A study on microstructural evolution and detwinning behavior of Ti–3Al–2.5V cold-rolled tube during annealing

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Abstract
Near alpha titanium alloys have been widely used in the aerospace and aircraft industries owing to their high specific strength, low-density and excellent corrosion resistance. Ti–3Al–2.5V is a typical near alpha titanium alloy that was usually used in the form of a tube in aircraft due to its excellent cold workability. In the present study, the microstructural evolution and detwinning behavior of cold-rolled Ti–3Al–2.5V alloy tubes during annealing were investigated. Upon annealing, the microstructure evolution strongly depends on the annealing temperature and holding time, which play important roles in the kinetics of the static recovery and recrystallization. In addition, there are two independent detwinning mechanisms involved during annealing. The first one is that detwinning occurs along the twin shear direction at grain boundaries, resulting in a shortening effect. The second one is that detwinning occurs in the center of twins, resulting in a thinning effect of twins. The understanding of the detwinning behavior and microstructural evolution during annealing helps with the tube processing and their applications in aircraft.

1. Introduction

Near α-titanium alloys have been widely used in the aerospace and aircraft industries owing to their high specific strength and excellent corrosion resistance (Gerd and Williams 2007). Ti–3Al–2.5V is a typical near α titanium alloy which can be used in the form of a tube in aircraft due to its good workability (Kumar and Gupta 2009, Li et al 2012, Liu et al 2014). The hexagonal closed packed (hcp) α-phase during deformation can activate various slip systems such as basal, prismatic and pyramidal, (first- and second-order) (Zeng et al 2009). Furthermore, twinning plays an important role in plastic deformation of hcp α-titanium (Hama et al 2014, Yan et al 2018). During cold deformation, twinning can accommodate plastic deformation by shear deformation (Christian and Mahajan 1995, Hong et al 2010), and also change the crystallographic orientation of α-phase which can promote more slip activations (Huang et al 2019).

In the case of cold-deformed Ti–3Al–2.5V alloys, the microstructure contains a large number of slip bands (high-density of dislocations) and deformation twins, as well as residual stress. In order to reduce the residual stress and modify their mechanical properties, a fully recrystallized structure free of residual stress is generally required by using annealing treatments. However, the microstructural evolution related to detwinning of cold-rolled Ti–3Al–2.5V alloy during annealing has rarely been investigated (Li et al 2017). Detwinning mechanisms in face-centered cubic metals have been frequently studied based on the dislocation mechanism (Gu et al 2014, Lee et al 2014, Szczepa et al 2016). However, detwinning process of titanium alloys has rarely been studied. Only a few studies have concentrated on detwinning in body-centered cubic beta titanium alloys, such as Ti-Nb (Qu et al 2013) and Ti–Mo (Gutierrez-Urrutia et al 2017). Furthermore, no systematic research has ever been carried out on the detwinning behavior of hcp Ti alloys, which is very important to the microstructural evolution in a cold deformed hcp Ti alloy during annealing.

Therefore, the present study aimed at studying the microstructural evolution and detwinning behavior of cold-rolled Ti–3Al–2.5V alloy during annealing. The objective is to understand the detwinning behavior during annealing and its effect on the microstructure evolution in the Ti–3Al–2.5V alloy tube. Optimal annealing treatments were provided for tube applications.
2. Material and experimental

The material used in this study was a Ti–3Al–2.5V (wt.%) alloy. An ingot of this alloy was prepared by triple vacuum arc remelting by using sponge titanium, aluminum foil and Al-55V master alloy. The ingot was first forged above the beta-transus temperature and then forged in the α + β phase field to a billet. The billet was hot extruded to a hollow tube (Φ 86 mm). The final tubes with dimensions of 6.35 mm in diameter and 0.508 mm in thickness were produced by using multi-pass two-roll cold rolling processing (LG-10H rolling mill). The tubes were annealed at temperatures from 300 to 750 °C and held for 1 to 5 h. The planes normal to the rolling direction were prepared and then mechanically polished for microstructural observation. Electron backscatter diffraction (EBSD) was used to observe the microstructures. EBSD scans were performed on a field emission gun scanning electron microscopy (JSM-7001F) equipped with an EDAX EBSD detector. The grain sizes of the samples were measured based on the EBSD analyses.

3. Result and discussion

3.1. Microstructural evolution during annealing at low-temperature

EBSD IPF maps in figure 1 show the microstructures of the samples annealed at low temperatures (300 °C–600 °C) for 5 h. It is observed that the samples annealed at low temperatures exhibit a recovered microstructure containing deformation-induced orientation gradients. Deformation twins are also observed as indicated by arrows in figures 1(a)–(d). With increasing annealing temperature, however, more recrystallized grains that are nearly free of orientation gradients can be observed. For example, a few recrystallized grains are observed in the sample annealed at 600 °C for 5 h, as shown in figure 1(d). The number of grains containing orientation gradients or deformation twins also decreases.

![Figure 1. EBSD maps of the samples after annealing at low temperatures for 5 h: (a) 300 °C (b) 400 °C (c) 500 °C (d) 600 °C.](image-url)
3.2. Microstructural evolution during annealing at high temperature

EBSD maps in Figure 2 show the microstructures of the samples after annealing at high temperatures and for different time. After annealing at 650 °C for 3 h, the microstructure is still a recovered structure as shown in Figure 2.

