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Stress release-induced interfacial twin boundary ω phase formation in a β type Ti-based single crystal displaying stress-induced α'' martensitic transformation

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Abstract

The ω phase transformation in numerous group IV transition metals plays a key role to change the phase stability and modify mechanical properties, although its formation mechanisms and effect to accommodate strains are still unclear. A $\langle 1\ 1\ 0 \rangle_{\beta}$ single crystal of β -type Ti-24Nb-4Zr-8Sn alloy (wt. %) is used to investigate the aforementioned problems. This alloy displays a β to α'' stress-induced martensitic transformation, which is reversible and leads to superelasticity. Profiles obtained from *in situ* synchrotron X-ray diffraction during cyclic tensile tests reveal definitely the appearance of ω phase due to applied stress releasing after unloading while these identified ω peaks shall vanish totally on subsequent loading. TEM microstructural observations show its morphology as thin layer locating on the boundary of $\{1\ 1\ 2\}\langle 1\ 1\ 1 \rangle_{\beta}$ twins coinciding well with the feature of interfacial twin boundary (ITB) ω phase, which is proved to be formed passively during the reversion of $\{1\ 1\ 0\}\langle 1\ 1\ 0 \rangle_{\alpha''}$ twins. The martensitic twinning components of $\{1\ 1\ 0\}\langle 1\ 1\ 0 \rangle_{\alpha''}$ are indeed confirmed from crystallographic reconstructions based on the orientation relationship between the β and α'' phases. A geometrical model is hence schematically used to analyze in details the orientation relationships between both deformation $\{1\ 1\ 2\}\langle 1\ 1\ 1 \rangle_{\beta}$ twins and ITB- ω phase, as well as with the original $\{1\ 1\ 0\}\langle 1\ 1\ 0 \rangle_{\alpha''}$ twinning.

Keywords: ITB- ω phase, deformation twinning, $\{1\ 1\ 2\}\langle 1\ 1\ 1 \rangle_{\beta}$, $\{1\ 1\ 0\}\langle 1\ 1\ 0 \rangle_{\alpha''}$, interfacial

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phase, stress-induced martensitic transformation.

1. Introduction

Metastable β -type titanium based alloys are currently developed for applications in medicine and aerospace. Phase transformations from the body-centered cubic β phase (Im-3m, N° 229) to base-centered orthorhombic α'' phase (Cmcm, N° 63) and hexagonal ω phase (P6/mmm, N° 191) are observed to be formed leading to several particular properties, such as superelasticity and shape memory effect. Although the stress-induced α'' phase transformation from β phase has been widely studied with the classic theory of martensitic crystallography (PTMC) [1-3], the role of ω phase was not clearly established. Especially, the importance of ω phase to accommodate internal strains in alloys, its effect on martensitic α'' phase transformation and the role as precursor for stable α phase are still in debates.

As brittle particles known to be deleterious on mechanical properties, the isothermal ω phase was firstly identified in aged Ti-Cr alloy [4]. Various external stimulation including rapid cooling from single β phase field in high temperature range, mechanical deformation, extremely high pressure and exposure to radiation were then claimed to enable the inducement of the ω phase in numerous group IV transition metals (Ti, Ta, Hf and Zr) based alloys [5-9]. Depending on different historical processes, the particle morphologies of ω phase can be ellipsoidal or cuboidal after ageing [10-12] and plate-like in strained titanium alloys [13-15]. These morphological differences for ω phase can be attributed to both compositional partition and stressing condition [10-12, 16-18]. However, the model of collapsing one pair of $\{1\ 1\ 1\}_{\beta}$ planes to an intermediate position leaving the adjacent $\{1\ 1\ 1\}_{\beta}$ planes unaltered were proved and widely accepted as the formation mechanism of ω phase for nearly all cases [3, 19, 20]. Amongst them, thin layer ω phase located along the twin boundary, which was called ITB- ω [18], deserves more investigation. The two twinning systems observed in β -type Ti alloys are $\{1\ 1\ 2\} \langle 1\ 1\ 1 \rangle_{\beta}$ [18, 19, 21-25] and $\{3\ 3\ 2\} \langle 1\ 1\ 3 \rangle_{\beta}$ [26-31]. In a recent work carried out in a Ti-Nb alloy [18], the formation of ITB- ω was observed due to a collapsing process of the $\{3\ 3\ 2\}_{\beta}$ twin planes. The formation of such ITB- ω phase along $\{3\ 3\ 2\} \langle 1\ 1\ 3 \rangle_{\beta}$ twins was also shown to be intrinsically due to the reverse α'' to β transformation and the subsequent stress relaxation along twin boundaries.

