Microstructure and twin behavior of Ti-2Al-2.5Zr during cold pilgering

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Abstract

The microstructure and twin behavior of Ti-2Al-2.5Zr tube during cold pilgering were investigated. During the deformation, the intragranular deformation is not uniform, and the microstructure characteristics are similar to low-cycle shear fatigue. Under alternating stress, the rotation tendency of grain is mainly related to the ratio of circumferential to radial strain. As the mainly primary twin, tensile \{10\text{–}12\langle10\text{–}11\rangle\} is activated in large number, which selection of twinning variant conforms to the Schmidt factor law. However, the selection of the secondary twinning variant is related to the slip system activated by the adjacent grains to accommodate the shear strain caused by the twins. The Schmidt factor of the secondary twin is determined by the slip system opened by the adjacent grains.

1. Introduction

Ti-2Al-2.5Zr is used as a heat transfer tube material for nuclear steam generators due to its excellent specific strength and low stress corrosion sensitivity\[1\]. The mechanical properties and corrosion performance of these tubes under aggressive reactor environment depends on the microstructure of the material\[2, 3\]. In order to improve the in-service reliability, it is necessary to investigate the evolution of deformation behavior during processing.

Much work has been reported on the microstructure evolution of titanium alloys\[4–12\]. Chun investigated the deformation behavior and microstructure evolution under different rolling reduction \[13\]. When the rolling reduction is less than 40\%, the strain is mainly accommodated by slip and twinning. In this stage, tensile \{10\text{–}12\langle10\text{–}11\rangle\} and compressive \{11\text{–}22\langle11\text{–}2\text{–}3\rangle\} twins are mainly produced. But, when the deformation is higher than 40\%, the twins reach saturation. Qin et al studied the initiation and accommodation of primary twins in titanium\[14, 15\]. The result shows that the shear strain caused by twin can be accommodated by adjacent grains initiating slip system, and the Schmidt factor of twin variants determined by different types of slip. These studies are useful for understanding microstructure during rolling, but not much is known about the rotating of grains and twin variants. Especially, there are few studies on the evolution of microstructure and the selection of twin variants under triaxial alternating load.

In this study, space strain vector is used to describe deformation state. The ratio of circumferential strain to radial strain is used to study the grain rotating tendency. And the selection of twin variants during deformation was also studied.

2. Experimental

The initial material used in this work is annealed Ti-2Al-2.5Zr tube with a diameter of 50 mm and a thickness of 9 mm, which chemical composition is given in table 1. The present study deals with the rolling from \(\Phi 50 \text{ mm} \times 9\) mm to \(\Phi 36 \text{ mm} \times 5.5\) mm using a typical LG40H-type mill at the rolling speed 100 \text{ r min}^{-1}, with the feed rate of 3 mm cycle \(^{-1}\). The obtained tapered tube is shown in figure 1. Since the deformation of the tube mainly occurs along the circumferential, axial and radial directions, the equivalent plastic strain is used to describe the amount of deformation in the test. The equivalent plastic strain calculation formula is as follows\[16\]:
Table 1. The chemical composition of Ti-2Al-2.5Zr.

| Element | Al  | Zr  | Fe  | Si  | C   | N   | O   |
|---------|-----|-----|-----|-----|-----|-----|-----|
| Composition (wt.%) | 2.20 | 2.35 | 0.25 | 0.12 | <0.07 | <0.04 | <0.13 |

Among them, $\varepsilon_f$, $\varepsilon_l$, $\varepsilon_r$, $\varepsilon_l$ represents circumferential, axial and radial strain, respectively. Samples with equivalent plastic strain of 0.92, 0.98 and 1.03 were taken at 5cm from the head and tail of tapered tube and at the middle part. Electron Back Scatter Diffraction (EBSD) and Transmission Electron Microscopy (TEM) techniques were used, respectively. The information about grain orientation and microstructure was gained by using FEI Quanta 450 Scanning Electron Microscope with Channel 5 software. For TEM studies, a FEI- Helios G4 CX TEM was used to observe the microscopic configuration of dislocations. The Schmidt factor is calculated by self-made program.

