Study of transformations and microstructural modifications in Ti-LCB and Ti-555 alloys using Differential Scanning Calorimetry

Marc Carton[1], Pascal Jacques[2], Nicolas Clément[2], Jacqueline Lecomte-Beckers[1]

[1] *Aerospace and Mechanical Engineering Department, University of Liège, Liège 4000, Belgium*
[2] *IMAP Department, Université Catholique de Louvain, Louvain-la-Neuve 1348, Belgium*

The aerospace industry is the single largest market for titanium products primarily due to the exceptional strength to weight ratio, elevated temperature performance and corrosion resistance. Titanium applications are most significant in jet engine and airframe components that are subject to high temperatures. The studied Ti-LCB and Ti-555 titanium alloys must present good properties to fatigue damage as they are located in a low-pressure compressor. In order to avoid this damage, an optimized alloy microstructure is required. In our case, the optimal microstructure would be a beta matrix in which small alpha particles are uniformly distributed, obtained after a combination of thermomechanical and thermal treatments. The understanding of the mechanisms ruling the microstructure evolution is achieved through the study of phase transformations versus temperature. During heating, the ω_{ath} phase is transformed into the ω_{iso} phase, allowing itself the appearance of the α phase. Then this α phase is transformed progressively into the β phase, and the structure becomes totally β when the transus temperature is reached. Therefore, a thermal analysis tool such as DSC (Differential Scanning Calorimetry) was used in order to characterise the phase transformations of the material during imposed controlled temperature programs.

Keyword: *titanium (Ti), Differential Scanning Calorimetry, Ti-555, Ti-LCB, SEM, phase transformations*

1. Introduction

Titanium alloys are more and more used in aerospace applications due to their good mechanical properties, good formability, corrosion resistance and low density. The microstructure plays an essential role in the good behaviour of these alloys during applications, and therefore a succession of several heat and mechanical treatments must be carried out to obtain the appropriate structure, which delay fatigue crack initiation[1,2]. For both β metastable titanium alloys Ti-LCB and Ti-555 used in aerospace industries, the final microstructure (named bimodal) consists of a β matrix with small α particles uniformly distributed, as shown in Figure 1. The chemical composition of both alloys is presented in Table 1.

Table 1. Chemical composition of Ti-LCB and Ti-555 alloys.

Alloys	Al	Mo	V	Cr	Fe
Ti-LCB	1.5	6.8	-	-	4.5
Ti-555	5	5	5	3	0.3

The good understanding of the alloys behaviour during applications is closely related to the knowledge of phase transformations and the effects of temperature on the microstructure. That is why titanium alloys Ti-LCB and Ti-555 transformations were investigated by Differential Scanning Calorimetry (DSC). For these investigations, two different states were considered (bimodal and as-quenched). The bimodal structure is the conventional structure delivered by the producer. The as-quenched state was obtained by reaching a temperature above the β transus, followed by an isothermal step (5 min at 810°C for Ti-LCB and 30 min at 865°C for Ti-555) and a water-quenching in order to obtain a structure exclusively β. Typical microstructures of Ti-LCB and Ti-555 both bimodal and as-quenched are presented in Figure 1 and Figure 2. Nevertheless, the appearance of very small particles named ω_{ath} and having a size about 20 Å can appear in titanium β metastable alloys during water quenching[3]. This nanoparticle ω_{ath} phase has the same composition as β and could be present in the β quenched structure[4].

Figure 1

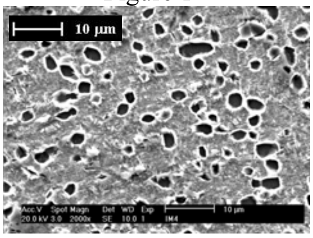

Figure 2

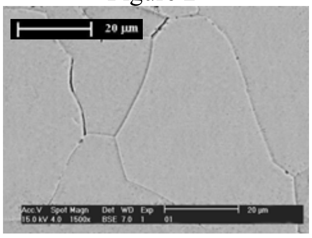

Figure 1. Typical microstructure of Ti-LCB/Ti-555 bimodal.
Figure 2. Typical microstructure of Ti-LCB/Ti-555 as-quenched.

2. DSC method

DSC is a technique in which the difference in energy input into a substance and a reference material is measured as a function of temperature, while the substance and reference material are subjected to a controlled temperature program. The basic principle underlying this technique is that, when the sample undergoes a physical transformation such as phase transformation, more (or less) heat will need to flow to it than the reference to maintain both at the same temperature.

