Tensile behavior of orthorhombic α'' -titanium alloy studied by *in situ* X-ray diffraction

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ABSTRACT

The tensile behavior of a Ti–11%Zr–14%Nb–10%Sn alloy with pure orthorhombic α'' phase was studied by *in situ* X-ray diffraction using synchrotron radiation. It is found that no phase transformation happens during the whole tensile process. The “double-yielding” platforms of this alloy are indeed due to a low stress yielding (~ 400 MPa) followed with a significant work-hardening before necking and fracture. In this process, the $[022]$ orientation of grains more approaches the tensile direction and the $[200]$ moves to the transverse, causing the lattice parameter a to be shrunk, and b and c elongated, and the formation of texture. The similar texture can also be produced upon cold rolling by which the yield strength of the α'' phase is largely improved to be over 900 MPa.

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1. Introduction

Titanium (Ti) based alloys have attracted a great deal of attention due to their unique properties, such as light weight, low elastic modulus, high strength and excellent biocompatibility and corrosion resistance, which make them the first candidate for biomedical and aerospace applications [1–3]. Four kinds of phases have been reported among the Ti-based alloys, i.e., high temperature bcc β phase with space group $Im\bar{3}m$ (No. 229), low temperature α phase ($P6_3/mmc$, No. 194), ω phase ($P6_3/mmm$, No. 191) and orthorhombic α'' phase ($Cmcm$, No. 63). Phase transformations have been reported between bcc β and hcp α phase, e.g., in Ti–Al–Nb [4] and Ti–6Al–4V [5] alloys, while phase transformations from β to α'' or from β to ω , in some cases to a mixture of α'' and ω , were observed upon rolling, under tension or high pressure [6,7]. Recently, the report of the so-called Gum metal with pure β phase [8] prepared by cold working triggers further interests to understand the mechanical behavior of Ti-based alloys under tension. Hot-rolled Ti–Zr–Nb–Sn [7] and Ti–Nb–Ta–Zr–O [9] alloys exhibit a low elastic modulus after preloading and a reversible phase transformation between β and α'' , leading to a non-linear elastic behavior. Since tiny ω -phase

grains are usually located at the β/α'' interface, they are suggested to suppress the β/α'' martensitic transformation by impeding the β/α'' interface movement [9,10].

Orthorhombic α'' phase has a close structural relationship to the hcp α , bcc β and hcp ω phases [11]. Upon ageing, bcc β and hcp ω phases can be precipitated from α'' phase [12]. Hence, an interesting question is raised: what is the mechanical behavior of Ti-based alloy with pure α'' phase upon deformation? To the best of our knowledge, this issue has not been addressed yet. In this work, we report the mechanical behavior of a new Ti-based alloy with pure α'' phase based on *in situ* X-ray diffraction (XRD). The tensile behavior and structural changes of the α'' phase have been carefully examined. Although the “double-yielding” platforms are observed, no phase transformation appears prior to the final fracture.

2. Experimental

Pre-alloyed ingots of nominal composition Ti–11%Zr–14%Nb–10%Sn (wt.%) were prepared by arc melting under a Ti-gettered purified argon atmosphere. Elements of Ti, Zr, Nb and Sn with purities higher than 99.9% were used as raw materials. Each alloy ingot of about 25 g was remelted several times to improve the homogeneity. From ingots, sheet-shaped samples in 1 mm thick, 10 mm wide and 60 mm long were prepared by suction casting in a water-cooled copper mould. The crystalline phase of the as-cast sample was determined to be single α'' phase. The sheets were selected to fabricate tensile test specimens (40 mm $\times$ 10 mm $\times$ 1 mm) with a

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Fig. 1. XRD patterns for the as-cast and heat-treated Ti-11%Zr-14%Nb-10%Sn samples. The Rietveld refinement shows only single α'' phase in the as-cast sample.

4 mm diameter reamed hole at each side. The experimental details can be referred to Ref. [13]. The beam size was $1 \text{ mm} \times 1 \text{ mm}$ and the wavelength was $0.12644 \text{ \AA}$. Diffraction patterns were recorded on an image plate (MAR 345, with $150 \mu\text{m} \times 150 \mu\text{m}$ pixel size) on the BW5 station, HASYLAB. The total scattering intensity $I(q)$ versus 2θ was obtained by integrating the full ring patterns. However, in order to know the texture evolution with the increase of stress, the $I(q)$ changing along the rings were extracted by azimuthally caking 10° fraction in steps along the ring pattern, and then integrating within these partial areas using the software package FIT2D [14]. The phase structures were refined using the Rietveld program [15]. Meanwhile, the tensile behavior of Ti-based samples was also measured on a universal testing machine CMT5205 SANS, China at a strain rate of $1 \times 10^{-4} \text{ s}^{-1}$.