3. Results

EBSD analysis results are shown in figures 2, (a)/(b) represent the initial annealed tube, (c)/(d), (e)/(f) and (g)/(h) represent the sample of $\varepsilon_e = 0.92$, $\varepsilon_e = 0.98$, $\varepsilon_e = 1.03$, respectively. Moreover, the orientation relationship between twins and parent grains is shown in figure 2(i). The different colors in the orientation triangle represent different orientations, red represents (0001) //TD (basal texture); green represents ($-12–10$) //TD; blue represents (01–10) //TD, respectively. The high angle grain boundary defined misorientation is greater than 10° and displayed as a black solid line. Since a large number of sub-grain boundaries are generated during the deformation process, the microstructure is not easy to be observed, so the sub-grain boundaries are not displayed. From figures 2(a) and (b), the structure of initial annealed tube are equiaxed grains, which has no twins. Most of these grains is (0001) //TD orientation, and a small part of the grains are ($-12–10$) //TD and (01–10) //TD orientation. When $\varepsilon_e = 0.92$ (figures 2(c), (d)), the grains are slightly elongated along the rolling direction, and some grains have obvious orientation gradients and twins (blue lines). With the equivalent plastic strain increases to 0.98 (figures 2(e), (f)) and 1.03 (figures 2(g), (h)), the intragranular orientation gradient increases and the twin boundaries become discontinuous. The grain deformation is serious, the intragranular orientation gradient is larger, the twin grain boundary is further broken, and most of the grains turn to (0001) //TD. In addition, figure 2(i) shows the orientation relationship between the twins and the parent grains during the rolling process are mainly {10–12} tensile twins.

Figure 3 is the TEM microstructure morphology image of different plastic deformation samples. Figures 3 (a)/(b)/(c), (d)/(e)/(f), (g)/(h)/(i) represent the sample of $\varepsilon_e = 0.92$, $\varepsilon_e = 0.98$, $\varepsilon_e = 1.03$, respectively. Figure 3 shows that the grains are divided into arc-shaped dislocation regions and dislocation-free regions in all sample, which indicates that the deformation of the material is uneven under the action of the alternating rolling stress. In the initial stage of deformation (figure 3(a)/(b)), not only the grain boundaries are straight and obvious, but also {10–12} tensile twins are induced due to the stress concentration of the grain boundaries. When the
equivalent plastic strain increases to 0.98 and 1.03, the grain deformation is serious. And the grain boundary becomes more curved and fuzzy. With the increase of strain, dislocations continue to increase and accumulate which increased the dislocation density of dislocation region. This result further leads to the increase of the intragranular orientation gradient. And it is also consistent with EBSD analysis.

For hexagonal close-packed structural metal tubes, the radial and circumferential Kearns coefficients play an important role in the properties of the material. Kearns coefficient is calculated by calculating the ratio of {0001} basal plane along a certain direction to determine the proportion of grains in this direction in all grains. F_x, F_y, and F_z represent axial, circumferential, and radial Kearns coefficient, respectively. Its calculation formula is as follows[17]:

$$f = \int_0^{\pi/2} I_\varphi \sin \varphi \cos \varphi \, d\varphi \quad (2)$$

Among them, $\varphi$ is the angle between the $c$-axis of the grain and the specified direction; $I_\varphi$ is the ratio of the $\varphi$ angle between the $c$-axis and the specified direction. Figure 4 is the {0001} pole figure and Kearns coefficient of different equivalent plastic strain samples. It can be seen from figure 4, $F_y$ first decreases and then increases with
the increase of strain, while Fz is the opposite. The change of Kearns coefficient represents the rotation of grains and is also the result of alternating to load. The change of grain rotation trend will be discussed later.

4. Discussion

4.1. Space deformation and kearns coefficient
In the tube rolling process, Q value is an importation parameter which is the ratio of wall reduction to diameter reduction. When Q > 1, the main deformation of the tube is wall reduction. When Q < 1, the main deformation of the tube is diameter reduction [18]. However, the Q value can only describe the overall
deformation mode, and cannot represent the deformation mode and degree of deformation at any position in
the deformation process. Aiming at this problem, the paper adopts the three-dimensional space true strain vector to describe the deformation process, and studies the deformation characteristics and texture changes. In addition to the equivalent plastic strain \( \varepsilon_e \) mentioned above, the strain ratio \( \alpha \) is also introduced to describe the spatially non-uniform deformation mode and degree of deformation during Pilger cold rolling. The calculation formula is as follows [16]:

\[
\alpha = \arctan \left( \frac{\varepsilon_y - \varepsilon_x}{\sqrt{3} \varepsilon_z} \right)
\]

