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Nanotwinned Ti(O,C) induced by oriented attachment in a hot-pressed Nb–Ti–Al alloy

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ABSTRACT

The microstructures and formation mechanisms of various kinds of Ti(O,C) twins in a Nb–23Ti–15Al alloy have been investigated. The crystallographic features of the twins are systematically characterized using transmission electron microscopy (TEM). Lamellar or corner Ti(O,C) twins consist of coherent {111} twin boundaries (TBs) and incoherent {112} TBs. The three-fold Ti(O,C) twins are newly found, with symmetric Σ9 {114} or asymmetric Σ9 {115}/{111} serving as the secondary TBs. Moreover, slabs on Ti(O,C) TBs showing three-layer periodic contrasts are identified to be the twin overlapping. According to the microstructure characteristics, we propose a new model based on oriented attachment to interpret the formation of Ti(O,C) twins, which involves the contact, rotation, attachment and Ostwald ripening of Ti(O,C) grains. Our results provide new insights on the formation mechanisms of nanotwinned grains.
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1. Introduction

In recent years, due to the increased requirement for materials operating at higher temperature in advanced turbine systems, considerable interests in Nb-based ultra-high temperature alloys have been ignited [1–3]. Nb–Ti–Al alloys, consisting of the Nb solid-solution phase (β phase, body-centered cubic (BCC) A2 structure) and the major strengthening phase Nb_3Al (δ phase, primitive cubic A15 structure), have exhibited great potential for high-temperature applications [4–6].

To further improve the high-temperature strength of Nb–Ti–Al alloys, Ti(O,C) [7] or TiC [8,9] phase displaying face-centered cubic (FCC) B1 structure has been introduced for dispersion strengthening. Ti(O,C) could be regarded as a solid-solution of TiC, with O on the lattice points substituting C [7]. Moreover, nanotwinning has attracted much attention to improve the strength of metals [10–12] without deteriorating the ductility. However, no investigation has been attempted for the strengthening of Nb–Ti–Al alloys utilizing nanotwinned Ti(O,C) or TiC.

It is well known that the twin boundary energy (TBE) of TiC is quite high (0.927 J/m^2) [13], therefore, TiC twins are scarcely observed. Nevertheless, impurity elements (such as Al, Si and B) or vacancies are found to stabilize TiC twins, since the TBE could be substantially reduced [13,14]. Multiple twins, such as three- or five-fold twins, are frequently observed in nanocrystallines with FCC structures [15,16]. However, although TiC twins in the form of nano-lamellae [17,18] and corner twins [19] have been observed, it remains unclear whether the Ti(O,C) or TiC twins with higher orders could appear, let alone the characterization and formation mechanism analysis of the multiple twins.

In this study, various kinds of Ti(O,C) twins in a Nb–23Ti–15Al alloy have been systematically examined using transmission electron microscopy (TEM). The morphology and crystallographic characteristics of Ti(O,C) twins have been studied, and the formation mechanisms discussed.

2. Experimental

The Nb–23Ti–15Al (at.%) alloy was fabricated via hot-pressing using mechanically alloyed (MAed) powder. The raw materials employed are commercial elemental powders of Nb, Ti and Al, with the purities of 99%, 99% and 99.5% (wt.%), respectively. The powders were mixed and then MAed in a Shunchi PMQW4L 3-D planetary ball mill filled with argon at 250 rpm for 12 h. Afterwards, the MAed powder was hot-pressed in a TenBoo RY70-16 vacuum hot-pressing furnace at 1350 °C for 1 h with a pressure of 30 MPa, followed by furnace cooling to room temperature.

The foils of the alloy for TEM examinations were mechanically

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thinned to <50 μm, and then electropolished in a twin jet polisher using a solution of 10 vol.% H_2SO_4 in methanol at −30 °C and 60 mA. Subsequently, the foils were further thinned via ion milling. Microstructural observations and selected area electron diffraction (SAED) analyses were conducted with a JEOL JEM-2100 TEM operating at 200 kV.

3. Results and discussion

3.1. Two-fold Ti(O,C) twins

As described in Ref. [7], the Nb–23Ti–15Al alloy contains δ, β and Ti(O,C) phases, and Ti(O,C) is formed due to residual C and O in the elemental powders. Interestingly, various nanotwinned Ti(O,C) are identified in TEM examinations. Fig. 1 shows typical TEM morphologies and SAED patterns of two-fold Ti(O,C) twins, which can be classified into two distinct types: lamellar twins (Fig. 1a–c) and corner twins (Fig. 1d. In a corner twin, the twin boundary displays as a single interface separating the twin and matrix portions [20–22]). The twinning relationships are reflected in the corresponding SAED patterns. Statistically, the thickness of lamellar twins ranges from 50 nm to 500 nm, and the average TB spacing is about 250 nm. It can be seen that the lamellar twins contain pairs of parallel Σ3 {111} twin boundaries (TBs), while the corner twins only consist of one Σ3 {111} TB.