3. Results

Ti-LCB and Ti-555 alloys samples (bimodal and as-quenched states) were heated from room temperature to a temperature above the β transus (900°C for Ti-LCB and 950°C for Ti-555) with a constant rate and were immediately cooled down with the same rate. Four heating/cooling rates (2K/min, 5K/min, 10K/min and 15K/min) were used to determine the kinetic effects on the alloys microstructure.

3.1 Ti-LCB

DSC measurements were carried out on Ti-LCB bimodal and as-quenched.

3.1.1 DSC of Ti-LCB during heating

3.1.1.1 Ti-LCB bimodal

The DSC curves of the Ti-LCB bimodal during heating are presented in Figure 3.

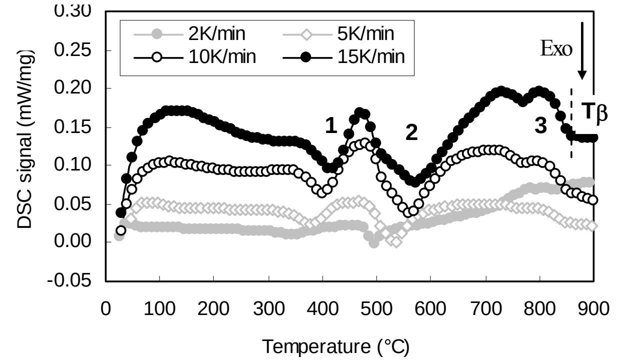

Figure 3. DSC curves of Ti-LCB bimodal during heating.

The influence of the heating rate on peaks position is clearly observed. The higher the heating rate, the larger the peaks and the higher the transformation temperatures. From room temperature to 300°C, no transformation is observed whereas three peaks are obtained from 300°C to about 830°C. The endothermic peak (named 3), positioned at about 800°C, corresponds to the α→β transformation. The end of this transformation Tβ is shown in Figure 3 for a heating rate of 15K/min. As each transformation temperature depends on kinetic effects, the β transus transformation is shifted toward higher temperatures with an increasing heating rate, as the end of the transformation takes place respectively at 832°C, 846°C, 855°C, 863°C with a heating rate of 2K/min, 5K/min, 10K/min and 15K/min. The theoretical β transus temperature is of 810°C. In order to interpret the exothermic peaks (named 1 and 2) obtained at about 400°C and 550°C, more investigations were carried out. Ti-LCB samples were heated (5K/min) from room temperature and respectively quenched at 300°C, 420°C and 700°C. The obtained microstructures are shown in Figure 4 and Figure 5.

Figure 4

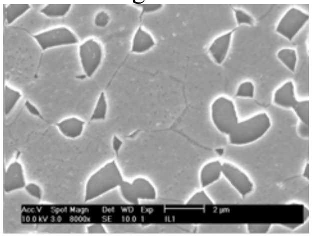

Figure 5

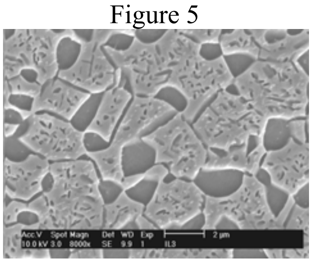

Figure 4. Microstructure of Ti-LCB bimodal after quenching at 300°C and 420°C.

Figure 5. Microstructure of Ti-LCB bimodal after quenching at 700°C.

A similar microstructure was obtained at 300°C and 420°C. This means that the peak 1 characterises a modification of very small particles not observable by SEM and then could correspond to the formation of ω_{iso} or nanoscale α. The ω_{iso} phase precipitates generally during heat treatments between 200°C and 500°C. However, its growing mechanism is not well known actually but can generate a very thin α phase interesting regarding mechanical properties. This nanoscale phase precipitation generates a pronounced increase in hardness, as values of 346HV0.5 and 409HV0.5 are respectively obtained at 300°C and 420°C. Figure 5 was obtained after quenching at 700°C. It shows the appearance of α particles within β grains. Therefore the peak 2 would correspond to the precipitation of α in β grains. But as the signal shows an inflexion point, meaning that this large peak could be the overlap of two different transformations, a deconvolution was carried out between 500°C and 750°C. As an example, Figure 6 shows the result of the deconvolution with a heating rate of 15K/min. The two peaks resulting are named peak 2' and peak 2''.

