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Decomposition Staging of β-metastable Solid Solution in $(\alpha + \beta)$ Titanium Alloy During Heating

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Abstract

The decomposition of β-metastable phase in (α+β)-titanium alloys during continuous heating needs a detailed investigation in the range of relatively small temperatures. The staging of β-solid solution decay was studied on the solution treated and water quenched VT16 (Ti-3Al-5Mo-5V) used for fasteners production. The thermal exo-effects of β-phase decomposition were examined using DSK. Corresponding effects in the in situ X-Ray diffraction patterns and storage modulus temperature dependence in the DMA curves are discussed. A three stage layout of β-decay is reported during continuous heating of VT16 with (α+β)-structure after quenching.

Keywords: metastable phase, phase transformation, in situ XRD, storage modulus, decay, quenching, continuous heating, dynamic mechanical analysis

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1. Introduction

VT16 is a thermally hardened (α+β)-titanium alloy with the Ti-3Al-5Mo-5V (wt.%) doping system, which is utilized mainly for the production of fasteners [1, 2]. Solution treatment followed by a water quenching may result in the formation of metastable β_m and/or α"-structural components depending on the heating temperature [3]. These phases can undergo decomposition in several stages upon subsequent heating [4, 5]. The decomposition of α"-martensite was studied fully in the VT16 alloy, since the solution treatment and quenching of this alloy are usually performed above the critical temperature, which lies within 750-800 °C. The metastable β-solid solution undergoes martensitic β→α"-transformation during cooling. However, the regularities of decomposition of the metastable β-solid solution are also important, because β-phase is partially preserved in the structure after quenching [3]. In addition, it is of interest how the decay of the β-solid solution affects the characteristics used in the thermal analysis of phase transformations.

Based on this, the staging of the decomposition of the metastable β-solid solution are studied in the VT16 during continuous heating using methods of XRD, DSC and DMA.

2. Material and Methods

The stages of metastable β-phase transformation for the VT16 alloy solution treated at 725°C and water quenched were studied using differential scanning calorimetry (DSK) on Netzsch Jupiter STA 449C with a heating rate of 20 °/min.

The variable-temperature X-ray diffraction (XRD) analysis was carried out in Cu Kα radiation at an accelerating voltage of 40 kV and a current of 40 mA, with a 2θ step of 0.02 °, the dwell time of 0.5 seconds using the Bruker D8 Advance equipped with an Anton Paar HTK 1200N high-temperature attachment and the LynxEye linear detector in a temperature range of 30-550° C with a step of 50 °.

The 3-point bending of 0,5x4x22 mm samples was employed using dynamic mechanical analysis (DMA) on Netzsch DMA 242C with a dynamic load of 5N and loading frequency of 1 Hz.

Scanning electron microscopy was performed using a Philips SEM 535 scanning electron microscope at an accelerating voltage of 25 kV.

3. Results and Discussion

SEM and XRD data (Fig. 1, 2) demonstrate that the alloy has the (α+β)-structure after quenching. The particles of the primary α-phase have predominantly globular forms which are regularly distributed in β-grains. The following phase lattice parameters were calculated: a_α = 0.4685 nm, c_α = 0.2942 nm, c/a_α = 1.592, a_β = 0.3249 nm.

The percentage ratio α/β of the volume fractions of α and β phases is 55/45. The estimated parameter $(c/a)_\alpha$ is higher than 1.587 of the pure titanium according to [6], which is typical of titanium alloys doped with aluminum.

The data of in situ XRD characterize the change in phase composition of the quenched VT16 alloy during continuous heating. DSC curves show corresponding thermal effects. The peaks of storage modulus accompany these effects in the DMA curves. Further, we analyze these data simultaneously.

An exo-effect of low intensity occurs in the DSK curve in the temperature range of 150...340 °C (Fig. 3). For almost the same temperature range of 250...330 °C two small peaks are observed in the temperature dependence of the storage modulus (Fig. 4).