Such kind of ITB- ω phase was also observed in the vicinity of the $\{1\ 1\ 2\} \langle 1\ 1\ 1 \rangle_{\beta}$ twin boundaries in a deformed Ti-15Mo-5Zr (at. %) alloy [32]. Further, ω -phase with a zigzag morphology was observed in a Ti-34Nb (at. %) alloy and its formation was proposed to be featured

by three-layer lattice shears arising from the dissociation and cross-slip of screw dislocations on consecutive $\{1\ 1\ 2\}_\beta$ planes [23]. A detailed dislocation mechanism using the dissociation of the core of $1/2\ \langle 1\ 1\ 1 \rangle_\beta$ dislocations into three $1/6\ \langle 1\ 1\ 1 \rangle_\beta$ twinning dislocations and $1/12\ \langle 1\ 1\ 1 \rangle_\beta$, $1/3\ \langle 1\ 1\ 1 \rangle_\beta$ and $1/12\ \langle 1\ 1\ 1 \rangle_\beta$ transition dislocations was described for interpretation of the mentioned boundary complexions [21]. Another mechanism was proposed from first principle calculations coupling with microstructural observations in the Ti-30Nb-3Pd (at. %) alloy: The nucleation and growth of $\{1\ 1\ 2\}_\beta$ twins can be described on the basis of the reverse transformation of ω phase into β phase [24, 33]. With such a mechanism, $\{1\ 1\ 2\}_\beta$ twins are formed inside ω particles and the ITB- ω phase observed along twin boundaries are the untransformed part of ω particles involved in the twinning process. The formation of ITB- ω phase along $\{1\ 1\ 2\}_\beta$ twin boundaries is thus mainly describe from mechanisms which do not consider the β - α'' transformation. But the comprehensive participation of martensitic α'' transformation and $\{1\ 1\ 2\}_\beta$ deformation twins on the formation mechanism for the ITB- ω phase is of great interest and a deeper understanding on the formation sequence or mediation details have never been reported.

In the present work, a β -type titanium based alloy with the chemical composition of Ti-24Nb-4Zr-7.9Sn (wt. %, short for Ti2448 thereafter) was investigated. This Ti2448 alloy was firstly designed for biomedical implants and prostheses because it exhibits a Young's modulus as low as 42 GPa and a superelastic behavior characterized by a maximum recoverable strain of 3.3% [25, 34-39]. As the origin of superelasticity, the stress-induced α'' martensitic transformation was evidenced in polycrystalline deformed specimens [37]. In the present case, a $\langle 1\ 1\ 0 \rangle_\beta$ single crystal of this Ti2448 alloy was elaborated to simplify the microstructure. *In situ* synchrotron X-ray diffraction (SXRD) under cyclic tensile test were used to detect the stress-induced martensitic α'' phase transformation and ITB- ω phase formation. Further TEM and orientation analysis were performed to reveal the transformation details. A new deformation sequence for deformation β twinning and ITB- ω formation, as well as the relevant geometrical model indicating the change of the crystallographic lattice were finally obtained.

2. Materials and methods

Ingot of Ti-24Nb-4Zr-8Sn (wt. %) alloy with diameter of 55 mm was made by arc melting under argon and hot forged at 1273 K into 25 mm diameter bar. A cylinder of 8.6 mm in diameter was cut from the as-forged bar and used to grow seed-crystal rods in an optical floating-zone furnace (FZ-T-12000-X-VP-S, Crystal System Inc.) under the protection of flowing argon. The growth was

carried out at a rate of 5 mm.h⁻¹ and a rotation rate of 25 rpm. The seed crystal was cut along the $\langle 1\ 1\ 0 \rangle_{\beta}$ direction after the orientation was determined by both X-ray diffraction (XRD) and Laue X-ray back reflection analyses within 1° of the desired orientation and then used to grow single-crystal rods. The orientation of the grown single crystal was also confirmed by Laue X-ray back reflection analysis. XRD and transmission electron microscopy (TEM) were used to examine the single β phase and confirm the absence of formation of both ω phase and α'' martensitic phase. Chemical analyses showed that the grown crystals has a similar composition compared to the forged bar.

Cyclic tensile test at room temperature consisting of strain increments of 0.5 % followed by stress release up to an elongation of 5.5 % was conducted with an INSTRON 3369 tensile machine (strain rate: 10⁻⁴ s⁻¹). An extensometer was used to ensure the accurate control of the strain. The tensile direction was chosen parallel to the single crystal growth direction, i.e. the $\langle 1\ 1\ 0 \rangle_{\beta}$ direction. In order to observe the deformed microstructure, single crystal specimens were cut after tensile tests and prepared by mechanically grinding with increasing grades of SiC abrasive paper, then polished with colloidal silica suspension and finally etched in a solution composed of 8 % HF, 15 % HNO₃ and 77 % H₂O (vol. %). Further observations were conducted using a JEOL 2100 transmission electron microscope (TEM) operating at 200 kV. Thin foils for TEM observations were cut as disks of 3 mm in diameter, further polished to a thickness of 150 μ m and prepared by a twin-jet electro-polishing technique with a 6 % perchloric acid and 94 % methanol (vol. %) solution at -25 °C. Twin-jet electro-polishing was stopped before perforation and the samples were finally thinned by ion milling with a Fischione 1010 Model.