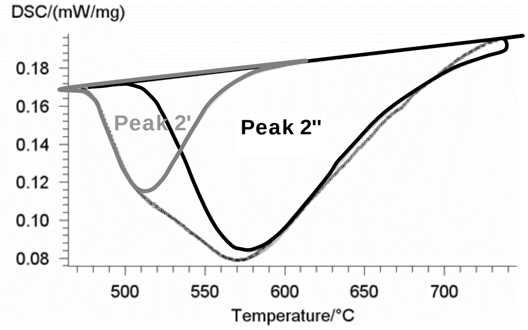

Figure 6. Peak deconvolution of Ti-LCB bimodal transformation between 450°C and 700°C (15K/min).

This deconvolution clearly indicates the presence of two peaks: one could correspond to the appearance of α from ω_{iso} (2') and the second to the precipitation of α from β (2'').

3.1.1.2 Ti-LCB as-quenched

A DSC study was also carried out with Ti-LCB alloy starting from an as-quenched state. The signal obtained for each heating rate is presented in Figure 7.

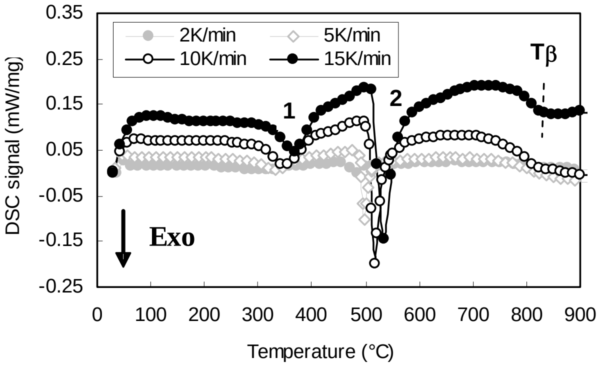

Figure 7. DSC curves of Ti-LCB as-quenched during heating.

As for Ti-LCB bimodal, the higher the heating rate, the higher the transformation temperatures. Another similar observation is the appearance of an exothermic peak (named 1) at 350°C, corresponding to a nanoscale precipitation (ω_{iso} or α). As for Ti-LCB Bimodal, this transformation generates a change in hardness, as Vickers harness tests show an increase from 365 HV0.5 to 439

HV0.5 between 300°C and 420°C. The exothermic peak (named peak 2) obtained at about 550°C shows the appearance of α. Finally, α is transformed into β (endothermic peak between 600°C and at Tβ) and the structure is β monophased at Tβ.

3.1.2 DSC of Ti-LCB during cooling

Immediately after reaching 900°C, Ti-LCB bimodal and as-quenched samples were cooled down and the resulting DSC signals are shown in Figure 8. Similar results were obtained for both alloys.

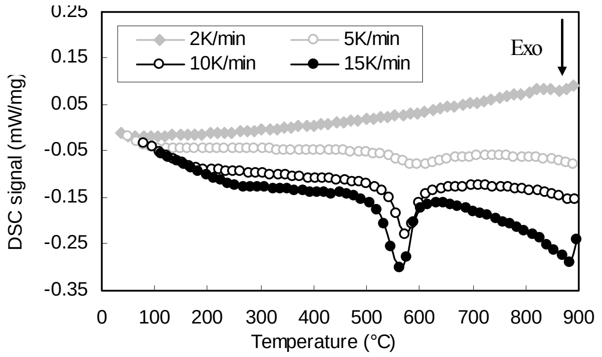

Figure 8. DSC curves of Ti-LCB bimodal and as-quenched during cooling.

A transformation at about 550°C is observed, corresponding to the α formation. Moreover, the lower the cooling rate, the flatter the transformation peak. This means that the quantity of α phase formed depends strongly on cooling conditions. Figures 9 to 12 show the α appearance in different forms whereas only one peak is obtained. This means that either some transformations do not require a lot of energy and therefore do not generate any peaks, or they appear at the same time and then contribute to the same peak. The cooling from temperature above the β transus generate the appearance of α located at grain boundaries (αGB), perpendicular to grain boundaries (αWGB) and within β grains (αM).