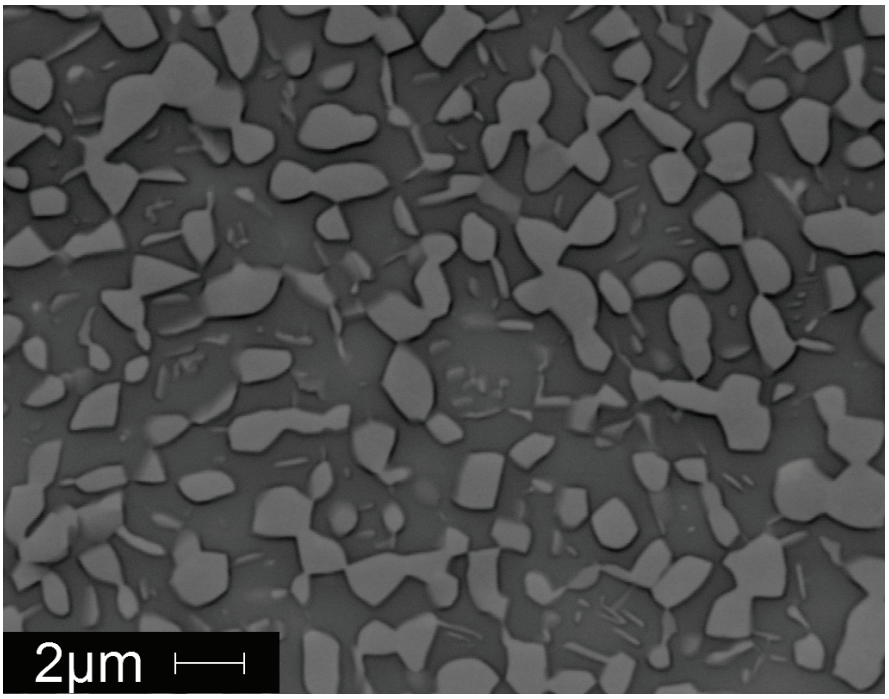

Figure 1: Microstructure of the VT16 (Ti-3Al-5Mo-5V) solution treated at 725° and water quenched.

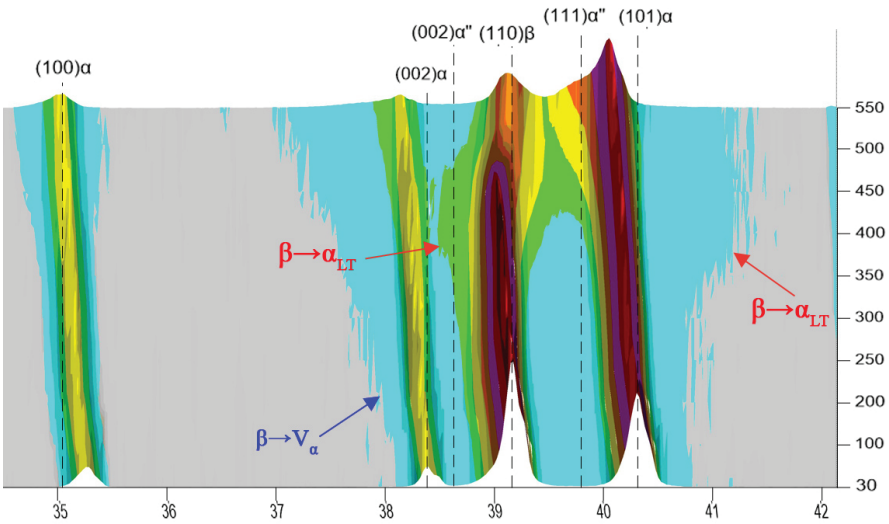

Figure 2: In situ X-Ray diffraction patterns of the VT16 solution treated at 725° and water quenched.

The first extreme corresponds to a slowdown in the decrease of the storage modulus change. It is not possible to isolate the peaks in the in situ X-Ray diffraction patterns of phases, which formed in the temperature range of 150...325 °C. However, some “spread” of the α-phase peaks is observed and the asymmetry of the $(110)_{\beta}$ peak increase towards the $(002)_{\alpha}$ (Fig. 2). The parameters of the α and β phases in the indicated range increase almost linearly with the temperature (Fig. 5), which is typical of the thermal expansion of phases that do not undergo phase transformations. Thus, the effects observed in the DSC, DMA and in situ X-ray diffraction patterns can be accounted for the development