In situ synchrotron X-ray diffraction (SXR) under cyclic tensile test was conducted on beam line ID31 at the European Synchrotron Radiation Facility (ESRF, Grenoble, France) with a 5 kN micro-tensile dispositive. The high-resolution ID31 beamline offers a high-energy incident X-ray beam ($\lambda = 0.040002106$ nm) coupled with a nine-channel multi-analyzer installed as detectors. The transmitted diffracted beams were collected over the θ angular range 2–14 ° with a scanning step of 0.005 °. The *in situ* cyclic tensile tests under synchrotron radiation were realized on the same single crystal tensile specimen (gauge width of 3 mm and a thickness of 0.5 mm) with increments of 0.5 % until 5.5 %. SXR scans were therefore obtained after each strain increment for both loading and unloading conditions. More details about the setup configuration can be found in reference [40].

3. Results

3.1. Tensile test

Fig.1 displays the cyclic tensile curve from 0 to 5.5 % strain for the $\langle 1\ 1\ 0 \rangle_\beta$ single crystal. On this curve, large hysteresis loops can be clearly observed due to the stress-induced martensitic transformation (SIM) occurring. Indeed, after the linear elasticity of the β phase, a stress plateau can be noticed in the strain range of 0.75 ~ 3 % which corresponds to the main stage of SIM α'' transformation. Thus, the critical stress for the SIM α'' transformation can be evaluated to be 250 MPa with the method of intersection point between two tangent lines. After 3 % of strain, the martensitic α'' phase begins to deform elastically followed by its plastic deformation. The recoverable strains used to evaluate the superelasticity at different applied strains are measured with the method illustrated in reference [41] and displayed with black open circles according to the right vertical axis. It can be noticed that the recoverable strain increases linearly with the applied strain up to 4 % of strain. Up to this value, the applied strain can be totally recovered due to the reversibility of the SIM α'' transformation. On the other hand, the incipient Young's modulus and the ultimate tensile strength are measured to be 51 GPa and 623 MPa, respectively.

3.2. *In situ* SXRD characterization

Fig. 2a presents the whole SXRD profiles of the single crystal during the cyclic tensile test on loading (upper in red) and after unloading (lower in black) between 0 and 5.5 % of applied strain. On the first initial profile, only $(1\ 1\ 0)_\beta$ and $(2\ 2\ 0)_\beta$ peaks are detected, which is in accordance with the $\langle 1\ 1\ 0 \rangle_\beta$ single crystalline state. When the applied strain increases, SIM α'' transformation occurs. However, the diffracted peaks belonging to α'' phase are very close to the $\{1\ 1\ 0\}_\beta$ and $\{2\ 2\ 0\}_\beta$ peaks that is why three enlarged angle ranges from Fig. 2a are presented in Fig. 2b, 2c and 2d for a better identification of the phase evolution.

In the Fig. 2b that shows profiles on loading, it can be noticed that the $(1\ 1\ 0)_\beta$ peak slightly shifts to the low angles due to the elastic expansion of the β lattice until the detection of the $(0\ 2\ 0)_{\alpha''}$ diffraction peak when the applied strain reaches 1 %. Then, the other typical diffraction peaks of $(0\ 0\ 2)_{\alpha''}$ and $(1\ 1\ 1)_{\alpha''}$ appear at higher applied strain. Once the strain exceeds 3 %, the β phase seems to disappear totally that will be confirmed hereafter from the calculation of transformation strains. This assumption is highlighted by the vanishing of $(1\ 1\ 0)_\beta$ peak replaced by the superimposition of the three $(0\ 2\ 0)_{\alpha''}$, $(0\ 0\ 2)_{\alpha''}$ and $(1\ 1\ 1)_{\alpha''}$ diffraction peaks. Under the

corresponding unloading conditions in Fig. 2c, nearly all the diffraction peaks of the α'' phase disappear while the peak of the β phase appears back, showing the α'' phase reverses back to its parent β phase before the strain reaches 3.5 %. Positions of peaks of α'' phase are also indicated, but the quantity of residual martensite is negligible for the 3.5% condition, while its presence is more obvious at higher strain, even if the β phase is the main phase remaining after unloading. Moreover, the reversed $(1\ 1\ 0)_\beta$ peak and remaining peaks of α'' (when there are) keep in the same position, implying that the shift of all peaks observed on loading is due to the elasticity of the two phases. For profiles under the unloading condition released from a higher applied strains at 4.5 ~ 5.5 % in Fig. 2c, diffraction peaks of $(0\ 0\ 2)_{\alpha''}$ and $(1\ 1\ 1)_{\alpha''}$ with tiny intensity are left around the main $(1\ 1\ 0)_\beta$ peak. This is because the SIM α'' transformation cannot be totally reversed due to the onset of plastic deformation which inhibits the reverse martensitic transformation.

