase is a diffusion-controlled process. For very slow cooling rates, the β phase transforms mainly into α_p . Thus, the resulting microstructure consists almost entirely of large equiaxed α_p with small amounts of retained β phase [2]. The amount of α_p decreases for increasing cooling rates and other morphologies of α phase are formed [3]. Semiatin et al. [4] observed that α_p exhibits epitaxial growth for Ti-6Al-4V, and that it is controlled by the diffusion of vanadium. The presence of a rim- α phase in a near α Ti60 [3] surrounding the α_p during cooling with the same crystallographic orientation, evidences the epitaxial growth of α_p phase. The nucleation and growth of allotriomorphic α (α_{GB}) is dependent on the cooling rate and follows a platelet-like formation [2],[4]. Extensions from α_p with same crystallographic orientation were observed preferentially along the β/β boundary suggesting symbiotic growth from the α_p phase [3].

Semiatin et al. [4] proposed a model for the growth of α_p that uses exact solutions of the diffusion equation and takes into consideration: a) the diffusion coefficients with a thermodynamic correction for the specific composition of the material, and b) the large super-saturation formed during cooling [4]. Meng et al. [5] complemented the model proposed in [4] by considering the effect of the thermal history on the diffusion field of growing particles, and the overlap of these diffusion fields [6].

The development of robust, accurate but computational simple models to simulate the $\beta \rightarrow \alpha$ transformation are of great importance to predict and control the evolution of the microstructure in complex shape and variable cross sections components during thermomechanical processing. A coupled model for the growth of primary α and formation of allotriomorphic α phases is here proposed. The results are compared with the measured data acquired for different continuous cooling experiments.

Experimental procedures

Material

A cogged Ti-6Al-4V in the β and $\alpha+\beta$ fields with further annealing at 730°C for 1 hour followed by air cooling was used for this investigation. The estimated β transus is ~1020°C [7]. Cylindrical samples with a diameter of 5.5 mm and a length 10 mm were utilized for the heat treatments in a dilatometer.

Table 4: Chemical composition of investigated Ti-6Al-4V [wt.%] [7]

Al	V	Fe	O	C	N	H	Y
6.54	4.21	0.20	0.185	0.028	0.023	0.000335	<0.001

Heat treatments

A dilatometer DIL 805A/D (TA Instruments, Hüllhorst, Germany) was used to perform continuous cooling heat treatments. The tests were carried out in a protective atmosphere of argon. After heating with a rate of 30°C/min, the samples were held for 1 h at two different holding temperatures in the $\alpha+\beta$ field, 930 and 960°C, followed by continuous cooling until room temperature. The cooling was conducted using five different cooling rates: 10, 30, 40, 100 and 300°C/min. Interrupted continuous cooling heat treatments were carried out in order to elucidate the mechanism/s governing the transformation during cooling. After heating with 30°C/min, the samples were held for 60 min at a constant temperature of 930°C or 960°C. The subsequent cooling was performed with 10 and 100°C/min. The samples were quenched using Argon at four different temperatures, 900, 875, 850 and 800°C.

Metallography and microstructure investigation

The samples were polished using OP-S (oxide polishing suspension) after a conventional grinding procedure from grit 500 until 2000. The samples were etched with Kroll's reagent, 91 ml water, 6 ml HNO₃ and 3 ml HF. The metallographically prepared and etched samples were investigated using light optical (LOM) and scanning electron (SEM) microscopy. The SEM analysis were conducted using a Tescan Mira3 microscope using an acceleration voltage of 10 kV and working distance of 12 mm. A minimum of five representative micrographs were analysed for each cooling rate and holding temperature for the quantification of globular α_p . The globular phases were marked using the software GIMP (GNU Image Manipulation Program) and analysed with ImageJ software.

