



Regular article

Transitional structure of $\{332\}\langle 113 \rangle_{\beta}$ twin boundary in a deformed metastable β -type Ti-Nb-based alloy, revealed by atomic resolution electron microscopy

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ABSTRACT

In this work, the boundary structure of the $\{332\}\langle 113 \rangle_{\beta}$ deformation twin in a deformed metastable β -type Ti-Nb-Pd alloy has been clearly revealed by means of high-angle annular dark-field scanning transmission electron microscopy at atomic-resolution level. It is found that stress-induced α'' martensite structure remains in the $\{332\}_{\beta}$ twin boundary region, exhibiting a gradual transition to the β structure with an orientation relationship of $(1\bar{3}0)_{\alpha''}/(332)_{\beta}$. This finding provides a direct experimental evidence to verify that the formation of a $\{332\}_{\beta}$ twin in deformed metastable β -type Ti-Nb-based alloys can be associated with a reversible β -to- α'' martensitic transformation.

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The $\{332\}\langle 113 \rangle$ deformation twinning is a unique deformation mode for metastable β -Ti alloys, which was first noticed early in the 1970s by Blackburn and Feeney in a Ti–11.5Mo–6Zr–4.5Sn alloy [1]. As deformation products, $\{332\}\langle 113 \rangle_{\beta}$ twins have been observed to form in many other deformed metastable β -Ti alloys [2–16]. The formation process of $\{332\}$ deformation twin has been attracted much interest because it will strongly influence the deformation behaviors and mechanical properties of those metastable β -Ti alloys. In general, the $\{332\}\langle 113 \rangle_{\beta}$ twinning is suggested to be responsible for low yield strength and large uniform elongation of these metastable β -Ti alloys [17–19].

Although it has been found that the $\{332\}\langle 113 \rangle_{\beta}$ twinning can be a predominant deformation mode in many metastable β -Ti alloys, however, why and how it can realize have still remained as unsolved questions. Several $\{332\}\langle 113 \rangle_{\beta}$ twinning models have been proposed so far based on different viewpoints. According to the conventional twinning theory, one-half of the atoms in the bcc β structure have to shuffle in addition to atom shearing operations in order to form a $\{332\}\langle 113 \rangle_{\beta}$ twinned structure [20]. However, it is hardly conceivable that the complex shuffle involved with so many atoms is energetically likely to occur. Within the framework of the dislocation theory, the formation of a $\{332\}\langle 113 \rangle_{\beta}$ twin has been described as an even more complex process including both the movement of partial dislocations along the $\{332\}_{\beta}$ twinning plane and necessary shuffles of atoms as well [21,22]. Taking

into account the lattice instability of the metastable β phases, Kim et al. pointed out that the $\{332\}\langle 113 \rangle_{\beta}$ twinning should be more favorable to be activated in a kind of stress-induced modulated structure rather than directly in the β structure [23]. More recently, the $\{332\}\langle 113 \rangle_{\beta}$ twinning system in the metastable β -Ti alloys with forming ability of stress-induced α'' martensite has been compared with the possible $\{130\}\langle 310 \rangle_{\alpha''}$ twinning system in the α'' structure and good crystallographic correspondence between them has been illustrated [24,25]. Accordingly, it has been inferred that the formation of a $\{332\}\langle 113 \rangle_{\beta}$ twin could be the consequence of the reversion from a parent $\{130\}\langle 310 \rangle_{\alpha''}$ twin formed in the stress-induced α'' martensitic structure [25]. However, the direct experimental evidence to confirm this speculation is still lacking. In so far as all of the $\{332\}\langle 113 \rangle_{\beta}$ twinning mechanisms proposed previously, the main effort is just to rationalize the possible twinning processes through geometrical or theoretical analysis, however, the fundamental question of what intrinsic structural features should be for their twinning boundaries (TBs) had not been the focus of these mechanisms. As well known, deformation twinning will influence the mechanical properties of deformed materials [26], and on the other hand, the twinning boundaries should provide some important hints for understanding the twinning process. It is therefore crucial to reveal interfacial structures of TBs at atomic resolution if we are to understand the related twinning mechanisms well. Due to the experimental difficulty to acquire structure images of TB through conventional HREM observation, structural characterization at atomic resolution level for the $\{332\}\langle 113 \rangle_{\beta}$ deformation twinning boundary is still an important issue to be tackled.