Another interesting phenomenon can be noticed in Fig. 2d. Indeed, two small but well-identified peaks at 5.6° and 7.6° are clearly visible. However, they cannot be indexed from any α'' or β phases. In fact, these two peaks can be indexed as the $(0\ 1\ -1\ 0)_\omega$ and $(0\ 0\ 0\ 1)_\omega$ planes of the ω phase, which is also a common observed phase in both deformed and quenched metastable Ti-based alloys. Such a formation of ω phase when the stress is released from the plastic deformation domain was never reported and the reason of its appearance will be explicated further from the microstructural analysis.

The deformation sequence of the present $\langle 1\ 1\ 0 \rangle_\beta$ single crystal can be therefore extracted on the basis of both the SXRD profiles and analysis of cyclic tensile curves in the Fig. 1. The deformation begins from pure elastic deformation of β phase. At the 0.5 % strain, the SIM α'' starts to occur and continues to coexist with the elastic deformation of the β phase until the fulfillment of the phase transformation at 3 % strain. At the strain range of 3 ~ 4.5 %, the SIM α'' phase deforms elasticity then stepped into a complete plastic deformation range of 4.5 ~ 5.5 % characterized by the stabilization of permanent deformation. During this last step, an unexpected formation of ω phase after releasing the stress is also evidenced.

Evolutions of the cell parameters of β and SIM α'' phases are also determined from the diffraction profiles and are plotted in Fig. 3 as a function of the applied percentage strain under loading conditions. The lattice parameter of β phase is continuously increasing when the strain increases until 3 % strain on loading while keeps constant after unloading. For the SIM α'' phase, the $b_{\alpha''}$ and $c_{\alpha''}$ parameters continuously increases when the strain increases while the $a_{\alpha''}$ parameter is roughly constant. It is also worth noting that lattice parameters of the α'' phase cannot be properly determined for applied strain smaller than 3 % due to a limited number of diffraction peaks

although SIM α'' transformation already occurs. The cell parameters of β phase, SIM α'' phase and ω phase, which are determined within the plastic deformation range after unloading, are presented in Table 1. These values are used to calculate the phase transformation strains for each possible variants of α'' martensite (Table 2). As generally assumed, this is the variant leading to the maximum of transformation strain that would be formed, i.e. the CV3 here with a value of about 4.3%. This means the recoverable strain would be measured as 4.3% if all the β phase transforms into α'' martensite with a full reversibility after unloading. As the maximum recoverable strain was indeed measured as 4.3% (Fig. 1), one can conclude that all the β phase is transformed during deformation as assumed from the description of SXRD results (Fig. 2).

3.3. Microstructural observations

An example of the plastically deformed microstructure observed by optical microscopy for a sample deformed at 4.5 % is presented in Fig. 4. Two groups of deformation bands crossed with each others can be clearly observed on this micrograph. In order to determine the nature of these bands, TEM observations were carried out.

Fig. 5 shows the TEM observations of the single crystal deformed at 4.5 % strain. The bright field image (BFI) in Fig. 5a presents dislocation pile-ups indicating the involvement of dislocation slip. The selected area electron diffraction (SAED) of Fig. 5b corresponding to the circled area of the matrix in Fig. 5a was indexed as the β phase with the $[1\ 1\ 3]_{\beta}$ zone axis. A careful analysis of the slip trace on the Fig. 5a according to the crystallographic orientation of the single crystal shows the dislocations slip in a $\{1\ 1\ 0\}_{\beta}$ plane (more details about the method can be found in reference [42]). This result is consistent with the most common slip planes observed in the β phase of titanium alloys but also in other bcc alloys [43-45]. A BFI from another typical area is presented in Fig. 5c and its corresponding SAED in Fig. 5d. It can be seen the superimposition of three sets of diffraction patterns marked by different colors on the SAED. After indexation, the deformation bands observed in Fig. 5c correspond clearly to the $\{1\ 1\ 2\}<1\ 1\ 1>_{\beta}$ twinning system but additional ω phase diffracted spots are also observed sharing the common zone axis for $[1\ 1\ 0]_{\beta\text{-matrix}} // [1\ 1\ 0]_{\beta\text{-twin}} // [-1\ -1\ 2\ 0]_{\omega}$.