Modelling strategy

The growth of α_p during cooling in heat treatments conducted below the β -transus temperature was modelled based on [4]. The growth of allotriomorphic α was modelled based on a classical model of nucleation and diffusion equation for growth of platelet. The microstructure is modelled as consisting of three major components: α_p , α_{SEC} and α_{GB} , as schematically shown in Figure 1. The nucleation and growth of α_{SEC} is not considered in the present model.

Figure 1: a) Schematic representation of the different morphologies of the alpha phase: primary (α_p), secondary (α_{SEC}) and allotriomorphic (α_{GB}), formed during cooling of a typical Ti-6Al-4V alloy; b) growth of a spherical particle of radius R and composition C_p immersed in a matrix of composition C_M , in which C_I is the chemical composition at the interface.

For Ti-6Al-4V Semiatiin et al. [4] showed that the growth rate of globular α_p phase is limited only by V diffusion. The intrinsic diffusion coefficient of V in beta titanium (D) is considered as obtained from Zwicker [8] and also adopted in the model and simulations of Semiatiin et al. [4].

The growth of a spherical particle immersed in a matrix of composition C_M can be given according to Equation 1.

$$\frac{dR}{dt} = 2\lambda^2 \frac{D}{R} \quad \text{Equation 1}$$

Where R is the radius of the particle, D is the diffusion coefficient and λ is a parameter that can be calculated according to Equation 2.

$$\left\{ \lambda^2 e^{\lambda^2} \right\} \left[e^{-\lambda^2} - \lambda \sqrt{\pi} \operatorname{erfc}(\lambda) \right] = \Omega/2 \quad \text{Equation 2}$$

The parameter Ω denotes supersaturation, and can be calculated according to Equation 3.

$$\Omega = \frac{(C_M - C_I)}{(C_P - C_I)} \quad \text{Equation 3}$$

Where C_I is the composition of the matrix-particle interface, C_p is the composition of the α_p phase. C_M is considered as the equilibrium phase composition of the alloy at the actual temperature, and C_p is considered as the equilibrium phase composition of the α phase. The equilibrium phase fraction as well as the chemical composition were calculated using the software JMatPro[®] v.10. In order to account for the soft impingement on the “far-field” matrix composition, C_M is calculated using a usual mass balance between the fraction of α (f_α) and β phases, as given by Equation 4.

$$C_M = \frac{(C_0 - f_\alpha C_\alpha)}{(1 - f_\alpha)} \quad \text{Equation 4}$$

Where C_0 is the nominal concentration of V in the material and C_α is the concentration of V in the α phase. Similar to the growth of α_p , the partial enrichment of V along the formed α/β phase boundary is observed. However, this phenomenon stabilizes locally the β phase. Thus, a plate-like morphology of α phase is obtained along the β/β grain boundaries. When the nucleation time is neglected, the rate of nucleation of precipitates can be given according to Equation 5.

$$\frac{dN}{dt} = N_0 (1 - f_{\alpha p} - f_{\alpha GB}) \exp\left(-\frac{Q}{RT}\right) \exp\left(-\frac{\Delta G_{Nuc}^*}{RT}\right) \quad \text{Equation 5}$$

Where N_0 is a pre-exponent term and considered as 4.5×10^4 for a prior β grain size of 200 μm , Q is the activation energy for diffusion, ΔG_{Nuc}^* is the energy barrier for heterogeneous nucleation, R the molar gas constant and T the temperature. $f_{\alpha p}$ and $f_{\alpha GB}$ are the volume fraction of α_p and allotriomorphic α formed along the prior beta grain boundaries, respectively. The energy barrier for heterogeneous nucleation can be calculated according to Equation 6.

$$\Delta G_{Nuc}^* = -\frac{(A^*)^3}{(RT)^2 \left[\ln\left(\frac{C_M}{C_I}\right) \right]^2} \quad \text{Equation 6}$$

Where A^* is a parameter obtained according to the nucleation at grain boundary, or sympathetic nucleation [9], or a sum of contributions of both (adopted in the current model).