(3)

The true strain vectors of the three principal strain can be used to represent any equivalent plastic strain state of the tube during the deformation process. The direction and magnitude of the true strain vector are directly related to the plastic deformation mode and degree. Therefore, strain ratio and equivalent plastic strain can be used to characterize the direction and magnitude of true strain vector respectively. The strain ratio \( \alpha \) represents the angle at which the true strain vector rotates clockwise around the positive direction of the strain axis, and its variation range is \(-180^\circ\) to \(180^\circ\). According to some results [16], when the strain ratio varies from \(-60^\circ\) to 0, the deformation of the tube is mainly manifested as diameter reduction. When the strain ratio is changed into the range of 0 to \(60^\circ\), the deformation of the tube is mainly to reduce the wall.

Through calculations, it is found that with the increase of the strain, the \( \alpha \) values of \( \varepsilon_c \) are 32°, 1.7°, and \(-5.7^\circ\) respectively. In other words, when \( \varepsilon_c \) is 0.92 and 0.98, the tube which undergoes wall reduction is mainly affected by radial force. when \( \varepsilon_c \) is 1.03, the tube which experiences diameter reduction is mainly affected by circumferential force. The grain rotates under the action of force, and the Kearns coefficient changes with the change of the main force axis. Figure 4 shows that although the strain increases continuously, the change of Kearns coefficient is non-monotonic. The larger the value of \( \alpha \), the more obvious the wall reduction, and the greater the tendency of grains to rotate toward RD. Conversely, the trend toward TD is greater. The changes of \( F_x \) and \( F_y \) are mainly related to \( \alpha \), and the change of \( \alpha \) is the result of the change of the primarily force axis. During the rolling process, as the strain increases, the principal force axis changes continuously. \( F_x \) and \( F_y \) also changes with the change of the force axis, and this trend of change is reflected on \{0001\} pole figure in figure 4.

4.2. Twin behavior during Pilger cold rolling
As an important deformation mode of HCP metals, twins play an important role in the deformation process. The generation of twins not only coordinate and accommodate the strain, but also has an important impact on the formability of the material. The research on the selection law of twinning variants is helpful to better understanding the evolution process of microstructure and provide reference for improving properties and optimizing process. Figure 5 is a common \{10–12\} tensile twin in cold pilgering process, in which the Euler angle of the parent grain is \((165^\circ, 96^\circ, 3^\circ)\), and the Euler angle of the twin is \((94^\circ, 31^\circ, 43^\circ)\). According to the orientation information of the grain, the Schmidt factor of each twin variant is calculated, and the selection rule of the twin variant is analyzed from a geometric view.

During the deformation process, the stress state during rolling can be approximately regarded as the axial tensile stress and the radial compressive stress. In order to further to reduce the errors caused by data collection and theoretical calculations, the calculated Schmidt factor is divided by the maximum theoretical Schmidt factor and normalized. To obtain the normalized Schmidt factor NSF, the calculation formula is as follows:
Figure 6. The position of \{11–22\}–\{10–12\} double twins in the pole figure. (a): inverse pole figure map, (b): point-point orientation distribution diagram, (c): \{0001\} and \{10–12\} pole figure.

Table 2. NSF values of twin variants.

| [10–12] twin | position | \(m\) (tensile) | \(m\) (compress) | NSF     |
|--------------|----------|----------------|----------------|---------|
| (01–12) \{0–111\} | \((-0.121, 1.109)\) | 0.0444 | 0.3789 | 0.531425732 |
| \((-1102)\) \{1–101\} | \((0.754, 0.895)\) | 0.1280 | 0.3346 | 0.568257525 |
| (10–1–2) \{10–11\} | \((-1.516, -0.946)\) | 0.4486 | 0.0037 | 0.504706182 |
| \((-101–2)\) \{-1011\} | \((0.894, -1.698)\) | 0.4104 | 0.0045 | 0.463146828 |
| (01–1–2) \{01–11\} | \((-0.56, -0.672)\) | 0.0608 | 0.3931 | 0.567766384 |
| \((-110–2)\) \{-1101\} | \((0.224, -0.904)\) | 0.1062 | 0.3481 | 0.561130913 |

Figure 7. In-Grain Misorientation Axes distribution map of adjacent grains. (a): grain M, (b): grain C.
Conversely, the twin variants have a higher Schmidt factor. Adjacent grains to activate a slip system with a higher CRSS value, the twin variant have a lower Schmidt factor. Shear stress growth of twins will cause to shear strain on adjacent grains, and adjacent grains will initiate relative slip in order.

