Dynamic phase evolution in titanium alloy Ti-6Al-4V

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Industrial processing of the Ti-6Al-4V alloy consists of different steps of heat treatments and hot deformation. The heat treatments are dependent on the kinetic of the allotropic phase transformation, which can be characterized by static and dynamic methods. The methods of post morten samples such as metallography and EPMA combined with Image Analysis were used to quantify the α phase by freezing the microstructure after different heat treatments. The dynamic methods allowed detecting the temperature of phase transformations during heating or cooling used. The aim of this work is to determine the phase transformation evolution of Ti-6Al-4V from defined starting microstructures. Samples heat treated in α+β and β fields with controlled cooling rates and water quenching were characterized by a combination of dilatometry, differential scanning calorimetry (DSC) and metallography. Pre annealing heat treatments in the 930-1030°C range using different cooling rates result in a wide combination of primary α, β, martensite and secondary α phases with different thickness. Continuous heating was carried out by DSC and the peaks observed were correlated with the microstructure obtained from interrupted tests. Transformation of α into β was identified as endothermic peaks, while the transformation of martensite into β and α produced an exothermic peak. Both phenomena provoke a detectable retardation in the rate of the length expansion, denoted by dilatometry experiments. Secondary α laths transformed into β at lower temperatures than primary α, and become thinner and shorter by increasing the temperature. The ageing treatment was determined to be more efficient for samples with large amount of martensite.

1. Introduction

Titanium alloy Ti-6Al-4V is the most widely used, high strength titanium alloy. It finds its main application in the aerospace industry and in medicine due to its high specific strength and corrosion resistance. The control and the optimization of the microstructure of this α/β alloy is one of the most important issues to achieve desired properties [1]. The cooling rate determines the characteristic features of the lamellar microstructure, such as the size of the α lamellae (α plates), the α colony size, and the thickness of α layers at prior β grain boundaries. In general it can be stated that both microstructural parameters, the width of individual α plates and the size of α colonies, decrease with increasing cooling rate [2].

Dilatometry is a technique based on the measurement of the change in length of a specimen as a function of the temperature during isothermal and continuous heating/cooling treatments. Dilatometry requires that the specific volumes of the untransformed and fully transformed states are different. An additional length change effect in the temperature range of the transformations will be due to normal thermal expansion of the untransformed and fully transformed specimens. In the dilatometer the actual signal recorded is proportional to the quantity (specimen length) as a function of time and temperature [3].

Differential Scanning Calorimetry (DSC) measures the change of heat flow rate difference, which is normally released or absorbed due to an alteration of the sample temperature; with respect to a reference. DSC is a technique suitable for investigating phase transformations and reactions which involve heat absorption and/or heat release. Such transitions correlate to endothermic or exothermic peaks in the DSC curve, resprectively [4].

Few works can be found in literature about differential scanning calorimetry of titanium alloy, specially in Ti-6Al-4V. Sha et.al. [1], showed 2 endothermic peaks between begin and end of α/β phase transformation: the lowest temperature peak was interpreted as the phase transformation of secondary α transforming into β phase and the second temperature peak was identified as the phase transformation of primary α into β phase up to the beta transus temperature.

The aim of this work was to determine the phase transformation evolution of Ti-6Al-4V from different starting microstructures during continuous heating by dilatometry and DSC measaurements. Phase transformation was verified by metallography observations of interrupted tests.

2. Experimental

Commercial Ti-6Al-4V alloy containing 6.0Al, 4.3V, 0.2Fe, 0.2Cr, 0.2O, 0.02Cu, 0.05Si, 0.02Ni, 0.007N, 0.003C and balance Ti (all compositions in wt%) was analyzed in this work. The beta transus temperature (T_β) for this material is approximately 1000°C. The as received material was heated at 0.3K/s to temperatures in α+β (930°C and 970°C) and β (1030°C) fields and held during 30 minutes to achieve equilibrium. All the samples were cooled afterwards with different cooling rates: water quenching, air cooling (rate to 600°C ~6K/s and between 600-300°C ~2K/s) and furnace cooling (rate to 600°C

~0.7K/s and between 600-300°C ~0.1K/s) and controlled cooling 0.5K/s. The heat treated samples summarized in table 1 were characterized by means of dilatometry, DSC and metallography.

