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Composite structure of α phase in metastable β Ti alloys induced by lattice strain during β to α phase transformation



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ABSTRACT

The β to α phase transformation of Ti alloys progresses in a displacive-diffusive mixed-mode. The associated transformation strain has important influence on the resultant microstructure. In this work, the microstructural features of α precipitates in a metastable β Ti alloy, Ti-7333, were thoroughly investigated. Special attention was paid to the intragranular α for the advantage of a stress-free transformation environment. Results show that the constituents of each α precipitate is not single. Two kinds of nano-sized α domains exist. One is situated on the broad face of the major α precipitate (named interface α), and the other goes through the major α (termed penetrating α). The interface α is related with the β matrix by the Burgers orientation relationship (BOR) and with the major α by a $60^\circ/\langle 11\bar{2}0 \rangle_\alpha$ rotation. The nucleation of such α particles is induced by the largest shear strain generated by the formation of the major α . They act as stress-associated sympathetic nuclei of the neighboring α precipitates and eventually contribute to the formation of the triangular α cluster. The penetrating α does not obey the BOR with the β matrix but is related with the major α by a 60° rotation around another $\langle 11\bar{2}0 \rangle_\alpha$ axis. The nucleation of such α is induced by the largest normal strain generated by the formation of the main α plate. This work provides comprehensive information on the displacive characters of the β to α transformation and their impact on the microstructure of metastable β Ti alloys.

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1. Introduction

The Al containing metastable β titanium alloys are considered as attractive materials for aeronautical applications due to their high yield strength, excellent fatigue crack growth resistance and good hardenability [1,2]. Their mechanical properties are strongly dependent on the microstructural characteristics, especially the volume fraction, the morphology and the distribution of α precipitates [3–6], therefore the β to α phase transformation has been a topic of intensive studies.

For the Al containing metastable β titanium alloys, the high temperature β phase with body-centered cubic (BCC) structure

transforms to the lower-temperature equilibrium α phase with hexagonal close-packed (HCP) structure when the alloy is slowly cooled across the β transus temperature (T_β), or isothermally held at a temperature below T_β . Generally, the β to α phase transformation involves two processes: diffusion of alloying elements and structure transformation of Ti atoms. The former results in a composition change of the product α phase by depleting β stabilizing elements, such as Mo, Cr, Nb, and enriching α stabilizing elements, such as Al, whereas the latter gives rise to the structure change from BCC to HCP [7–9]. Due to the displacive nature of the β to α transformation, the transformation demonstrates specific crystallographic features, like the case of martensitic transformation. The transformation usually follows a certain orientation relationship (in most cases the Burgers orientation relationship (BOR), i.e., $\{110\}_\beta/\{0001\}_\alpha$, $\langle \bar{1}1\bar{1} \rangle_\beta/\langle 11\bar{2}0 \rangle_\alpha$) with specific transformation habit plane [10,11]. The α phase is usually in plate shape. A maximum of 12 crystallographic orientation variants can be produced due to the cubic symmetry of the parent phase and the

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BOR between the parent and the product phases. The transformation strain can be self-accommodated by locally forming specific variants, giving rise to the appearance of characteristic microstructure patterns depending on the internal stress and the external stress conditions [12–19].

Recently, the displacive process of the β to α transformation has been experimentally investigated in a Ti-5553 alloy [20]. It evidenced that at the very beginning of the transformation α plates precipitate via a displacive process with a composition very close to that of the β matrix. Then they grow through a coupled displacive-diffusional process with the partitioning of the alloying elements being transformation rate controlling [20]. The strain characters of the transformation and strain interaction between different variants have been analyzed theoretically [21–24]. It turns out that the transformation strain has strong influence on the formation of secondary α particles and on local variant selection in relation to the need of minimizing the overall transformation strain energy.

Although the studies on the displacive characters of the β to α transformation have greatly advanced our knowledge on the formation of specific microstructural features of the α phase in Ti alloys, investigations on the constituents of individual α precipitates and their correlation with the transformation strain are still limited. In view of such a situation, we conducted a thorough crystallographic study on α precipitates, especially their sub-structures, in a metastable β Ti alloy (Ti-7333) in the present work. The selection of this alloy is due to the fact that it demonstrates much faster β to α transformation [25] and thus possesses more weighed displacive character for the transformation.

To obtain the α precipitates produced by the displacive process of the transformation that happens at the very beginning of the formation of α phase according to [20], the samples were aged very shortly after the over β transus solution treatment. In order to obtain the accurate transformation strain values, the lattice constants of the two phases were measured in-situ by neutron diffraction at the transformation temperature.