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The present work is concerned with the structures of $\{332\}\langle 11\bar{3}\rangle_{\beta}$ twinning boundaries in metastable β -type Ti-Nb-based alloys. An as-cast Ti-30Nb-3Pd (wt. %) alloy was chosen as research object because of its good ductility at room temperature [27] and good $\{332\}\langle 11\bar{3}\rangle_{\beta}$ twin formability. We will report the structural characterization on the TBs of $\{332\}\langle 11\bar{3}\rangle_{\beta}$ deformation twins at atomic resolution level by means of aberration corrected high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM), which has allowed us to find the existence of a kind of transition structure in the $\{332\}\langle 11\bar{3}\rangle_{\beta}$ deformation twin boundary. It will be discussed that this finding can be a direct evidence for rationalizing the formation mechanism of a $\{332\}\langle 11\bar{3}\rangle_{\beta}$ deformation twin in the metastable β structure.

Alloy ingots with composition of Ti-30Nb-3Pd (wt%) were prepared by arc melting pure Ti, Nb and Pd metals with a purity of 99.99% under an Ar atmosphere. Each alloy ingot was melted several times to ensure compositional homogeneity, and then cooled down in the arc furnace. Tensile specimens with a gauge length of $8 \times 2 \times 1$ mm were cut from the as-cast alloy ingots by electric discharge machining, and tensile deformation was carried out to 20% strain at room temperature with a strain rate of $8.3 \times 10^{-3} \text{ s}^{-1}$. TEM and STEM samples were prepared by mechanical grinding and then by twin-jet polishing at -30°C using the electrolyte consisted of 12.5 vol% sulfuric acid and 87.5 vol%

methanol. HAADF-STEM observations were made in a 300 kV probe corrected electron microscope (Titan G² 60-300 S/TEM).

Plenty of $\{332\}\langle 11\bar{3}\rangle_{\beta}$ deformation twins can be introduced into the metastable β -type Ti-30Nb-3Pd alloy by tensile deformation to 20% at room temperature. Fig. 1a shows the bright-field TEM image of a typical twin plate in the deformed sample. The selected-area electron diffraction pattern taken from the region marked by red circle in Fig. 1a along the $[1\bar{1}0]_{\beta}$ zone axis is presented in Fig. 1b, from which one can clearly identify that this deformation twin plate is of $(332)[11\bar{3}]_{\beta}$ type. Notice that in addition to the reflections from the matrix and twin, some extra weak reflections which can be indexed as the α'' martensite structure are also visible. Also in Fig. 1c and d, which are TEM dark-field images corresponding the matrix and twin plate in Fig. 1a, one can see the existence of some strap-shaped contrasts in both matrix and twin plate other than the complex contrast related with the dislocations of high density, which cannot simply be attributed to the formation of stress-induced ω phase. These observations imply that the formation of $\{332\}_{\beta}$ deformation twin must have undergone a complex process. The nature of these strap contrasts need to be investigated later in more details. Here we just place our emphasis to interfacial structural characterization for the $\{332\}_{\beta}$ deformation twin boundary.