Figure 9

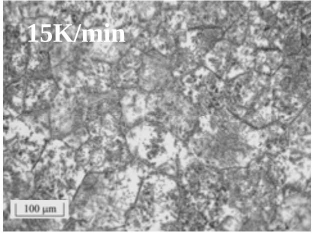

Figure 10

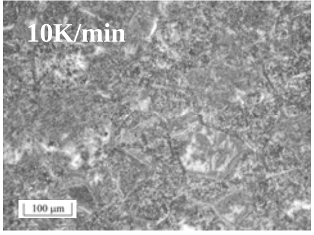

Figure 11

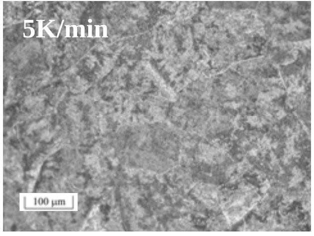

Figure 12

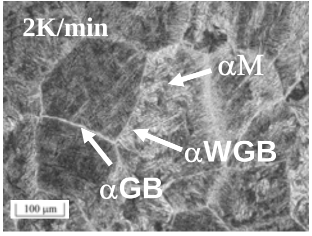

Figure 9. Typical microstructure of Ti-LCB after cooling (15K/min).
Figure 10. Typical microstructure of Ti-LCB after cooling (10K/min).
Figure 11. Typical microstructure of Ti-LCB after cooling (5K/min).
Figure 12. Typical microstructure of Ti-LCB after cooling (2K/min).

Low cooling rates favour the appearance of α in the form of needles. Moreover, the lower the cooling rate, the larger the quantity of α formed.

3.2 Ti-555

As for Ti-LCB, DSC measurements were carried out on Ti-555 bimodal and as-quenched.

3.2.1 DSC of Ti-555 bimodal during heating

3.2.1.1 Ti-555 bimodal

The results of DSC measurements on Ti-555 bimodal during heating are shown in Figure 13.

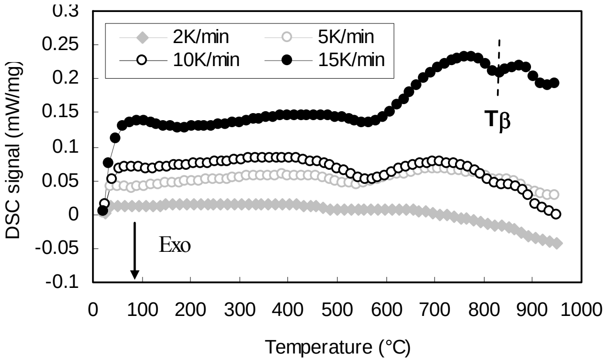

Figure 13. DSC curves of Ti-555 bimodal during heating.

The β transus transformation appears higher than for Ti-LCB (about 880°C). Comparing to Ti-LCB, no peak is obtained at 350°C and the only transformation observed takes place at about 550°C. One hypothesis of this phenomenon would be the absence of ω and α nanoparticles. In order to better characterise this weak peak, quenching tests were carried out at 300°C and 700°C after heating (5K/min).

Figure 14

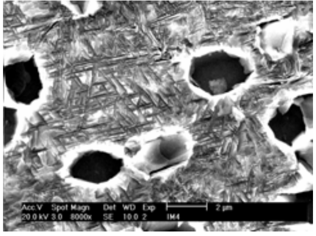

Figure 15

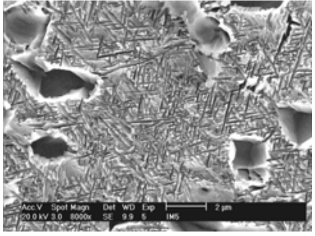

Figure 14. Microstructure of Ti-555 bimodal after quenching at 300°C.
Figure 15. Microstructure of Ti-555 bimodal after quenching at 700°C.

The structure before heating (Figure 1) as well as those obtained after heating at 300°C and 700°C seem to be similar, with the presence of α particles and α needles in a β matrix. Therefore the peak observed in DSC could correspond to the appearance of α from β within the matrix and between the needles initially existing.

3.2.1.2 Ti 555 as-quenched

The results of the tests carried out on Ti-555 as-quenched alloy are presented in Figure 16.

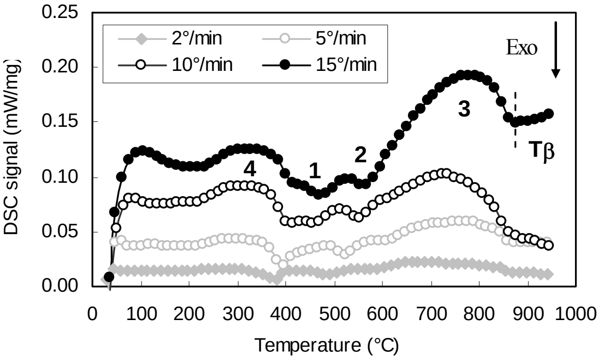

Figure 16. DSC curves of Ti-555 as-quenched during heating.