3. Results and discussion

3.1. Phase structure and tensile behavior

Fig. 1 shows the XRD pattern of the as-cast Ti-11%Zr-14%Nb-10%Sn sample at 0 MPa together with the Rietveld refinement. As a comparison, XRD pattern of bcc β phase was also examined for this alloy. Here β phase is formed by heat treatments in two steps from the as-cast sample. They are initially annealed at 1173 K in a sealed quartz tube for 1 h, then quenched into the cooled water, and the second step is to reanneal the sample at 673 K for 0.5 h in a sealed quartz tube, then cooled in the air. Note that the Bragg peaks (1 1 0) and (2 1 1) of β phase almost overlap with (0 0 2) and (1 1 3) peaks of α'' phase while the peak (2 0 0) of β phase does not overlap with any peak of α'' phase. Thus, the peak (2 0 0) of β phase from XRD can be used to identify the absence of β phase in the as-cast sample at zero stress. The XRD pattern of the as-cast sample can be refined using an orthorhombic α'' phase. The unit cell parameters for the α'' phase are found to be $a = 3.1144 \text{ \AA}$, $b = 4.9716 \text{ \AA}$ and $c = 4.7274 \text{ \AA}$. Fig. 2(a) plots the stress-strain curve for the as-cast sample upon tension. The “double-yielding” platforms at about 400 MPa and 800 MPa obviously appear. Are “double-yielding” platforms similar to the non-linear elastic behavior for the Ti-Nb-Ta-Zr-O [9] alloy with a β phase? To address this question, cyclic loading for the as-cast Ti-11%Zr-14%Nb-10%Sn sample was conducted in Fig. 2(b). The alloy undergoes plastic deformation when the stress is up to 400 MPa, which is revealed by the presence of residual strain after unloading. When reloading the sample to 600 MPa and 700 MPa, the yield stress is greatly improved. This is most likely due to the work-hardening, suggesting that the low yield strength of the as-cast α'' phase might be enhanced by deformation. This is supported by the

Fig. 2. (a) Stress-strain curves for the as-cast and 50% cold-rolled Ti-11%Zr-14%Nb-10%Sn samples at a strain rate of about $1 \times 10^{-4} \text{ s}^{-1}$. (b) Stress-strain curves of cyclic loading and unloading for the as-cast sample at a strain rate of about $1 \times 10^{-4} \text{ s}^{-1}$.

enhancement of yield strength up to 900 MPa for a 50% cold-rolled sample in Fig. 2(a).

3.2. Structural evolution upon tension

To further study possible structural evolution during tension, *in situ* X-ray diffraction measurements for the as-cast sample were performed as shown in Fig. 3(a). From the peak positions and intensity difference between the experimental and the calculated ones, it was found that the only orthorhombic α'' phase was detected while no any other new Bragg peaks were observed during the whole tensile process. The change of intensity for major diffraction peaks upon tension could indicate the formation of texture. Fig. 3(b) displays the two-dimensional diffraction images for the sample at 0 MPa and 900 MPa, from which the texture can be clearly observed at 900 MPa. To understand which orientations are preferred in the α'' phase during tension, the ring patterns were caked azimuthally in 10° fractions and integrations were performed within these partial areas along the ring patterns in 36 steps. Fig. 4(a) plots intensity profiles of three plane indices (0 0 2), (0 2 0) and (1 1 1) as a function of azimuth angle φ at 0 MPa and 700 MPa. It is found that the variation in intensity versus azimuth angle φ demonstrates a nearly symmetrical distribution along the tensile direction (180°) and the transverse (90° and 270°). Intensity changes in the tensile direction and in the transverse can represent the major characteristics of orientations in the α'' phase upon tension. Fig. 4(b) shows the intensities of the diffraction from the (1 1 1) and (2 0 0) planes become weaker, while those from the (0 2 0) and (0 2 1) planes become stronger with the increase of tensile stress in the tensile direction.

Fig. 3. (a) Structural evolution in the as-cast Ti–11%Zr–14%Nb–10%Sn sample upon tension detected by *in situ* X-ray diffraction. (b) Two-dimensional (2D) images show the existence of preferred orientations at 900 MPa compared with that at 0 MPa.

By contrast, in the transverse direction as plotted in Fig. 4(c), the diffraction from the (020) and (021) planes becomes weaker, and almost disappeared before the fracture. The major plane is (002), accompanied with the (022), (200) and (113) planes.

