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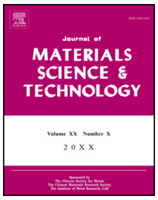

Formation of face-centered cubic titanium in laser surface re-melted commercially pure titanium plate

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ABSTRACT

Micron-scale face-centered cubic titanium phase (named as δ phase) were noticed in the re-melted zone of laser surface re-melted commercially pure titanium plate. The morphology, sub-structure, orientation and distribution of δ phase were investigated by scanning electron microscopy, electron back-scattered diffraction and transmission electron microscopy. Three kind formation processes of δ phase were put forward based on the investigation. The first one is $\alpha' \rightarrow \delta$ transformation which takes place in single α′ grains and leads to the orientation relationship $\{001\}\delta//\{0001\}\alpha' < 110 > \delta// < 11\bar{2}0 > \alpha'$. The second one is $\beta \rightarrow \alpha' + \delta$ transformation which takes place at α'/α' interfaces and leads to the orientation relationship $\{001\}\delta//\left\{1\bar{1}0\right\}\beta\ \langle 110\rangle\delta//\langle 111\rangle\beta$. The third one is another kind of $\beta \rightarrow \alpha' + \delta$ transformation that takes place at α'/α' interfaces and leads to the orientation relationship $\left\{1\bar{1}1\right\}\delta//\left\{1\bar{1}0\right\}\beta < 110 > \delta// < 111 < \beta$. It is believed that the transformations of δ phase are stress assistant ones and in the present investigation, the phase transformation stress of $\beta \rightarrow \alpha'$ transformation acts as the assistant driving force for the formation of δ phase.

1. Introduction

Titanium alloy is widely used in modern society because of its good combination of mechanical properties and corrosion resistance [1,2]. Titanium alloy exhibits a body-centered cubic (BCC) structure (β phase) at high temperature, and a hexagonal close-packed (HCP) structure (α phase) at low temperature. The phase transus temperature (T_β) of pure titanium is $882 \pm 5\,^\circ\mathrm{C}$ [2]. When being cooled slowly, the $\beta \rightarrow \alpha$ transformation happens through long-term diffusion but when being cooled quickly, the transformation would happen through shear or twinning without long-term diffusion. The daughter phases of non-equilibrium transformation involve α′-HCP martensitic phase, α″-Orthorhombic martensitic phase, ω-HCP phase and so on. Generally speaking, face centered cubic (FCC) phase, a common close packed phase in metals, seems not to be abundantly got through heat treatment in titanium and its alloys.

A way to get FCC-Ti is to deposit Ti thin film on some certain substrates, e.g. NaCl single-crystal, Al single crystal and so on [3–5]. Recently, other ways to get FCC-Ti phase has been reported. If block pure titanium undergoes severe plastic deformation like milling [6–8], compression [9] or rolling [10], some nanoscale FCC-Ti phase can be made. At the same time, other researchers reported that FCC-Ti phase appeared in some kinds of titanium alloys after plastic deformation [11,12] or heat treatment [13,14]. There is a certain crystallographic orientation relationship (OR) between the FCC-Ti phase and the surrounding HCP phase, which is $\{0001\}_{\mathrm{HCP}}//\{001\}_{\mathrm{FCC}}$ and $<11\bar{2}0>_{\mathrm{HCP}}//\langle 110\rangle_{\mathrm{FCC}}$. Based on the above OR, two types of HCP to FCC transformation models were put forwarded by Hong et al. [9] and Wu et al. [10], separately. Hong et al. [9] considered that the glide of Shockley partial dislocations of $\frac{a}{6} < 11\bar{2}0 >$-type on prism planes led to the transfer from α phase to FCC phase. Wu et al. [10] came up with a nucleation model with a minimums stable thickness of three atomic layers and a grow model with a minimums grow step thickness of two or four atomic layers. Zhang and Ying [8] owe the FCC-Ti to the nanocrystallization and the density and influence of crystal defects introduced by ball milling. The FCC phase caught researchers' attention as it reveals an unusual transformation process in titanium and its alloys, and appropriate FCC phase is thought to provide extra ductility since glide and twinning in FCC structure are more easily than those in HCP structure [11].