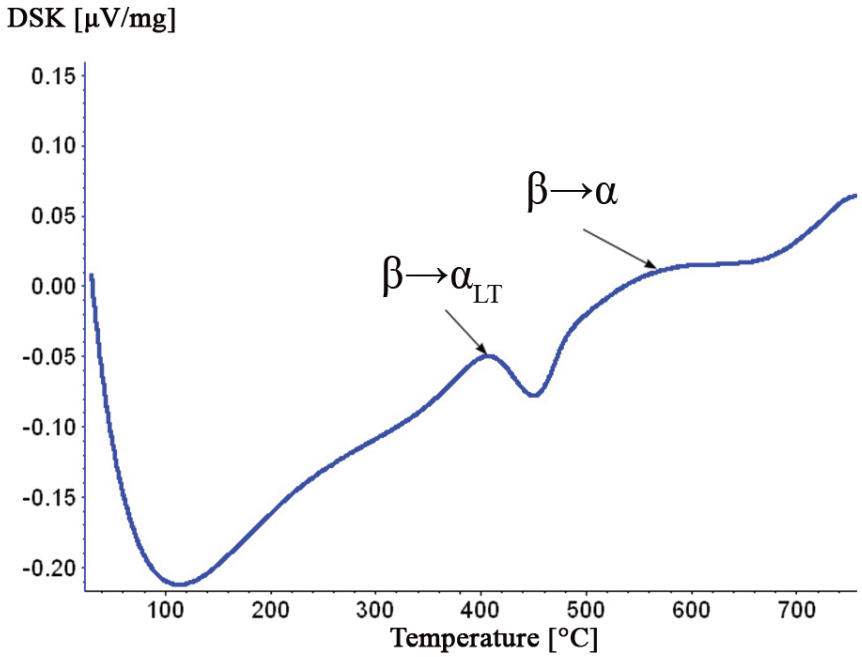

Figure 3: DSC curve of the solution treated and quenched VT16.

of the subtransient phenomena [7], namely the formation of α-shaped displacements in the metastable β-solid solution, referred to as V_α in [8]. Firstly, an exo-effect was observed in [9] in the DSK curves for the same temperature range, which characterized the formation of α-shaped displacements in the metastable solid solution. Secondly, study [10] reports that in titanium alloys, the α-phase has a higher elastic modulus than the β-solid solution. In this regard, the formation of α-shaped displacements in the β-solid solution contributes to a higher elastic modulus compared to the β-solid solution without such displacements. This slows down the decrease in the elastic modulus during heating, noted above (Fig. 4). Thirdly, the formation of α-shaped displacements in the β-solid solution also spreads the peaks of α-phase and results in the asymmetry of the β-phase peaks in X-Ray diffraction patterns (Fig. 2).

The second exo-effect corresponds to the temperature range of 340-450 °C in the DSK curve (Fig. 3). The following two kinks can be revealed in the storage modulus temperature dependence, which correspond to the same temperature range (Fig. 4). The first indicates a slowdown in the decrease in the elastic modulus with increasing temperature. An addition, the $(200)_{\alpha''}$ peak is detected in the X-ray diffraction pattern starting from heating temperatures above 350 °C and up to 475 °C. Moreover, the background amplifies near the other α′′-phase peaks with a rhombic lattice (Fig. 2). These facts allow us to conclude that the β-metastable solid solution decomposes with the formation of the so-called low-temperature α_{LT} phase [11] in the temperature range of 330 (350) - 430

(475) ˚C. The crystal lattice of α_{LT} is characterized by the rhombic distortions in relation to equilibrium α-phase with hcp lattice.

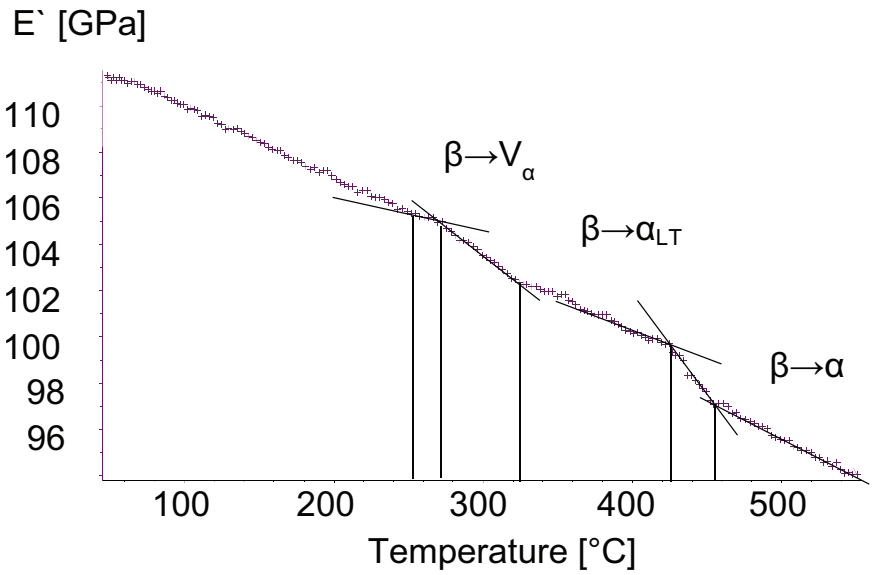

Figure 4: The storage modulus temperature dependence of the solution treated VT16.