Starting from a β monophased microstructure at room temperature (Figure 2), the thermal treatment occurring during DSC measurements shows that the β microstructure is transformed between 200°C and 600°C (peaks 4, 1 and 2) and that the progressive transformation to the high temperature monophased β microstructure is marked (peak 3). A second endothermal peak is obtained around 300°C (peak 4), which could correspond to a dissolution as only exothermal transformations were obtained in this temperature range for the previous states. Quenching tests were carried out after heating from room temperature at different temperatures. Analysis carried out by SEM showed a β monophased microstructure for each quenched samples until 425°C, although an increase of hardness is observed (298HV0.5 at 200°C and 342HV0.5 at 425°C). This would mean that a fine ω or α precipitation not revealed by SEM could take place, ω acting as a precursor of the α precipitation. Therefore more investigations are necessary to understand the three exothermal transformations obtained at 400°C, 450°C and 550°C, keeping in mind that the α appearance takes place before about 580°C, as the endothermal peak 3 is observed between 600°C and Tβ (correspond to the continuous transformation from α to β).

3.2.2 DSC of Ti-555 during cooling

The evolution of the DSC signal obtained for Ti-555 alloys during cooling (figure 17) is similar for both bimodal and as-quenched states. As for Ti-LCB, a single peak is observed at about 550°C.

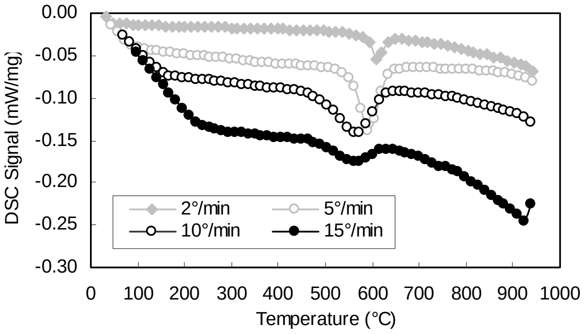

Figure 17. DSC curves of Ti-LCB 555 alloy during cooling.

As for Ti-LCB, this peak corresponds to the formation of α from β. Even if one single peak is observed, the influence of the cooling rate is clearly pointed out through the differences of structure after cooling, as shown in Figures 18 to 21. The lower the cooling rate, the larger the quantity of α formed. Moreover, α appears exclusively in a form of needles.

Figure 18

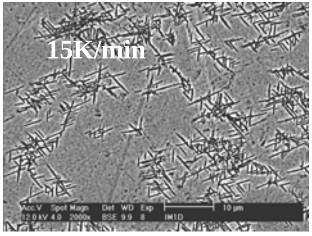

Figure 19

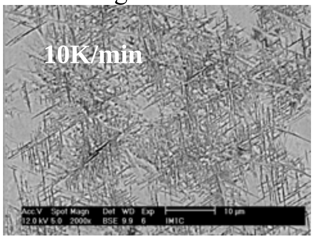

Figure 20

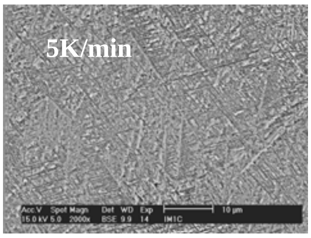

Figure 21

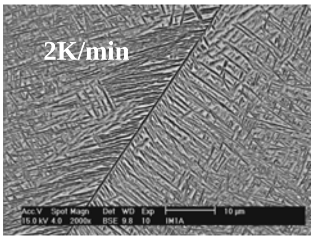

Figure 18. Typical microstructure of Ti-555 after cooling (15K/min).
Figure 19. Typical microstructure of Ti-555 after cooling (10K/min).
Figure 20. Typical microstructure of Ti-555 after cooling (5K/min).
Figure 21. Typical microstructure of Ti-555 after cooling (2K/min).

Conclusion

Ti-LCB and Ti-555 alloys were investigated by DSC and several phase transformations were characterised, with the presence of micro sized particles ($\omega_{ath,}$ ω_{iso} and α) as well as the formation/transformation of α and β. The tests showed the influence of the kinetic on the microstructure as well as the diversity of α shape appearing during temperature cycle. However, many questions remain unanswered and need more investigations, especially concerning the ω phases appearance and transformations.

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