The TEM observations of another area in Fig. 6a and 6b also confirms the formation of plastic $\{1\ 1\ 2\}<1\ 1\ 1>_{\beta}$ twins and the formation of ω phase but with a different diffracting condition corresponding to $[-1\ -3\ 1]_{\beta\text{-matrix}} // [-1\ -1\ -3]_{\beta\text{-twin}} // [1\ -2\ 1\ -3]_{\omega}$ zone axis. It can be noticed that, for this specific orientation, the common $\{1\ 1\ 2\}$ plane between the twin and the matrix, which can be seen on the SAED pattern, is not the twinning plane. The twinning plane will be further indicated in

the Fig. 7a and 7b. Diffracted spots related to the twin and ω phase are selected and relevant dark-field images are shown in Fig. 6c and 6d. It is worth noting that the ω phase shows a strong contrast, which allows to observe its localization at the twin boundary. This indicates its nature of interfacial twin boundary phase (ITB- ω). The ω phase layer appears here rather thick because the twin interface is inclined according to the electron beam. Due to the crystallographic orientation of this twin, it was not possible to get the twin interface edge-on (see Fig. 7a and 7b). It can also be noticed that such an ITB ω phase exhibiting a thin layer morphology was already reported in other β -metastable titanium alloys [16, 18, 19].

This ITB ω phase is thus the ω phase that is detected during the SXR D experiments, as no nano-sized athermal ω phase can be detected from SAED patterns. Indeed, no diffuse streaks are visible on the $[113]_{\beta}$ zone axis SAED pattern (Fig. 5b) as commonly observed when precipitation of athermal ω phase occurs during the quench [3, 10, 13]. Therefore, the ω phase detected by SXR D is not athermal ω phase initially present in the microstructure but is truly the ITB ω phase observed by TEM along the twin boundaries.

4. Discussion

We showed by SXR D analysis that the microstructure of samples after stress released from the plastic deformation range consists mainly of β phase with a small amount of residual SIM α'' phase and ITB- ω phase. However, the TEM observations on sample released from 4.5 % do not exhibit any detection of SIM α'' phase. The reason is due to internal stress relaxation occurring during the thin foil preparation, which is reasonably accepted. The point to elucidate now concerns the mechanism of the ITB- ω phase formation. Indeed, this phase is never observed when the sample is under stress but only when the sample is released after being deformed in the plastic domain according to our SXR D analyses. The problem is that many transformations occurred in this metastable alloy such as the stress-induced martensitic transformation, twinning and ω -phase formation, which can be reversible and depend on each other.

The SIM α'' transformation was confirmed by SXR D experiments with a phase-transformed fraction of 100 % when a strain of 4.5 % is reached. That means that the plastic deformation mechanisms, such as twinning and dislocation slip, occur in this SIM α'' phase and not in the β phase. Indeed, the plastic deformation starts between 4 and 4.5% of applied strain, as some residual strain starts to be measured after unloading from 4.5% (Fig. 1). In addition, as the formation of ω phase is observed only after unloading from 4.5% of applied strain, that means the ω phase is formed after plastic deformation has occurred, and thus after twinning of martensite has occurred. It is also worth

noting that the ω phase is not detected after further loading (5%) but appears again when the stress is removed again: The formation of ω phase is thus reversible, meaning that the observed twins in the β phase with the ITB ω phase can transform again into twins in the α'' martensite without any ω phase during subsequent loading. Plastic deformation mechanisms of α'' martensite are not exhaustively known but twinning was already reported in the literature as the $\{1\ 3\ 0\}\langle 3\ 1\ 0\rangle_{\alpha''}$ or the $\{1\ 1\ 0\}\langle 1\ 1\ 0\rangle_{\alpha''}$ twinning systems [46-48]. Therefore, the observed $\{1\ 1\ 2\}\langle 1\ 1\ 1\rangle_{\beta}$ twins combined with the stress release-induced ITB- ω phase formation is believed to be linked directly with the α'' martensitic twinning under stress condition. In order to prove this hypothesis, the α'' martensitic twinning components has to be detailed on the basis of the well-known orientation relationship (OR) between the body-centered cubic β phase and the orthorhombic α'' phase given in Table 2 [49]. These data are used to make the crystallographic reconstruction of α'' twins on loading from the crystallographic orientations of the β phase observed after the stress is removed.

By way of illustration, the crystallographic orientations of the β -matrix and β -twin observed in the Fig. 6 are presented on stereographic projections in Fig. 7a and 7b. The common $(-1\ -2\ -1)_{\beta}$ twinning plane is indicated as well as the common $\langle 1\ 1\ 1\rangle_{\beta}$ twinning direction. The corresponding crystallographic orientations of matrix and twin after transformation into α'' phase were then determined by applying the OR. Actually, six possible orientations of α'' , named variants (V1 to V6) were deduced from a certain orientation of β -matrix or β -twin. All the combinations between both orientations of matrix and twin were tested in order to identify a potential twinning relationship in the α'' phase under stress condition. For that, a rotation of 180° around the potential common twinning direction or reflection through the common twinning plane were made to confirm the twinning relationship. The tested potential common twinning planes and directions were referenced in several articles related to α'' martensite or similar structure in α -uranium [47, 50-52]. Finally, only one convincing possibility was found between the α'' -matrix-V3, and α'' -twin-V3, both represented in Fig. 7c and Fig. 7d, respectively. The twinning relationship found here corresponds thus to a compound $\{1\ 1\ 0\}\langle 1\ 1\ 0\rangle_{\alpha''}$ twin.