The law of twin variants in double twins does not follow the Schmidt factor law. In titanium alloys, the activation and growth of twins will cause to shear strain on adjacent grains. This result indicating that if the formation of a variant requires the M twin position m and grain C are analyzed in figure 6. It can be seen from the point-point orientation distribution diagram in figure 6 that the M (Euler angle: 35°, 174°, 10°) is the matrix, and the C (Euler angle: 87°, 117°, 53°) is primary twin, T is the second twin (Euler angle: 50°, 37°, 2°). The NSF of the primary twin and the secondary twin are calculated as shown in table 3.

Corresponding to the location of {11–22} in figure 6 and the NSF in table 3, it can be seen that the selection law of twin variants in double twins does not follow the Schmidt factor law. In titanium alloys, the activation and growth of twins will cause to shear strain on adjacent grains, and adjacent grains will initiate relative slip in order to accommodate the strain. The in-grain misorientation axis (IGMA) distribution characteristics of the adjacent grain M and grain C are analyzed in figure 7. It shows that the non-prismatic slip with a higher critical resolved shear stress (CRSS) value is initiated in the grains. This result indicating that if the formation of a variant requires adjacent grains to activate a slip system with a higher CRSS value, the twin variant have a lower Schmidt factor. Conversely, the twin variants have a higher Schmidt factor.

5. Conclusions

(1) When the strain ratio is 0–60°, the Fz increase, and the grains rotate in the radial direction. When the strain ratio is −60°–0, Fy increases and the grains rotate in the circumferential direction.

(2) The selection of a primary twin variants is following Schmid factor law. But the selection of the secondary twinning variant is mainly related to the slip system initiated by the coordinated strain in the adjacent grains. The more easily the slip system in adjacent grains starts, the greater the Schmidt factor of the twin is, and vice versa.

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| Table 3. NSF value of double twins. |
|---|
| {11–22} twin position | m (tensile) | m (compress) | NSF |
| (11–2–2) (−1–12–3) | (1.025, −0.033) | 0.4684 | 0.4715 | 0.98668458 |
| (−21–2–2) (2−1–12–3) | (−0.667, −0.833) | 0.1315 | 0.4592 | 0.61435917 |
| (1−2–2) (−12–12–3) | (−0.539, 1.127) | 0.0663 | 0.9343 | 0.47771192 |
| (−1−12–2) (11−2−3) | (−1.191, 0.159) | 0.4160 | 0.0315 | 0.476579535 |
| (−12−1–2) (−12–1–23) | (0.402, −0.892) | 0.0885 | 0.4844 | 0.594470358 |
| (2−11–2) (−21–13) | (0.609, 0.988) | 0.1618 | 0.4279 | 0.614396057 |
| {10–12} twin position | m(tensile) | m(compress) | NSF |
| (10–12) (−1011) | (1.6, 0.628) | 0.2943 | 0.1248 | 0.611431951 |
| (01–12) (0−111) | (1.507, −0.513) | 0.2890 | 0.2039 | 0.756079983 |
| (10−12) (10–11) | (−0.425, 0.473) | 0.3444 | 0.1746 | 0.770158499 |
| (01−12) (01–11) | (−0.406, −0.260) | 0.3434 | 0.2613 | 0.934793619 |
| (1−10–2) (1−101) | (−1.041, 1.127) | 0.3945 | 0.1007 | 0.692183937 |
| (−110–2) (−1101) | (−0.925, −0.911) | 0.3949 | 0.0931 | 0.678147723 |

\[

\text{NSF} = \frac{\cos \alpha \cos \beta}{(\cos \alpha \cos \beta)_{\max}} + \frac{\cos \gamma \cos \delta}{(\cos \gamma \cos \delta)_{\max}} \quad (4)

\]

α, β are the angles between the axial tensile stress and the twin shear direction and the normal of the twin plane, and γ, δ are the angles between the radial compressive stress and the shear direction and the twin plane normal.