3.3. Lattice stain and major texture axes

By noticing how the peak position of each plane is shifting as a function of external stress, one can calculate the lattice strain accumulated on the selected planes. In polycrystalline materials, grains are ideally randomly oriented. Thus, planes, in which atoms are easily moved under the stress, can undergo relatively large deformation. Step by step, these planes tend to orient along the loading direction. According to this scenario, lattice strains accumulated in planes are shown in Fig. 5(a). Three features can be drawn: (1) The elastic deformation exists under the stress below 400 MPa, then plastic deformation begins. By fitting the stress–strain relationship within the elastic regime of single α'' phase in the tensile direction, Young's modulus E is obtained to be about 79 GPa, which approximates to 76 GPa detected by our ultrasonic measurement (Poisson's ratio 0.389 and density 5.31 g/cm³). (2) The lattice strain in the plastic regime generated on three planes in the tensile direction increases as the sequence of (020) < (021) < (022) [the strains of (132) and (041) are not shown, but they are close to the value of (020) plane and less than that of the (021) plane]. The larger lattice stain in (022) planes indicates that the [022] orientation is much closer to the tensile direction. (3) Based on the lattice strain variations on different planes in the transverse direction, the [200] direction is most likely perpendicular to the loading direction.

Fig. 5(b) shows the atomic packing in unit cells of orthorhombic α'' phase and the structural correlations with the tensile stress. It is found that the longest bonds (3.88 Å) are almost along the [022]

Fig. 4. (a) Intensity profiles along the ring pattern scattered by (002) (020) and (111) planes at zero stress and at 700 MPa. (b) Preferred orientation effects changing with the stress in the tensile direction and (c) in the transverse direction.

direction. The bond length in the [020] direction is 2.93 Å, longer than 2.89 Å in the [002] direction. The longer the bond length, the weaker the bond stiffness is. When the tensile stress is along the [022] direction, the angle between [022] and [020] is about 43°, and about 47° between [022] and [002], causing higher partial force in the [020] direction than that in the [002] direction. Moreover, atoms in the [020] direction are closely packed on one plane, while in the [002] direction they are arranged into terraces/steps. Based on the above considerations, we conclude that atoms in the [020] direction move more easily than in the [002] direction. That might be the reason that the b axis increases faster than c axis in the unit cell upon tensile stress, as observed in Fig. 5(c). Since the [200] direction is perpendicular to the [022] direction, and the bond lengths in the [200] direction is about 3.11 Å, it can be understood that atomic pairs along the a axis are more easily compressed. Furthermore, from Rietveld refinements of diffraction patterns in Fig. 3(a), it was found that at the low stress (<400 MPa) the [022]

Fig. 5. (a) Strains located on different planes along the tensile and the transverse direction of the single α'' phase. (b) Structural characteristics in packing of unit cells for the single α'' phase. (c) Changes in lattice parameters of the unit cell for the pure α'' phase with increasing of tensile stress. (d) XRD patterns for the as-cast fractured, 50% rolled and 50% rolled-fractured samples (solid lines). The pattern of the as-cast fractured sample was refined using single α'' phase with [200] orientation (short dotted line).

orientation along the tensile direction is preferred, while the major orientation shifts to the [200] direction together with plastic deformation when the stress is above 400 MPa.

In addition, XRD patterns of the as-cast fractured, 50% rolled and 50% rolled-fractured samples are shown in Fig. 5(d). Three samples have nearly the same diffraction patterns, containing the pure α'' phase with preferred grain orientations. Using Rietveld refinement for the as-cast fractured sample, we found that the lattice constants of this sample changed to $a = 3.0045$ Å, $b = 5.0482$ Å and $c = 4.7535$ Å with preferred orientation of [200]. Due to intensity increase of (021) and decrease of (111), the (111) and (021) peaks become overlapped under plastic deformation. Three major planes are (020), (002) and (021). Upon rolling, the plastic deformation happens. Similar diffraction patterns appear for the three samples, although different stress states from uniaxial tension. This result suggests that similar preferred orientation of single α'' phase can be achieved by grain reorientation under plastic deformations.

4. Conclusions

Using *in situ* X-ray diffraction, no phase transformation is detected upon uniaxial tension in the as-cast Ti–11%Zr–14%Nb–10%Sn sample with a pure orthorhombic α'' phase. Only orientational changes are observed in this phase during tension, leading

to a large change in lattice parameters, i.e., the a axis of the unit cell shrinks while the b and c increase. [022] and [200] directions are inclined to be the major texture axes for preferred orientations in the tensile direction and the transverse, respectively. Unlike β -to- α'' transformation-induced non-linear elastic deformation, the first inflection on the stress-strain curve of the as-cast Ti–11%Zr–14%Nb–10%Sn sample is indeed due to its yielding rather than a non-linear elastic deformation. The sample undergoes significant work-hardening and necking before fracture. By work-hardening treatment, the yield strength of the alloy can be largely improved, e.g., above 900 MPa after cold rolling with about 50% thickness reduction, which is much higher than the yield strength of the as-cast sample.

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