Figure 2. EBSD maps of cold-rolled Ti–3Al–2.5V alloy tube after annealing at high temperatures: 650 °C for (a) 3 h, (b) 5 h, and 700 °C for (c) 3 h, (d) 5 h, and 750 °C for (e) 3 h, (f) 5 h.
A considerable amount of recrystallized grains is observed whereas a small number of orientation gradients can be observed. In addition, some deformation twins still exist as indicated by arrows in figure 2(a). With increasing annealing time to 5 h, the volume fraction of recrystallized grains increases, however, it is still a partially recrystallized structure that consists of both recrystallized grains and orientation gradient grains as shown in figure 2(b). Upon annealing at 700 °C, the higher temperature accelerates the recrystallization process as shown in figures 2(c) and (d). In the case of 700 °C for 3 h, it is clearly observed that the number of recrystallized grains increases accompanied by the disappearance of orientation gradient grains. In addition, a considerable number of twins can be observed as indicated in figure 2(c). With increasing annealing time to 5 h, the microstructure is a fully recrystallized structure without showing any twins as shown in figure 2(d). The average grain size is measured to 8.6 ± 3.0 μm based on the EBSD analysis.

In the case of annealing at 750 °C for 3 and 5 h (figures 2(e) and (f)), the samples consist of equiaxed grains with average grain sizes of 8.8 ± 3.0 μm and 9.5 ± 3.4 μm, respectively. Very few twins and orientation gradients can be observed, indicating that nearly full recrystallization occurs at this annealing temperature. Note that the grain size in the sample of annealing at 750 °C for 3 h is similar to that of annealing at 700 °C for 5 h. It could be attributed to the fast grain growth at a higher temperature (Ghosh et al 2017). The fast grain growth leads to an inhomogeneous grain size distribution. For example, some grains have a size of approximately 20 μm whereas some have a finer size, less than 5 μm as shown in figures 2(d) and (e).

Based on the microstructural observation, annealing treatments of 700 °C for 5 h and 700 °C for 5 h, which result in a fully recrystallized structure with an appropriate grain size, are suggested for the actual tube application of this alloy.

### 3.3. Detwinning behavior

Figure 3 shows the detwinning process of the samples during annealing at 700 °C. After annealing at 700 °C for 1 h (figure 3(a)), the microstructure is very similar to a deformed structure. Only a few {10–12} twins can be
observed in the sample owing to the large plastic deformation. The microstructure still has a high density of geometrically necessary dislocations (GNDs) as seen from the KAM map in figure 3(d). In addition, the sample annealed at 700 °C for 1 h contains a high fraction of low angle grain boundaries (LAGBs) as shown in figure 3(g), reaching to 43.2%. This indicates that the annihilation of the dislocations formed during the cold rolling is insufficient in the microstructure after annealing for 1 h at 700 °C. Therefore, there are no significant occurrence of both the dislocation annihilation and detwinning process. After annealing for 3 h at 700 °C, however, dislocation annihilation becomes significant and some recrystallized grains can be observed in the microstructure as shown in figure 3(b). Meanwhile, most of the grains in figure 3(b) have a similar size to the deformed grains in figure 3(a), indicating that the microstructure is dominated by recovery during annealing from 1 to 3 h. The recovery behavior reduces the total fraction of GNDs as shown in figure 3(e), causing a decrease in the fraction of LAGBs as shown in figure 3(h). Furthermore, {10–12} twins can be clearly observed in the annealed microstructure and its fraction reach to 28.4%. As seen in figure 3(b), most of the twins exhibit an irregular shape with serrated twin boundaries or terminated within grains. This indicates that the detwinning process occurs under this condition. Upon annealing at 700 °C for 5 h, dislocations and twins disappeared in the sample as shown in figure 3(c), indicating of a fully recrystallized microstructure. There are no twin boundaries remained in the sample so that the detwinning process completely finished in the sample after annealing at 700 °C for 5 h. In addition, the misorientation of the grain boundaries is also uniform as shown in figure 3(f).

In summary, in the case of annealing at high temperatures, the time needed for the dislocation annihilation is shorter than that of the detwinning. The former occurs at the initial stage of annealing whereas the detwinning dominates the microstructure evolution at the later-stage of annealing.

3.4. Detwinning mechanism

The detailed detwinning behavior is shown in figure 4. Based on the pole figures, the blue zones in the grains (figures 4(a) and (b)) are the matrix, and two variants of {10–12} twins occur in the grains. The matrixes formed
by detwinning originate simultaneously from various places of the grain boundary, and develop to the twin, as shown in figure 4(a). The twins are then divided into some parallel bands by the newly formed matrix. The twins are swallowed gradually by the matrix boundary migration as shown in figure 4(b). In figure 4(c), the center of one twin band transforms into the matrix, which leave some dislocations in the place of detwining as indicated by red arrows in figures 4(c) and (d). This process is similar to the detwining in Mg alloy as reported in (Li et al 2014). In summary, there are two independent mechanisms for the detwining process. First, the detwining occurred initially at the grain boundaries as shown in figure 4(a), where the detwining direction is along the twinning shear direction. This results in a shortening of twins against the grain boundaries at one side or both sides. Second, the detwining occurred in the center area of twins, of which direction is perpendicular to the twin shear direction as shown in figure 4(b). This mechanism results in a thinning effect of twins.

4. Conclusion

We have investigated the microstructural evolution and detwining behavior of cold-rolled Ti–3Al–2.5V alloy tubes during annealing at different temperatures for various time. The following conclusions can be drawn based on the results:

(1) Dislocations disappear at the earlier stage of annealing whereas the detwining dominates the microstructure evolution at the later stage of the annealing process.

(2) There are two independent detwining mechanisms. The first one occurs at grain boundaries, resulting in a shortening effect of twins. The second one occurs in the center of twins, resulting in a thinning effect of twins.

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