DSC tests were carried out in Netzsch 404C device under a flow of argon 5.0 of 80ml/min to avoid sample oxidation. The samples were heated up to 1100°C at 0.2K/s and cooled down to room temperature at 0.3K/s. Each sample of 1.2mm thickness and 5.2mm diameter and its corresponding alumina reference were both placed in Al_2O_3 pans.

Dilatometry experiments were performed on a dilatometer BÄHR model DIL 805A/D. The tests were carried out using the same time-temperature programme than for the DSC tests. All experiments were performed under the flow of argon 5.0. The physical coefficient of thermal expansion (CTE) was calculated using the following equation:

$$CTE = dl/ldT \quad (1)$$

Where *l* is the length of the sample at the time t and *dl* / *dT* is the variation of the length with the temperature [3].

Table 1. Heat treatment program of the samples

	Pre heat treatment		
Sample name/ number	Temp. [°C]	Cool. rate [K/s]	α prim. content [%]
930WQ/1	930	Water quenched	27±2
930A/2		Air cooled (to 600°C ~6K/s and 600-300°C ~2K/s)	30±3
930F/3		Furnace cooled (to 600°C ~0.7K/s and 600-300°C ~0.1K/s)	46±3
930_0.5/4		0.5K/s	49±2
970WQ/5	970	Water quenched	21±1
970A/6		Air cooled	22±2
970F/7		Furnace cooled	30±5
1030WQ/8	1030	Water quenched	
1030A/9		Air cooled	
1030F/10		Furnace cooled	

The micrographs were taken by FEGSEM microscope FEI Quanta 200 FEG using the BSE mode to distinguish α (dark) from β (bright) phases. Quantitative analysis of amount and size of globular α and thickness and length of α laths was carried out by means of Axiovision 4.4 software.

The electron probe micro-analysis (EPMA) was used to quantify the elements partition in primary and secondary α phases by freezing the microstructure after heat treating a sample at 930°C for 30 minutes and cooling to room temperature with cooling rate of 0.5K/s. The measurement of Al, V and Ti elements was carried out with a step size of 1µm.

3. Results and discussion

3.1 As received condition

The microstructure of as received material showed primary α grains (dark), secondary α_s laths (dark) and β phase (bright).

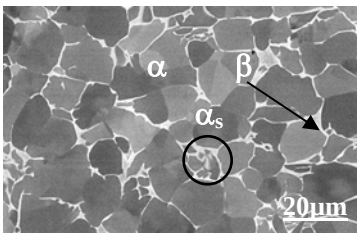


Figure 1. SEM/BSE mode micrograph of Ti in as received condition

3.2. Samples heat treated in alpha/beta field

The microstructures of the heat treated samples in α+β field are shown in Figure 2. The samples were heat treated at 930°C (a,c,e) and 970°C (b,d,f) and water quenched (a,b), cooled in air (c,d), and cooled in furnace (e,f). This microstructure consisted of primary α grains (α_p-dark), α laths between the grains (α-dark) and β matrix in between (light). The content of primary alpha phase increasing with decreasing cooling rate and decreasing temperature as shows Table 1.