It is demonstrated clearly that the α'' -matrix-V3 and α'' -twin-V3 keep a $\{1\ 1\ 0\}\langle 1\ 1\ 0\rangle_{\alpha''}$ twinning relationship, because (i) the two projections share a common $(-1\ -1\ 0)_{\alpha''}$ pole and a common $[-1\ 1\ 0]_{\alpha''}$ direction indicated in Fig. 7c and 7d and (ii) the operation of a reflection through the $(-1\ -1\ 0)_{\alpha''}$ plane or a rotation of 180° around the $[-1\ 1\ 0]_{\alpha''}$ direction for the α'' -matrix-V3 leads to the orientation of the α'' -twin-V3. Therefore, the α'' -matrix-V3 and α'' -twin-V3 are in the compound $\{1\ 1\ 0\}\langle 1\ 1\ 0\rangle_{\alpha''}$ twinning relationship. Further, the loading direction of the single crystal was ascertained as $[1\ 0\ 1]_{\beta\text{-matrix}}$. Along this loading direction, the phase transformation strain

283 can be thus calculated according to theory of PTMC [53] and results were presented in Table 2. The
 284 maximum value is calculated as 4.29 % for V3. As it is generally assumed that the variant that is
 285 formed during a tensile test is the one giving the maximum of transformation strain, the occurrence
 286 of V3 is then in good agreement with the orientation of the single crystal of β phase. It can also be
 287 noticed that, according to the Fig. 1, the residual strain of approx. 0.2 % is measured at the applied
 288 strain of 4.5 % which is exactly the sum of phase transformation strain (4.29 %) and residual strain
 289 (0.2 %). Thus, the results of crystallographic reconstruction to determine the twinning system in α''
 290 martensite are in perfect accordance to the variant selection of α'' due to the external stress.

291 Moreover, the twinning plane $(-1\ -1\ 0)_{\alpha''}$ and the twinning direction $[-1\ 1\ 0]_{\alpha''}$ indicated in Fig. 7c
 292 coincide exactly with the twinning plane $(-1\ -2\ -1)_{\beta}$ and the twinning direction $[1\ -1\ 1]_{\beta}$ in Fig. 7a,
 293 respectively, indicating a clear lattice correspondence between the deduced $\{1\ 1\ 0\}\langle 1\ 1\ 0\rangle_{\alpha''}$
 294 twinning system in the α'' phase and the observed $\{1\ 1\ 2\}\langle 1\ 1\ 1\rangle_{\beta}$ twinning system in the relaxed β
 295 phase. The crystallographic orientation of the ITB- ω phase obtained from the DP in Fig. 6b is also
 296 shown with a stereographic projection in Fig. 7e. Some interfacial features can be found obviously:
 297 the twinning planes of $(-1\ -2\ -1)_{\beta}$ and $(-1\ -1\ 0)_{\alpha''}$ are indeed parallel to the $(0\ -1\ 1\ 0)_{\omega}$, while the
 298 twinning directions of $[1\ -1\ 1]_{\beta}$ and $[-1\ 1\ 0]_{\alpha''}$ are parallel to the $[0\ 0\ 0\ -1]_{\omega}$ of the ITB- ω phase. The
 299 present orientation relationship between β and ω phase is specific for ITB- ω phase with a strong
 300 correlation between the twinning elements and the ω phase. Indeed, the twinning plane is parallel to
 301 $(0\ -1\ 1\ 0)_{\omega}$ and the twinning direction is parallel to the $[0\ 0\ 0\ 1]_{\omega}$ direction. This specific orientation
 302 relationship was already pointed out in a gum metal alloy for the same twinning system [16] and
 303 also in a Ti-Nb binary alloy for the $\{3\ 3\ 2\}\langle 1\ 1\ 3\rangle_{\beta}$ twinning system [18], both exhibiting such
 304 ITB- ω phase. An explicit illustration of these orientation relationships is exhibited in Fig. 7f based
 305 on eighteen unit cells of β phase highlighted with red color. The unit cell of α'' phase and ITB- ω
 306 phase are then highlighted with blue and green colors, respectively. Moreover, the path for atoms
 307 displacements which are necessary to realize the phase transformation are marked by hollows atoms
 308 and corresponding arrows.

309 The release of applied strain from the plastically deformed single crystal causes the reverse
 310 martensitic transformation, thus the microstructure is observed as the resultant $\{1\ 1\ 2\}\langle 1\ 1\ 1\rangle_{\beta}$ type
 311 with ITB- ω phase that is formed in order to minimize the surface energy of complex boundaries. As
 312 reported in a recent work [18], the ITB- ω phase is assumed to accommodate strains that are
 313 originated from the reverse martensitic transformation of the $\{1\ 3\ 0\}\langle 3\ 1\ 0\rangle_{\alpha''}$ twin boundary into
 314 $\{3\ 3\ 2\}\langle 1\ 1\ 3\rangle_{\beta}$ twin interfaces in a metastable β -type Ti-Nb alloy. The ITB- ω phase formed in the
 315 present case during the transformation from $\{1\ 1\ 0\}\langle 1\ 1\ 0\rangle_{\alpha''}$ twin boundary into $\{1\ 1\ 2\}\langle 1\ 1\ 1\rangle_{\beta}$

twin actually follows the similar mechanisms but is activated along different crystallographic planes and directions.