2. Experimental details

The materials used in the present study is the Ti-7Mo-3Nb-3Cr-3Al (wt. %) (Ti-7333 [26]) alloy prepared first by multiple vacuum arc-melting and then by forging in the β and the $\alpha+\beta$ phase region. The composition was analyzed by capacity chemical analysis method and is given in Table 1. The β transus temperature measured by the metallographic method is approximately 850 °C.

Cylindrical samples with 15 mm in height and 10 mm in diameter were cut out of the center part of the forged Ti-7333 bar (150 mm in diameter). The samples were first solution-treated at 900 °C in the β phase region for 30 min followed by water quenching to obtain a homogeneous single β microstructure. Then the solution treated samples were further aged at 700 °C for 5 min and quenched in ice water to allow one part of the β phase to transform to α phase.

The lattice constants of the constituent phases at the aging temperature (700 °C) were measured in-situ by neutron diffraction. The through-volume measurements were performed with the neutron diffractometer STRESS-SPEC located at a thermal beam port of FRM-II in Garching, Germany. The Ge (311) monochromator was selected to produce neutrons with a wavelength of 1.618 Å. The

bulk samples with dimensions of $\Phi 5 \times 15$ mm were inserted into a vanadium crucible and immersed in the neutron beam with a size of $5 \times 10 \times 10$ mm³ under vacuum to prevent oxidation of the samples at elevated temperatures. The sample was heated to 700 °C and isothermally held for 50 min, and then cooled at a rate of 12 °C/min. A thermocouple was inserted from the top of the crucible to record temperature. Neutron diffraction patterns were collected in-situ during isothermal holding and cooling at each 30 s. The (110) $_{\beta}$ and (100) $_{\alpha}$; (002) $_{\alpha}$; (101) $_{\alpha}$ diffraction peaks were captured at the detector position $2\theta = 41^\circ$ with a detector window of 15° . The instrument parameters were fitted by the measurement of Si powder. The software StressTextureCalculator (STeCa) [27] was used to extract diffraction patterns.

Mesoscopic microstructural features were examined by scanning electron microscopy - electron backscatter diffraction (SEM-EBSD), using a JEOL 6500F SEM equipped with an EBSD acquisition camera and the Aztech online acquisition software package (Oxford Instruments). In order to achieve the surface quality required for EBSD measurements, the samples were first mechanically polished and then electrolytically polished with a solution of 5% per-chloric acid in ethanol at a temperature lower than 15 °C and at 35 V for 15 s. The EBSD patterns were acquired at the accelerating voltage of 15 kV under beam controlled mode with a step size of 0.1 μ m.

The nano scaled microstructural and crystallographic features of the constituent phases were analyzed using a Philips CM 200 transmission electron microscope (TEM) operated at 200 kV. The TEM is equipped with a LaB6 cathode, a Gatan Orius 833 CCD camera, and homemade automatic orientation analysis software – Euclid's Phantasies (EP) [28]. TEM thin films were prepared first by mechanical thinning to a thickness of about 70 μ m and then by electrolytic polishing to perforation at -25 °C in a solution of 5% perchloric acid in methanol at a voltage of 45 V, using a Struers Tenupol-5 twin-jet electropolisher.

For TEM examination, the crystallographic orientation of the microstructural constituents were determined by indexing the TEM Kikuchi line patterns obtained in the nanoprobe mode using the software EP and expressed in a triplet of Euler angles in Bunge's notation [29]. Orientation relationships (OR) between the microstructural constituents were analyzed by misorientation calculation. Dislocations were detected and analyzed by dislocation line match analysis method described in Ref. [30]. To avoid accidental match, at least 3 sample positions were used for each determination. The atomic correspondences between the parent β phase and the product α phase were analyzed using the Crystal Maker[®] [31] software.

3. Results

3.1. Microstructure of the initial β phase

Fig. 1(a) shows the microstructure of the solution-treated Ti-7333 alloy that consists of equiaxed β grains with an average size of about 200 μ m. The disorientation angle distribution of the β grains is shown in Fig. 1 (b). It is seen that the β grains do not display any preferred crystallographic orientations or texture. Moreover, the amount of low angle disorientation ($<5^\circ$) is very low, indicating that the β grains possess high crystalline perfection.

3.2. Lattice constants of the parent β and the product α phase at the transformation temperature

Fig. 2 shows the diffraction pattern of the β phase and α phase acquired at 700 °C (the aging temperature). Analysis of the diffraction data confirmed that the β phase has a BCC structure with lattice parameters $a = 3.2471$ Å; whereas the α phase possesses a

Table 1
Chemical composition of Ti-7333 (wt. %).

Mo	Nb	Cr	Al	Fe	C	O	H	Ti
7.18	2.99	2.94	3.00	0.038	<0.1	0.11	<0.1	Bal.

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