A third exo-effect extends in the DSK curve up to a temperature of 680 ˚C (Fig. 3). This is accompanied by a smaller slope of the storage modulus temperature dependence in the DMA curve starting with a temperature of 450 ˚C. Such modulus behavior persists up to the maximum measurement temperature of 550 ˚C (Fig. 4). A decrease in the peak intensity of $(110)_{\beta}$ and an increase in the intensity of the α-phase peaks can be observed in the in situ X-ray diffraction pattern at heating temperatures above 400 ˚ C (Fig. 2). The lattice parameter of the α-phase decrease up to a maximum investigated temperature of 550 ˚C in contrast to the linear growth accounted for thermal expansion observed at heating temperatures below 400 ˚C (Fig. 5c). Therefore, the decomposition of the metastable β-solid solution occurs by the diffusion mechanism with formation of the α-phase. As a result, the β-solid solution is enriched with the β-stabilizing elements Mo, V, which have smaller atomic radii than titanium [8]. This leads to a decrease in the β-phase lattice spacing, which compensate for its thermal expansion. The effect typical of this transformation [12] appears in the DSC curve due to the precipitation of the α-phase from the β-solid solution during decomposition (Fig. 3). A smaller slope of the elastic modulus in the DMA curve typical for this temperature range results from the increase in the volume fraction of α-phase having the higher Young‘s modulus compared with the matrix β-solid solution.

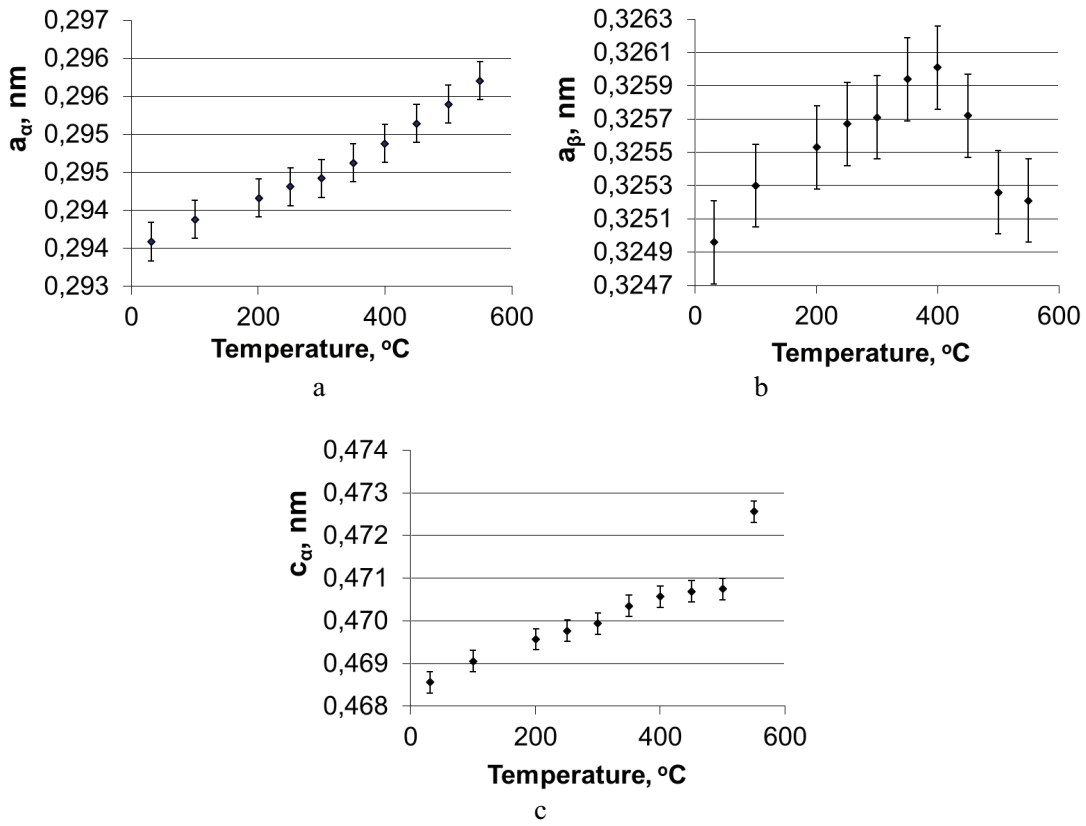

Figure 5: Heating-induced variation of the unit cell parameters of α and β-phases of the solution treated VT16.

Therefore, the decomposition staging of the metastable β-solid solution fixed by quenching from a solution treatment temperature of 725 °C in the Ti-3Al-5Mo-5V alloy during continuous heating can be represented by the following layout: β→Vα → α_{LT}→ α.

A similar scheme was proposed in [12, 13] for the decomposition of metastable β-solid solution during continuous heating of Ti alloys. It is also similar to the classical decomposition scheme of metastable phases in non-ferrous metals [14]. This scheme includes the initial stage, where the zones of a metastable solid solution are formed. We attribute the regions with α-shaped displacements - V_α to such zones in this study. Note, that these metastable zones do not form their own crystal lattice. On the next stage an intermediate or several intermediate phases form with a certain crystal lattice, which differs from the stand out equilibrium phase (e.g. the low-temperature α_{LT} phase with a rhombic lattice in this study). An equilibrium phase with a stable crystal lattice forms in the final stage of decay. In this study, it refers to the α-phase with hcp lattice.

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