The geometrical models for the deformation twinning and ITB- ω phase are presented in Fig. 8 according to the lattice correspondence in Fig. 7f. The projection of the lattice is chosen to be along the planes of $(0\ 0\ -1)_{\alpha''\text{-matrix}} // (0\ 0\ 1)_{\alpha''\text{-twin}} // (-1\ 0\ 1)_{\beta\text{-matrix}} // (1\ 0\ 1)_{\beta\text{-twin}} // (2\ -1\ -1\ 0)_{\text{ITB-}\omega}$. All solid circles indicate the atoms in the plane of figure, while the open circles the atoms below the plane of figure. The red, blue and green atoms refer to the initial lattice, the twinned lattice and the resultant ITB- ω lattice, respectively. The Fig. 8a firstly illustrates $\{1\ 1\ 0\} \langle 1\ 1\ 0 \rangle_{\alpha''}$ twinning formed via plastic deformation on the basis of fully transformed α'' matrix. The twinning plane $(-1\ -1\ 0)_{\alpha''\text{-matrix}}$ and twinning direction $[-1\ 1\ 0]_{\alpha''\text{-matrix}}$ are marked. It is worth noting, that the atomic layers A, B, C are not at equal distance to each other. The distance between these planes is dependent on the value of y , which is a parameter related to the positions of atoms in the orthorhombic cell of the α'' phase. Its value lies between $1/6$ and $1/4$, while the value $y=1/6$ describes an hcp structure (α phase) and the value $y=1/4$ describes a bcc structure (β phase).

The observed $\{1\ 1\ 2\} \langle 1\ 1\ 1 \rangle_{\beta}$ twin and ITB- ω phase which are passively formed after releasing the stress are also presented in Fig.8b. It can be noticed that the reverse transformation from α'' to β leads to an equal distance between A, B and C atomic layers. Moreover, these atomic layers are also equally distanced in the ω phase. So, the reverse transformation is helpful to form an ITB- ω phase during the phase rerversion. Some atoms have thus to be slightly moved to form the ω phase (arrows on the fig. 8b). The final configuration of the twin interface after removing the stress is then illustrated in the Fig.8c with the orientation relationship between the ITB- ω phase and both the twin and matrix of the β phase which are in a $\{1\ 1\ 2\} \langle 1\ 1\ 1 \rangle_{\beta}$ twinning remationship.

5. Conclusions

The present investigation employed a single crystal with a nominal chemical composition of Ti-24Nb-4Zr-8Sn (wt. %) to investigate the ω phase on the twin boundaries. Techniques including cyclic tensile test, *in situ* synchrotron X-ray diffraction, TEM observations and crystallographic orientation reconstruction are used to comprehensively analyze the deformation features. Results as following are obtained:

- (1) The $\langle 1\ 1\ 0 \rangle_{\beta}$ Ti2448 single crystal exhibits a maximum recoverable strain of 4.2 % at the applied strain of 4.5 %. Incipient young's modulus and ultimate tensile strength were measured to be 51 GPa and 623 MPa. SXRD profiles confirmed the superelasticity is due to phase transformation and cell parameters were thus obtained.

- (2) Surprisingly, apparition of diffracted peaks related to the ω phase is found on SXRD profiles when the stress is removed, only from the plastic deformation range. These peaks disappear after further loading and appear back after subsequent unloading. The $\{1\ 1\ 2\}\langle 1\ 1\ 1\rangle_{\beta}$ deformation twinning is identified with diffraction patterns and TEM dark field images.
- (3) Taking the sample deformed up to 4.5 % as an example, the applied strain is accommodated by martensitic α'' phase transformation strain (4.29 %) and plastic deformation leading to residual strain after unloading (0.2 %). The TEM analysis also shown the presence of ITB- ω phase located along twin boundaries. Accordingly to SXRD results, this ITB- ω phase is formed when the stress is released.
- (4) From crystallographic reconstruction based on the orientation relationship, results illustrate that the observed $\{1\ 1\ 2\}\langle 1\ 1\ 1\rangle_{\beta}$ and ITB- ω phase are the resultant lattice complexion due to the reversion of $\{1\ 1\ 0\}\langle 1\ 1\ 0\rangle_{\alpha''}$ twins. The ITB- ω phase formed is due to the compression strains during the reversed martensitic transformation on the twinning boundaries.
- (5) The sequence of deformation can thus be described as follow:
- ① during loading, the β phase firstly transforms into α'' martensite;
 - ② when this transformation is fully accomplished, $\{1\ 1\ 0\}\langle 1\ 1\ 0\rangle_{\alpha''}$ twins are formed in the α'' martensite during subsequent plastic deformation, as well as dislocation slip occurs;
 - ③ when the stress is removed from the plastic deformation range, the reverse transformation from α'' to β phase occurs;
 - ④ the twins formed into α'' martensite reverse thus into a deformation band exhibiting a $\{1\ 1\ 2\}\langle 1\ 1\ 1\rangle_{\beta}$ twinning relationship;
 - ⑤ a thin layer of ω phase lying along the twin boundary (ITB ω phase) is also formed in order to relax the stress accompanying this reverse transformation.