The critical thickness (B_{crit}) for a disk-like α_{GB} to nucleate can be calculated according to Equation 7.

$$B_{crit} = -\frac{4\gamma_{\alpha\beta}}{\Delta G_V} \quad \text{Equation 7}$$

Where $\gamma_{\alpha\beta}$ is the interface energy between α and β phases, ΔG_V is the chemical free energy of phase transformation obtained using Equation 8 [9].

$$\Delta G_V = \frac{C_I - C_P^{\alpha GB}}{1 - C_I} \frac{RT}{\left(1 + \frac{\partial \ln V}{\partial C_I}\right)} \ln\left(\frac{C_I}{C_M}\right) \quad \text{Equation 8}$$

Where $C_P^{\alpha GB}$ is the concentration of V in the α_{GB} , considered to be equal to the V concentration in the α_p ($C_P^{\alpha GB} = C_p$).

The nucleus of α_{GB} will grow by diffusion process, and it is considered to be similar to the growth of the platelets, i.e. thickening of a planar disordered boundary via ledge growth mechanism. In this case, the lengthening of a platelet is significantly faster than the thickening due to high anisotropy in interfacial energy. Therefore, the volume fraction of α_{GB} is only dependent on the number density and its thickness. The thickening of the α_{GB} can be then modelled according to Equation 9.

$$\frac{dB}{dt} = \frac{2m\lambda_{GB}^2 D}{B} \quad \text{Equation 9}$$

Where B is the thickness of the α_{GB} , m is a ledge coefficient to account for the planar disordered growth, D is the diffusivity of V in the β matrix and λ_{GB} is parameter that can be calculated according to Equation 10.

$$\sqrt{\pi} \exp(\lambda_{GB}^2) \operatorname{erfc}(\lambda_{GB}) = \Omega_{\alpha GB} \quad \text{Equation 10}$$

Where $\Omega_{\alpha GB} = (C_I - C_M) / (C_I - C_P^{\alpha GB})$ is a dimensionless supersaturation parameter, and considered equal to Ω because $C_P^{\alpha GB} = C_P^\alpha$. Similar to the growth of precipitates, the mean thickness of the platelets ($\bar{B}$) is calculated according to Equation 11 [10].

$$\frac{d\bar{B}}{dt} = \frac{dB}{dt} + \frac{1}{N} \frac{dN}{dt} (B_{crit} - \bar{B}) \quad \text{Equation 11}$$

The first term corresponds to the growth of the existing platelets of allotriomorphic α , while the second represents the contribution of new nuclei of critical size calculated according to Equation 7.

A Matlab[®] routine was developed to implement the model for the growth of the α_p previously described.

Results and discussion

Figure 2 shows the microstructure of the Ti-6Al-4V argon quenched after 1 h holding at 960°C . Sparsely and nearly separated α_p particles are found in a matrix of martensite (α'), originally a matrix of β phase.

Figure 2: Representative optical micrograph of the investigated Ti-6Al-4V after 1 h at 960°C followed by argon quenching.

From the microstructure shown in Figure 2, different fractions and sizes of α phase are formed during cooling, as shown in Figure 3. The cooling rate of 10°C/min (Figure 4a) lead to a nearly fully equiaxed microstructure, thus mostly growth of α_p is exhibited. It is difficult to distinguish the lamellas of α_{SEC} from the globular α_p for the cooling rate of 10°C/min. The increase in cooling rate leads to formation of α_{SEC} , as well as α_{GB} phase. For 30°C/min and 40°C/min (Figure 3b and Figure 3c, respectively), α_{GB} exhibits irregularities in the interface surface, as well as the α_p phase. Higher cooling rates lead to less pronounced growth of α_p and α_{GB} (Figure 3d and Figure 3e). Moreover, regular plate-like morphology of α_{GB} is observed for 100 and 300°C/min.

Figure 3: Representative light optical micrographs of the investigated Ti-6Al-4V after holding at 960°C for 1 h followed by continuous cooling at: a) 10°C/min; b) 30°C/min; c) 40°C/min; d) 100°C/min; e) 300°C/min.