The NSF of {10–12} twin variants are shown in table 2. Corresponding to the position of {10–12} in figure 5, it can be determined that the twin is (−1102)/(1−101) twins. From table 2 that the twin has the largest NSF, which indicates that the selection of primary {10–12} tensile twins in the cold pilgering process follows the Schmidt factor law. When \( \varepsilon = 0.92 \), a {11–22} compression twin and a {11–22}−{10–12} double twins was also found, as shown in figure 6. It can be seen from the point-point orientation distribution diagram in figure 6 that the M (Euler angle: 35°, 174°, 10°) is the matrix, and the C (Euler angle: 87°, 117°, 53°) is primary twin, T is the second twin (Euler angle: 50°, 37°, 2°). The NSF of the primary twin and the secondary twin are calculated as shown in table 3.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files). Data will be available from 21 June 2021.

Declarations of interest

none

Author Contributions

Wujun arranged experiments, performed tests and wrote the paper; Mazhaodandan carried out rolling experiment; wanghui helped revise and proofread the language; Wang li and Liaojinjing provide the alloys and they are project administrators.

All authors read and contributed to the manuscript.

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References

[1] Oryshchenko A S, Kudryavtsev A S, Mikhailov V I and Leonov V P 2012 Titanium alloys for shipbuilding and nuclear power engineering Inorg. Mater. Appl. Res. 3 497–506
[2] Rba A, Cce B and Mg C 2019 Irradiation creep and growth of zirconium alloys: a critical review J. Nucl. Mater. 521 167–244
[3] Choi Y, Shin E J and Inoue H 2006 Study on the effect of crystallographic texture on the corrosion behaviour of pilgered zirconium by neutron diffraction Phys. B 385–386 529–31
[4] Xu-hu Zhang, Bin Tang, Xia-lu Z, Hong-choa K, Jin-shan Ll and Zhou Lian 2012 Microstructure and texture of commercially pure titanium in cold deep drawing Transactions of Nonferrous Metals Society of China 22 496–502
[5] Liu N et al 2018 Microstructure and textural evolution during cold rolling and annealing of commercially pure titanium sheet Transactions of Nonferrous Metals Society of China 28 1123–31
[6] Bozzolo N, Chan L S and Rollett A D 2010 Misorientations induced by deformation twinning in titanium J. Appl. Crystallogr. 43 596–602
[7] Huang Z W et al 2019 Slip, twinning and twin-twin interaction in a gradient structured titanium Mater. Charact. 149
[8] Wu H C et al 2016 Rolling-induced face centered cubic titanium in hexagonal close packed titanium at room Temperature. Scientific Reports 6 24370
[9] Kim W J, Yoo S J and Lee J B 2010 Microstructure and mechanical properties of pure Ti processed by high-ratio differential speed rolling at room temperature Ser. Mater. 62 451–4
[10] Bozzolo N et al 2007 Microstructure and microtexture of highly cold-rolled commercially pure titanium J. Mater. Sci. 42 2405–16
[11] Nasiri-Abarbekoh H et al 2012 Effects of rolling reduction on mechanical properties anisotropy of commercially pure titanium Mater. Des. 34 268–74
[12] Zeng Z, Jonsson S and Roven H J 2009 The effects of deformation conditions on microstructure and texture of commercially pure Ti Acta Mater. 57 5822–33
[13] Chun Y B, Yu S H, Seoniati S L and Hwang S K 2005 Effect of deformation twinning on microstructure and texture evolution during cold rolling of CP-titanium Mater. Sci. Eng., A 398 209–19
[14] Qin H and Jonas J 2014 Variant selection during secondary and tertiary twinning in pure titanium Acta Mater. 75 198
[15] Qin H, Jonas J, Yu H B, Brodusch N, Gauvin R and Zhang X Y 2014 Initiation and accommodation of primary twins in high-purity titanium Acta Mater. 71 293 (SCI)
[16] Hla B et al ‘Texture evolution and controlling of high-strength titanium alloy tube in cold pilgering for properties tailoring - ScienceDirect J. Mater. Process. Technol. 279
[17] Deng S et al 2019 Selection of deformation modes and related texture evolution in Zircaloy-4 during one pass cold pilgering Materials Science and Engineering 764 1–10
[18] Lebensohn R A et al 1996 Measurement and prediction of texture development during a rolling sequence of Zircaloy-4 tubes J. Nucl. Mater. 229 57–64