So far, the FCC-Ti phases found in block pure titanium are all products of plastic deformation and the length of them is smaller

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than 1 μm [6–10]. But in the melted zone of laser surface re-melted TA2 plate, widely distributed micron-scale FCC-Ti phase has been observed. In the present paper, the distribution, morphology, sub-structure and orientations of FCC-Ti grains has been investigated and the transformation mechanisms of them have been put forward.

2. Materials and experiment

In the present study, commercially pure titanium TA2 (Grade 3 in USA standard) plate was used as substrate for the laser surface re-melting experiment. The received heavy TA2 plate was cut into small plates of 50 mm × 20 mm × 15 mm by wire-electrode cutting. The broad surface of these small plates were mechanically polished first and re-melted by a continuous wave fiber laser under an argon atmosphere with oxygen content less than 80 ppm later. The parameters of laser working were 4 kW of power, 6 mm of beam diameter and 10 mm/s of scanning speed.

Secondary electron microscopy (SEM) and electron backscattered diffraction (EBSD) of the re-melted zone were carried out using an FEI NANO SEM 430 microscope after electrochemically polished. The scan gap of EBSD was 0.15 μm. A 0.3 mm thin film specimen of re-melted zone was grinded to 50 μm in thickness followed by ion-milling and then transmission electron microscopy (TEM) was carried out on a JEOL JEM-2100F transmission electron microscope.

3. Results

3.1. Morphology and orientation of δ-FCC phase

For better description, the FCC-Ti phase is named as δ phase in the present paper. SEM image of the electrochemically polished re-melted zone of laser surface re-melted TA2 plate is shown in Fig. 1(a). On this SEM image, δ-FCC phase can be easily picked up from the α'-HCP matrix as the δ grains are protuberant on the polished surface because of their better corrosion resistance. On the EBSD phase map shown in Fig. 1(b), δ-FCC phase is colored in yellow while α' matrix is colored in red. The distribution of yellow area in phase map matches the distribution of protuberances in SEM image well, which confirms that the protuberances on the polished surface are δ grains. Based on the orientation imaging map (OIM) shown in Fig. 1(c), the α' matrix consists of several grains, which belong to several variants. The δ grains, like α' matrix, belong to three variants, which are the purple ones in the top and bottom (named as δ_1 and δ_4), the blue one in the middle-up (named as δ_2) and the orange one in the middle-low (named as δ_3 and pointed by red circle). Not like the FCC phase reported by the previous literature [6–10] that are nanoscale, the δ-FCC grains found in laser surface re-melted TA2 plate are in micron-scale. The δ_3 grain, the smallest δ-FCC grain found in Fig. 1, is 0.6 μm wide and 2.4 μm long while the δ_4 grain, the largest δ-FCC grain found in Fig. 1, is 2.7 μm wide and 12.4 μm long.

The micron-scale δ-FCC grains showed certain orientation relationships with the surrounding α' grains. On the SEM image shown in Fig. 2(a), there is a protuberance of δ grain which is 2.3 μm wide and 14.2 μm long. Based on the OIM image shown in Fig. 2(b), one can know that the δ grain is inner single α' grain (named as α'_1). Fig. 2(c) and (d) shows the compound pole figures of δ grain and surrounding α' grains and the parallel orientations between δ and α'_1 grain are pointed out by single arrows. The double arrow on Fig. 2(d) shows the related $\langle 111\rangle_{bcc}$ orientation of the prior β grain. The OR between δ and α'_1 is $(001)\delta//(0001)\alpha'_1$ and $[110]\delta//[\bar{2}110]\alpha'_1$, the same as the reported ORs in the deformed block pure titanium [9,10].