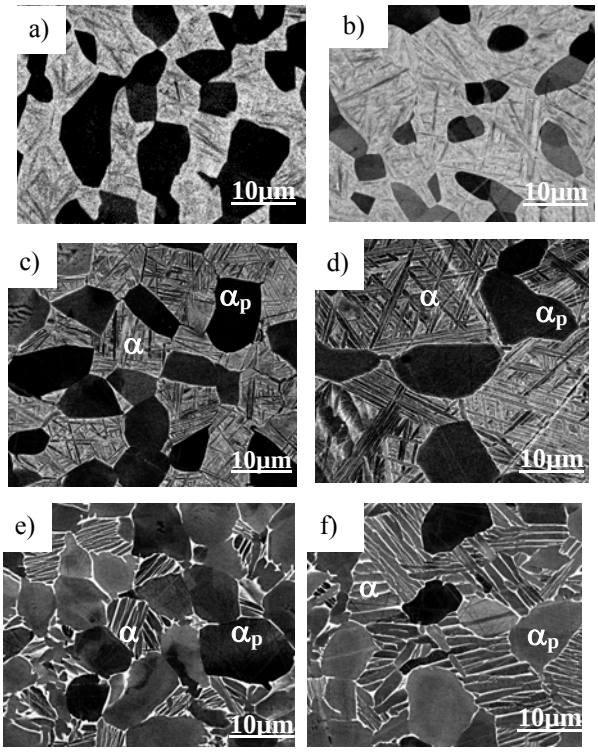


Figure 2. FEGSEM BSE mode micrographs of samples heat treated at 930°C (a,c,e) and 970°C (b,d,f) and water quenched (a,b), cooled in the air (c,d) and cooled in furnace (e,f)

Figure 4 shows CTE curve of 1st heating cycle of heat treated samples. The dilatometry curves showed two detectable retardation in the rate of the length expansion in the temperature ranges of the secondary α into β and

primary α into β transformations in 750-930°C (a) and 930-1050°C (b) temperature ranges, respectively. Martensite into α and β also produced a retardation at around 450°C for the WQ sample (c). In both cases, α and martensite were less dense than β phase.

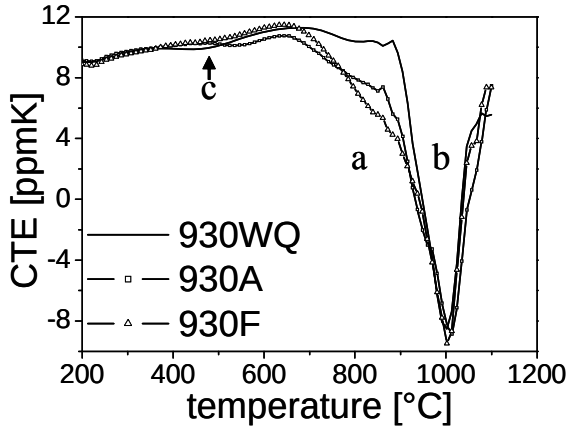


Figure 4. CTE curve of 1st heating cycle of samples exposed at 930°C

Figures 5 and 6 show DSC measurements of samples 1-7. In all cases 2 endothermal peaks (a,b) were observed at ~700°C and 970-980°C. The high temperature peak was accompanied by a small shoulder (d) appearing only for samples heat treated at 930°C and 970°C. The water quenched samples showed additionally an exothermic peak (c) at ~500°C. Transformation of α into β was identified as endothermic peak, while the transformation of martensite into β and α produced an exothermic peak. The transformation temperatures were slightly shifted to lower temperatures compared to dilatometry results due to a gradient of temperature in the dilatometer samples

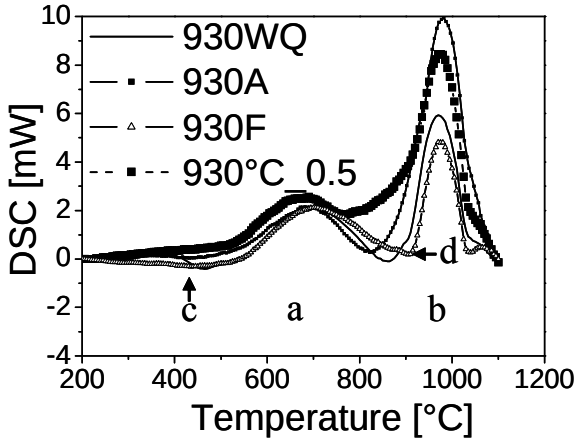


Figure 5. DSC curves of 1st heating cycle of samples exposed at 930°C (exo down)

During the continuous heating α laths dissolved at the lowest temperature. This transformation was completed at 930 or 970°C corresponding to the pre-treatment of the sample.