The lamellar Ti(O,C) twins terminate either at Ti(O,C) grain boundaries (Fig. 1a) or inside a Ti(O,C) grain with incoherent twin boundaries (ITBs) (Fig. 1c). In the latter case, ITBs vertical to the coherent twin boundaries (CTBs) are introduced. Another scenario to produce ITBs is the formation of steps on CTBs, as indicated by circles in Fig. 1c. Analogously, ITBs of corner Ti(O,C) twins are also occasionally observed on the steps of CTBs. According to the SAED patterns in Fig. 1, the CTBs and ITBs of the two-fold Ti(O,C) twins are Σ3 {111} and Σ3 {112}, respectively. Since the TBE of ITBs is extraordinarily higher than that of CTBs [23], ITBs in TiC or Ti(O,C) are only sporadically observed previously [23,24].

It is worth noting that a small quantity of internal porosity indicated by short arrows in Fig. 1a appears in the Ti(O,C), which has been repeatedly verified by tilting experiments. Fig. 1b shows an enlargement of the porosity nucleating on the TB. It is revealed that the porosity takes the shape of internal crystalline facets of {111} and {002} to decrease the inner surface energy, due to the low surface energy of the planes [25]. The porosity is formed via condensing supersaturated vacancies [26]. Similarly, the porosity inside the Ti(O,C) grain, marked by a double-headed arrow in Fig. 1a, is also faceted in shape.

3.2. Three-fold Ti(O,C) twins

Apart from the two-fold twins mentioned above, three-fold Ti(O,C) twins are also observed in the Nb–Ti–Al alloy, which have not been reported previously. Fig. 2 shows the morphologies and SAED patterns of two three-fold Ti(O,C) twins. The twin in Fig. 2a consists of three segments labeled as T1, T2 and T3, respectively. According to the SAED pattern in Fig. 2c, the three-fold twin is formed via two consecutive rotations of 70.5° counterclockwise (CCW) about the [011] axis, since the angle between the $(11\bar{1})$ and $(\bar{1}1\bar{1})$ is 70.5°. In other words, T2 is rotated 70.5° CCW relative to T1, and T3 is rotated 70.5° CCW relative to T2. The T1/T2 and T2/T3 are the primary Σ3 {111} TBs, which are sharp and coherent. Based on the SAED pattern in Fig. 2c, the T1/T3, a secondary TB, is determined to be the symmetric Σ9 {114}, and thus the mirror relationship is kept between T1 and T3. In sharp contrast to the coherent primary TBs, the secondary TB shows dotted character, indicating that the Σ9 {114} secondary TB might be semicoherent. The atomic configurations of Σ9 {114} grain boundaries in the Au bicrystal are studied in Ref. [27], which show small structural repeat distance of the interface. Therefore, the dotted character of the Σ9 {114} secondary TB might be ascribed to the defects introduced by structural units.

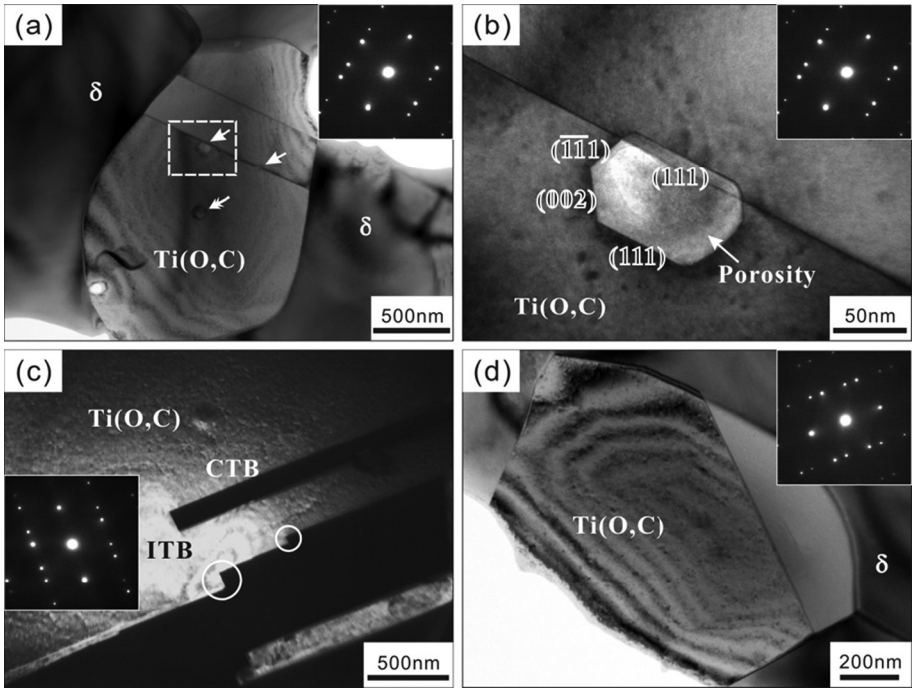

Fig. 1. TEM observations showing morphologies and SAED patterns of two-fold Ti(O,C) twins. (a) Bright-filed (BF) image of a lamellar twin; (b) high-magnification image of the rectangle region marked in (a) showing the internal porosity; (c) dark-field (DF) image of lamellar twins with coherent twin boundaries (CTBs) and incoherent twin boundaries (ITBs); (d) BF image of a corner twin. The SAED patterns of the twins are inserted in the corresponding images.

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