Figure 4 exhibits the typical micrographs after interrupted continuous cooling heat treatments. The α_{GB} is highlighted in red dotted line, and the α_{SEC} in dotted green circles. The formation of α_{GB} initiates from the globular α_p , i.e. symbiotic growth. The 2D interconnectivity of the plates of α_{GB} along the grain boundaries is very low for 900°C (Figure 4(a,b)). At 850°C the grain boundaries are nearly decorated with α_{GB} . Irregular growth of α_{GB} seems to occur at ~800°C, and is more pronounced for 10°C/min cooling rate (Figure 4g). The nucleation and growth of α_{GB} do not seem to be significant at temperatures higher than 900°C for the two investigated cooling rates, leading to comparable evolution behaviour of the α_{GB} after holding at temperatures of 930°C and 960°C (Figure 4a). Formation of α_{SEC} is not pronounced until ~850°C. Although Figure 4e and Figure 4g show the presence of α_{SEC} for 10°C/min cooling rate, its area fraction is notable small. Similar conclusions can be obtained comparing Figure 3a with Figure 3d.

Figure 4: Representative SE-SEM micrographs of the interrupted heat treatments after holding at 960°C for 1 h followed by continuous cooling at 10°C/min (a,c,e,g) and 100°C/min (b,d,f,h). The tests were interrupted at: a,b) 900°C; c,d) 875°C; e,f) 850°C; g,h) 800°C.

The mean α_p diameter, area fraction of α_p and the mean thickness of α_{GB} is compared for the simulated and measured results in Figure 5. The diameter of α_p increases with decrease of cooling rate and the model predicts with good accuracy this behaviour. The increase in area fraction of α_p is pronounced for decreasing cooling rates. The developed model describes well the area fraction evolution apart from 30 and 40°C/min for

holding temperature of 960°C. The thickness of the α_{GB} was in the range of 1-4 μm for the investigated conditions and it increased with decreasing cooling rates. The differences between 930°C and 960°C are not significant.

Figure 5: Measured and simulated microstructural features for the investigated cooling rates for the holding temperatures of 930°C and 960°C: a) mean α_p phase diameter, and b) area fraction of α_p ; c) measured and simulated thickness of the α_{GB}

To illustrate the results of the developed model, the evolution of the mean diameter of α_p , mean thickness of α_{GB} and vanadium supersaturation is exhibited in Figure 6 for the holding temperature of 960°C. A significant growth of α_p as well as α_{GB} is predicted down to 800°C, which was also observed in Figure 4. The supersaturation of V in β phase matrix is predicted to increase sharply until ~850°C, when the α_{5EC} is observed to be formed more pronounced (Figure 4).

Figure 6: Simulation results for the holding temperature of 960°C: a) mean α_p phase diameter; b) mean thickness of the α_{GB} ; and c) V supersaturation in the β matrix evolution.

Summary and conclusions

The sequence of growth of the α phases (primary, secondary and allotriomorphic) during cooling from an isothermal temperature below the β -transus is clarified and modelled for Ti-6Al-4V. Nucleation of α_{GB} starts at the globular α_p , as well as in other regions. A coupled physical model for the growth of α_p phase and the growth of the α_{GB} is developed and the following conclusions can be drawn:

- Growth of α_p is more pronounced for low cooling rates and occurs more notable until $\sim 800^\circ\text{C}$.
- Regular (planar) interface shape of α_{GB} is observed decorating the prior β grain boundaries for high cooling rates. For low cooling rates irregular shapes are observed.
- α_{GB} nucleates preferentially from existing α_p phase. For higher cooling rates nuclei in triple points and other regions of the prior β / β grain boundary are observed. The higher nucleation rate for those cooling rates can explain the different behaviour.
- A sharp increase of V supersaturation in the β matrix is observed until $\sim 850^\circ\text{C}$, which is attributed to contribute to the formation of α_{SEC} , especially for cooling rates higher than 10°C/min .

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