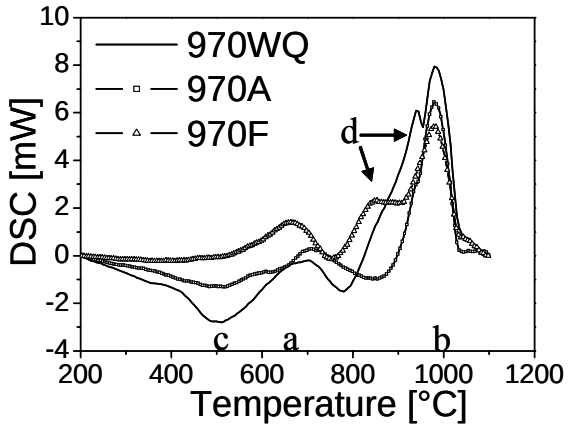


Figure 6. DSC curves of 1st heating cycle of samples exposed at 970°C (exo down)

Figure 7 shows FEGSEM images of samples heat treated at 930°C for 30min, cooled in air, reheated at 0.2K/s to different temperatures and water quenched to freeze the microstructure. The dissolution of the different phases during continuous heating was simulated. It was shown that small α laths were transforming into β phase, the transformation of them was completed at ~930°C.

The analysis of α laths showed that during heating the laths became shorter and thinner (Figure 8). The small laths produced during slow cooling from β field transformed into β phase before the large ones due to different V content. The analysis of α laths shown in Figure 8a) showed that the mean size of the laths was ~2.5μm and the average length ~6.1μm. The mean size of laths shown in Figure 8b) was ~1,3μm and their average length was ~5.1μm.

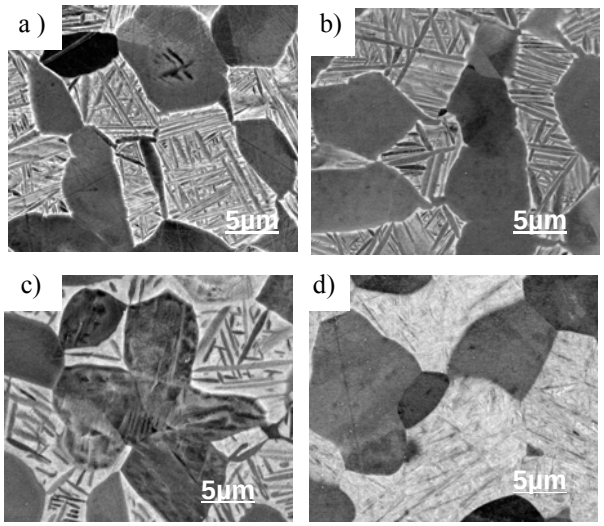


Figure 7. FEGSEM images of sample 2 (a) heat treated at 930°C for 30min, cooled in air and reheated at 0.2K/s to b)740°C, c)850°C and d) 930°C

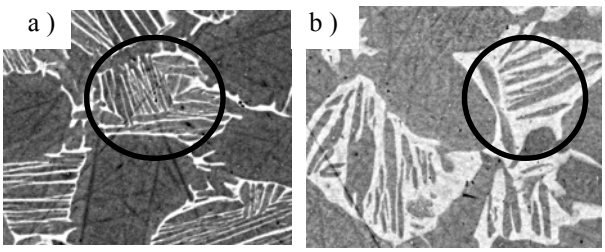


Figure 8. FEGSEM images of sample 3 -pre heat treated at 930°C, cooled in furnace- and heated to a)655°C and b)820°C

EPMA measurements of sample 4 was done to measure the vanadium and aluminium content in primary and secondary α and in β phase. Secondary α laths showed less Al and more V compared to primary α, although it should be pointed out, that the spatial resolution of this analysis was poor (Figure 9).