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498

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Table 1. Cell parameters of different phases after releasing stress at higher applied strain in the plastic deformation range.

Cell parameters (Å)	loading		Unloading			
	SIM α'' phase		β phase		ω phase	
	4.5 %	5.5 %	4.5 %	5.5 %	4.5 %	5.5 %
a	3.261	3.260	3.298	3.298	4.732	4.732
b	4.864	4.865			4.732	4.732
c	4.848	4.851			2.991	2.991

Table 2. The six lattice correspondence of variants for martensitic α'' phase derived from the parent β phase and the calculated strain of phase transformation at the external loading direction of $[1\ 0\ 1]_{\beta}$.

Variants correspondence	$[100]_{\alpha''}$	$[010]_{\alpha''}$	$[001]_{\alpha''}$	Transformation strain along the loading direction of $[101]_{\beta}$	
				Applied strain of 4.5%	Applied strain of 5.5%
CV1	$[100]_{\beta}$	$[011]_{\beta}$	$[0\bar{1}1]_{\beta}$	1.50	1.50
CV2	$[100]_{\beta}$	$[0\bar{1}1]_{\beta}$	$[0\bar{1}\bar{1}]_{\beta}$	1.50	1.50
CV3	$[010]_{\beta}$	$[101]_{\beta}$	$[10\bar{1}]_{\beta}$	4.29	4.31
CV4	$[010]_{\beta}$	$[10\bar{1}]_{\beta}$	$[\bar{1}0\bar{1}]_{\beta}$	3.94	4.01
CV5	$[001]_{\beta}$	$[110]_{\beta}$	$[\bar{1}10]_{\beta}$	1.50	1.50
CV6	$[001]_{\beta}$	$[\bar{1}10]_{\beta}$	$[\bar{1}\bar{1}0]_{\beta}$	1.50	1.50

Fig. 1. Cyclic tensile curve of the single crystal Ti-24Nb-4Zr-8Sn alloy with the corresponding measured recoverable strains (open circles).

Fig. 2. Synchrotron X-ray diffraction profiles of the single crystal Ti-24Nb-4Zr-8Sn alloy: (a) the full diffractograms on loading and corresponding unloading and (b-d) enlarged partial diffractograms marked in (a) with precise indexation.

Fig. 3. Evolution of the cell parameters of the different phases determined from the SXRD profiles.

Fig. 4. Optical microstructure of the single crystal Ti-24Nb-4Zr-8Sn alloy after deformation up to 4.5 % strain.

Fig. 5. TEM observations of the sample deformed up to 4.5 % strain: (a) pile-ups of dislocations and (b) corresponding selected diffraction pattern of the area marked by white circle in (a); (c) two deformation bands crossing with each other and (d) the selected diffraction pattern of the area marked by white circle in (c).

Fig. 6. TEM observations of one single deformation band for the sample deformed up to 4.5 % strain: (a) bright field image and (b) the corresponding selected area diffraction patterns; (c) and (d) the dark field images taken with the spots highlighted by red and green circles in (c) corresponding to $(-1\ 1\ 0)_{\beta}$ and $(-2\ 1\ 1\ 0)_{\omega}$, respectively.

Fig. 7 Crystallographic orientations represented as stereographic projections used to determine the orientation relationship between the matrix and twin shown in Fig. 6: the $(-1\ -1\ -2)[1\ -1\ 1]_{\beta}$ twinning relationship between the β -matrix (a) and the β -twin band (b); the compound $(-1\ -1\ 0)[-1\ 1\ 0]_{\alpha''}$ twinning relationship between the α'' -matrix-V3 (c) and the α'' -twin band-V3 (d); (e) the orientation of ITB- ω phase and (f) the lattice correspondence of β -matrix, α'' -matrix-V3 and ITB- ω phase.

(a) loading

(b) unloading

(c) unloading

Fig. 8 Schematic illustration of orientation relationships for single crystal Ti-24Nb-4Zr-8Sn alloy: (a) the compound $(-1 -1 0)[-1 1 0]_{\alpha'}$ twinning relationship on loading, (b) the $(-1 -1 -2)[1 -1 1]_{\beta}$ twinning relationship and formation of ITB- ω after unloading and (c) the whole reversed morphology under unloading condition.