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Three-dimensional analysis of microstructures in titanium

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Abstract

Samples of Ti–4.3Fe–6.7Mo–1.5Al were isothermally annealed in the temperature range of 730–780 °C for various times to study the β–α transformation. Serial sectioning in conjunction with both optical and EBSD analyses was applied to determine the three-dimensional (3-D) morphologies of primary α phase. The 3-D analysis proved to be essential for characterization of the complex morphologies of α grains and consequently for the identification of growth behavior. It showed that nucleation of α grains takes place at β–β grain boundaries and significant branching takes place after initial growth of α grains along β–β grain boundaries. Some branches grow inside the β grain interior. The branching behavior is shown to interact with β–β grain boundaries, leading to a zig-zag morphology. The presented 3-D analysis of α grains and their influence on β–β grain boundaries clearly show that 2-D observations of the microstructural morphologies are not sufficient to adequately represent the transformation characteristics.

Keywords: EBSD; Titanium alloys; Grain morphology; Serial sectioning

1. Introduction

The very wide range of properties that can be effectuated in structural materials, with steel and titanium the most striking examples, is mainly due to a large variety of solid-state phase transformations that occur in these alloys when cooling down from the high-temperature single-phase state. Titanium alloys and iron alloys are very similar in their basic transformation behavior. Although the equilibrium phases at low-temperatures are well defined, the kinetic limitations of the phase transformation in the solid-state lead to the formation of low-temperature microstructures that contain morphologically and compositionally complex phases like bainite and martensite. And although the thermodynamic basis has been well established, many aspects of the solid-state phase transformations that are related to the kinetics of the process remain not completely resolved.

The transition temperature (T_β) in pure titanium between the high-temperature phase with body-centered-cubic crystal structure (bcc, indicated by β) and the low-temperature phase with hexagonal-close-packed structure (hcp, indicated by α) is 882 °C, and it changes with the addition of various alloying elements. The characteristics of transformations that occur in Ti-alloys strongly depend on composition, and the transformation temperatures can even decrease to below room temperature. Therefore, Ti-alloys are classified according to the dominant phase at room temperature [1]. In the case of the so-called α + β alloys, when cooling down from temperatures above T_β, at a high cooling rate, a martensitic reaction can occur. At slow cooling, during which the main alloying elements can partition between β and α phase, primary α is formed. At intermediate cooling rates, the α phase is formed in a distinctly different morphology, a reaction that has been

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shown to occur according to a different mechanism [2–4]. This α phase is often called secondary α, but has also been designated as black plates or bainitic α by several authors [2–6].

Although the properties of different phases in steel are much better known than those in titanium, there are important advantages in studying phase-transformation phenomena in Ti-alloys: (i) the transformation takes place at a longer time scale; (ii) the size distribution of the phases involved is often coarser; and (iii) the β phase can be retained upon cooling to room temperature. All three effects enable a much more detailed and accurate characterization of the microstructure and of the distribution of alloying elements in titanium than in steel.

The microstructural data used for understanding and developing models for phase transformations in metals are usually obtained from the surface of polished specimens. However, in order to come to a description of the actual microstructure, this approach requires assumptions to convert two-dimensional features into three dimensions, which can lead to serious errors, as was for instance shown already in the 1960s by Hillert [7] for the case of interconnectivity of ferrite in pearlite. For an adequate microstructural characterization it is necessary to understand how phase constituents are distributed in three-dimensional space, which can be achieved by serial sectioning and three-dimensional (3-D) reconstruction using advanced hardware and software. Recently, the combination of focused ion beam scanning electron microscopy (FIB-SEM) [8] (so-called dual beam microscopy) and 3-D X-ray diffraction (XRD) [9], among other techniques, have become popular to make complete 3-D reconstructions of microstructures in solids. The FIB-SEM technique is characterized by a considerable resolution, but suffers from the drawback that the maximum observed volume is still quite small. 3-D XRD has the advantage of being a non-destructive technique which allows for in situ observation of transformation kinetics, but is, apart from being time-consuming, not accessible on a laboratory scale.

Although serial sectioning has its beginnings in the early 20th century, it was not until the early 1990s, with the advent of sophisticated computer programs, that it became feasible to study 3-D structures in detail. Most studies were performed on steel microstructures, such as pro-eutectoid ferrite [10,11], pearlite [7,12] and cementite [13–15]. In addition, some microstructures in Ti-alloys [16], Ni alloys [17] and Al alloys [18] have been investigated.

The present work focuses on the formation (nucleation and growth) of the primary α phase in the β Ti alloy Ti–4.5Fe–6.8Mo–1.5Al. With respect to its formation mechanism and morphology, the primary α phase is similar to allotriomorphic ferrite in steels. Since the first observations by Aaronson et al. [5] it has been considered that nucleation occurs at the grain boundaries and is followed by further nucleation and growth of α plates inside the β grains [19–21]. Furthermore, on the basis of conventional two-dimensional (2-D) analysis of microstructures, coalescence [22,23] and spherodization [5] are often assumed to take place at later stages in the transformation. Although there have been a few studies on 3-D microstructures in titanium [24–26], the incompleteness of information available is a major driving factor to conduct further research. In the present paper it will be shown that 3-D observations of the microstructure give new essential information about the morphological features of α grains.

The present study combines serial sectioning employed with optical microscopy and crystallographic orientation analysis of sections by means of electron backscatter diffraction (EBSD). Such an approach has the advantage that a large volume can be studied while ensuring orientation information, which is essential for an unambiguous analysis of the nucleation and growth behavior of the grain morphologies in 3-D space.

2. Experimental procedure

Samples measuring 4.5 mm in diameter and 10 mm in length were used (composition: 4.3 wt.% Fe, 6.7 wt.% Mo, 1.5 wt.% Al, balance Ti). The transus temperature, T_β, of this alloy, determined experimentally, is 800 °C. The heat treatments were performed using a Bähr 805 A/D dilatometer. A thermocouple was spot-welded in the middle of each sample for temperature control. Samples were held between two quartz rods and heated at a rate of 15 °C s^{-1} to a temperature of 900 °C, solution-treated for 10 min at a pressure of less than 3×10^{-8} bar, and cooled to the holding temperature in approximately 2 s to prevent transformation during cooling. Three isothermal treatments were carried out at (a) 780 °C for 18 h; (b) 760 °C for 5 h; and (c) 730 °C for 50 min. The microstructure formed was frozen in by cooling the samples to room temperature at a cooling rate of approximately 40 °C s^{-1}, which is fast enough to prevent formation of secondary α phase. The high cooling rate results in the formation of nano-sized ω precipitates inside β grains [27], but they do not appear in the analysis of the microstructures since they can only be resolved by employing high-resolution microscopy or X-ray methods. No cooling gas was used, in order to prevent oxidation of the specimen surface. After the heat treatment, each sample was cut into two halves in order to analyze the microstructure in the bulk of the samples.

To prepare samples for optical microscopy, all samples were first plane-ground with SiC paper up to 2400 grit finish, each step for 3 min. This was followed by fine-polishing using a mixture of colloidal silica (OP–S) and H_2O_2 for 8 min. Special care was taken to avoid deformation during polishing. Kroll-etching solution (90 ml water, 3 ml HF and 7 ml HNO_3) was used to reveal the microstructure. Optical micrographs were taken using an Olympus BX60 M optical microscope.

For serial sectioning, 11 regions in different samples were chosen and Vickers' hardness indents were made using a Buehler Omnimet® Microhardness tester for subsequent section alignment. Samples were fine-polished using