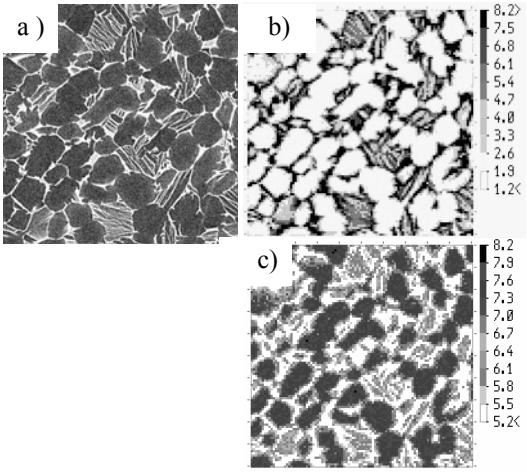
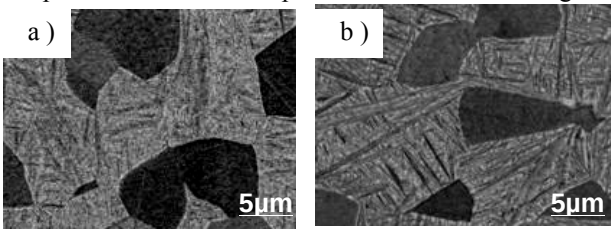


Figure 9. a) SEM micrograph of sample 4, b) concentration map of Al (wt.%), and of c) V (wt%)

Figure 10 shows FEGSEM images of sample 1 reheated at 10K/min to different temperatures and water quenched to freeze the microstructure. The transformation of martensite into alpha+beta was observed. At higher temperatures the small alpha laths are transforming into



beta phase.

Figure 10. FEGSEM images of sample 1 heated to a)670°C and b)790°C

3.2. Samples heat treated in beta field

The samples heat treated at 1030°C and cooled in furnace and air showed thinner α laths in the case of faster cooling, while furnace cooling (rate of 0.7K/s between 1030°C-600°C) showed thicker laths.

The DSC curves of samples treated at 1030°C after different cooling rates showed the two peaks but no small shoulder before the second peak (Figure 11). Due to the relatively fast cooling, phases with different sizes transformed at different temperatures. The interrupted tests of samples pre heat treated in β field showed dissolution of small laths during heating (Figure 12).

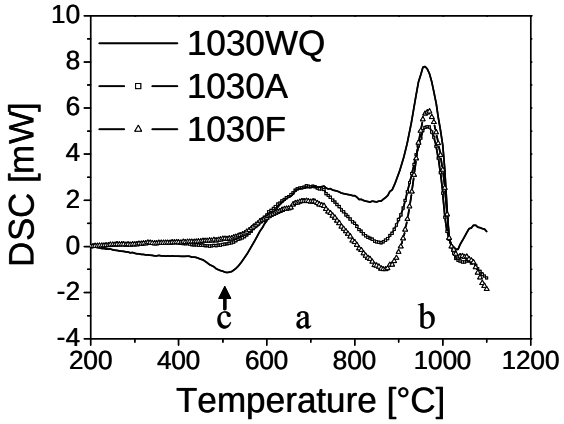


Figure 11. DSC curves of 1st heating cycle of samples exposed at 1030°C (exo down)

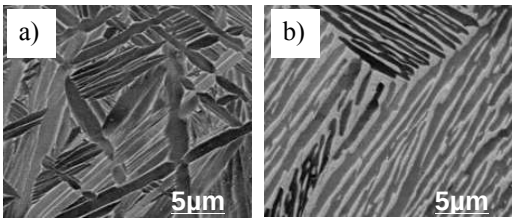


Figure 12. FEGSEM images of sample 10 heated to a)655°C and b)875°C

4. Conclusions

Phase transformation of α into β was identified as endothermic peak, while the transformation of martensite into β and α produced an exothermic peak. Both phenomena provoked a detectable retardation in the rate of the length expansion, denoted by dilatometry experiments. Secondary α laths transformed into β at lower temperatures than primary α, and became thinner and shorter by increasing the temperature. Different vanadium and aluminium contents between primary α and α laths and martensite needles were seen.

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REFERENCES

1) Sha,W., Malinov,S. *Titanium alloys:modelling of microstructure, properties and applications* (Woodhead Publishing limited,UK, 2009)
2) Williams, C. J., Lütjering, G. Titanium, (Springer, Germany, 2003)
3) Mittemeijer,E. Funadamentals of materials science, (Berlin, 2010)
4) Höhne H. W. G., Hemminger F. W., Flammersheim J. H., *Differential Scanning Calorimetry*,(Springer, Berlin, Germany, 2003)