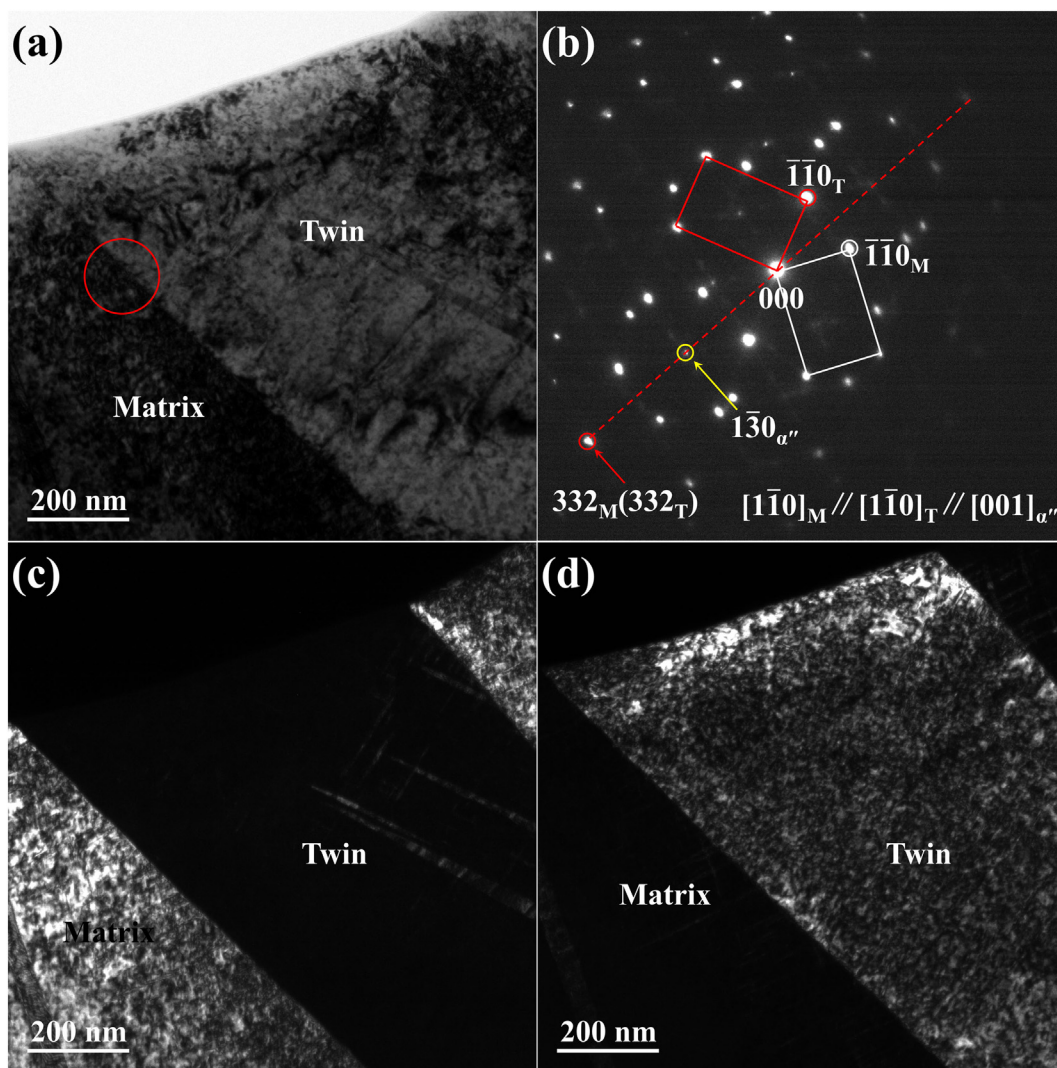


Fig. 1. (a) TEM bright-field image of a $(332)[11\bar{3}]_{\beta}$ deformation twin formed in the deformed Ti-30Nb-3Pd alloy; (b) electron diffraction pattern taken from the twin boundary region marked by red circle in (a) along the $[1\bar{1}0]_{\beta}$ zone axis; TEM dark-field images of the matrix (c) and twin (d), taken using diffraction spots $(\bar{1}\bar{1}0)_M$ and $(\bar{1}\bar{1}0)_T$ marked in (b), respectively. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

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