A δ grain that located at α'/α' interface is shown in Fig. 3. As shown in Fig. 3(a), this δ grain is surrounded by α'_1 grain in three sides and contacts α'_2 grain in right side. The dimension of this δ grain is 7.2 μm wide and 14.6 μm long and the boundaries between δ grain and α' grains are zigzag. Inner the δ grain, there are some fragments of α'_1 grain, which indicates a phase transformation process of $\alpha'_1 \rightarrow \delta$ or $\beta \rightarrow \alpha'_1 + \delta$. The orientations of δ grain and surrounding α' grains are shown in Fig. 3(b) and (c) by the compound pole figures. The parallel orientations between δ grain and α' grains are pointed out by the single arrows, which are $(111)\delta//(0001)\alpha'_2$ and $[1\bar{1}0]\delta//[11\bar{2}0]\alpha'_1//[11\bar{2}0]\alpha'_2$. The double arrow shows the related $\langle 111\rangle_{bcc}$ orientation of the prior β grain. On the assumption of $\beta \rightarrow \alpha'_1 + \delta$ transformation, the OR can be written as $(111)\delta//(10\bar{1})\beta$ and $[1\bar{1}0]\delta//[1\bar{1}1]\beta$ considering the Burgers OR between α' and β grain. In this OR, the close packed plane of β grain, $(10\bar{1})\beta$ plane, is parallel to the close packed plane of δ grain, $(111)\delta$ plane, and the close packed orientation of β grain, $[1\bar{1}1]\beta$ orientation, is parallel to the close packed orientation of δ grain, $[1\bar{1}0]\delta$ orientation. On the assumption of $\alpha'_1 \rightarrow \delta$ transformation, there is no low Miller index plane of α'_1 grain that parallel to the $\{111\}\delta$ planes of δ phase. Thus the δ grain in Fig. 3 may be the daughter phase of $\beta \rightarrow \alpha'_1 + \delta$ phase transformation process.

Fig. 4 shows another α'/α' interface, at which there exists two kinds of δ variants. The δ_1 grains (colored in orange in Fig. 4(a)) are surrounded by α'_1 grain in three sides and contact with α'_2 grain in right side. The OR between δ_1 grains and α' grains is $(001)\delta_1//(0001)\alpha'_2$ and $[110]\delta_1//[11\bar{2}0]\alpha'_1//[11\bar{2}0]\alpha'_2$, which can be rewritten as $(001)\delta_1//\left(\bar{1}10\right)\beta$ and $[110]\delta_1//[11\bar{1}]\beta$. Like the δ grain in Fig. 3, the δ_1 in Fig. 4 is believed to be the daughter phase of $\beta \rightarrow \alpha' + \delta$ transformation. The δ_2 grains (colored in green in Fig. 4(a)) are surrounded by α'_2 grain in three sides and contact α'_1 grain in left side. The OR between δ_2 and α'_2 is $(001)\delta_2//(0001)\alpha'_2$ and $[110]\delta_2//[\bar{2}110]\alpha'_2$. This OR is same as the OR between δ grain and α' grain in Fig. 2, which is regarded as a result of $\alpha' \rightarrow \delta$ transformation [9,10].

3.2. Sub-structure of δ-FCC phase

The sub-structure of δ-FCC phase was investigated by TEM and the result is shown in Fig. 5. A 2.3-μm-wide δ-FCC zone is found and illustrated in Fig. 5(a). As can be seen, the δ-FCC zone can be divided into four small plates by three interfaces (pointed out by arrows). The width of each δ plate varies from 260 nm to 730 nm. Fig. 5(b) shows the composite selected area diffraction patterns (SADP) of these four δ-FCC plates in $[\bar{1}12]_{fcc}$ axis. The diffraction spots are distorted, which indicates small misorientation among the four δ plates. A conclusion can be made that the large FCC grains found in SEM images and OIM images in Figs. 1–4 are actually made up by a series of small δ-FCC plates that have small misorientation with each other. Dark field (DF) images of the δ-FCC zone using $\mathbf{g} = [220]_{fcc}$ vector and $\mathbf{g} = [1\bar{1}1]_{fcc}$ vector are shown in Fig. 5(c) and (d) separately. Plenty of equal inclination interference lines can be seen in the DF images. This is a sign of internal stress and plastic deformation. These δ plates are partially bright in the two vectors, indicating the existence of misorientation inner each plate and the existence of same orientation among different plates. This is a sign of internal stress and plastic deformation, too.

4. Discussion

4.1. Distribution of δ grains

In the re-melted zone of laser surface re-melted TA2 plate, the volume fraction of δ phase is about 0.7%, an average fraction acquired by EBSD analysis. Most of the δ grains distributed at α'/α'

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