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Twinning-Induced Formation of Nanostructure in Commercial-Purity Titanium

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Abstract. In the present work the influence of various parameters on formation of nano- or ultrafine-grained structure in commercial-purity titanium during large deformation was quantified using TEM and EBSD. The beneficial effect of twinning on the kinetics of microstructure refinement in titanium was revealed. It was shown that deformation twinning (and therefore nanostructure formation) can be intensified via decrease in temperature, increase in the initial grain size and decrease in the impurities content. The minimum grain size at which twinning can still operate in commercial-purity titanium was determined to be ~1μm. It was shown that rolling to a thickness strain of 93% at -196°C resulted in the formation of a microstructure with a grain/subgrain size ~80 nm.

Introduction

In contrast to many other metals and alloys with cubic crystalline lattice (BCC or FCC) which deform mainly by slip alone, the microstructure evolution of a commercial-purity (CP) titanium with the low-symmetry HCP lattice is associated with two deformation mechanisms: slip and twinning.

As one of the main deformation modes, twinning in titanium typically occurs during the early stages of deformation. Being high-angle in nature, twin boundaries transform from well-defined coincident-site-lattice-type boundaries into arbitrary ones due to subsequent interaction with lattice dislocations [1], thereby refining the microstructure considerably. Twin boundaries can have a marked influence on the evolution/refinement of microstructure during further straining; i.e., following the saturation of twinning. However, the nature of such microstructure evolution has been investigated to only a limited extent [2, 3, 4].

Prior work has suggested that twinning is one of the main factors enabling the formation of a very fine microstructure in CP titanium [2 – 5]. For example, a microstructure with a grain size of 100-200 nm was attained during plane-strain rolling at room temperature to a true thickness strain $\varepsilon_{th} \approx 2.6$ [3]. Microstructure evolution in CP titanium during such cold rolling was found to be associated with twinning at the initial stage of deformation and then the formation of deformation-induced high-angle boundaries [3]. Decrease in temperature resulted in the intensification of twinning and considerable microstructure refinement [4]. The aim of the present work is to quantify the influence of various parameters on twinning and to establish the effect of twinning on the microstructure refinement during large deformation of CP titanium.

Material and Procedures

A 4mm thick slab of CP titanium VT1-0 (Ti-balance; impurities in wt.% less than: 0.2 Fe, 0.1 Si, 0.07 C, 0.04 N, 0.12 O) was used in the present investigation. In the as-received condition, the plate had a homogeneous, equiaxed microstructure with an average grain size of 15μm. Also specimens with the mean grain size of 1, 7 and 30 μm were obtained by controlling annealing of ultrafine-grained preforms produced by the isothermal multiaxial forging [6] at 480, 600, 700 and 800°C, respectively to check the effect of initial grain size on twinning. The influence of chemical composition on twinning was studied using CP titanium Grade4 (Ti-balance; impurities in wt.% less than: 0.5 Fe, 0.1 C, 0.05 N, 0.4 O).

Samples measuring $4\times10\times30mm^3$ were rolled unidirectionally in few passes at room and liquid nitrogen (-196°C) temperatures using a fixed rolling speed of 30 mm/s to a total thickness strain of 93%. In order to evaluate the effect of strain path some specimens were rolled with changing rolling direction by 90° each next pass to the total reduction of 30%.

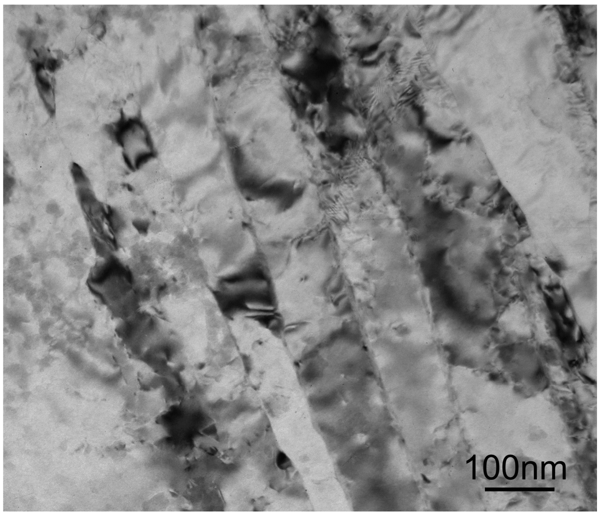
The microstructure at the mid-thickness rolling plane of each deformed sample was determined using a JEOL JEM-2100FX transmission electron microscope (TEM) and electron-backscatter-diffraction (EBSD) conducted in a Quanta 600 scanning-electron microscope. The border between low-angle boundaries (LABs) and high-angle boundaries (HABs) was taken to be 15°. Grain-boundary misorientations below 2° were excluded from the data analysis.

Results and Discussion

Microstructure Evolution during Cold Rolling. Rolling at room temperature (20°C) to a thickness strain of 15% resulted in the appearance of twins and a considerable dislocation density (Fig. 1a). The twins were distributed inhomogeneously in the microstructure, often appearing as clusters in various places. With increasing strain dense dislocation walls and pileups were also observed (Fig. 1b). Further deformation to ε=60% gave rise to a cellular microstructure with a high dislocation density (Fig. 1c). The boundaries of the cells were rather wide and loose. The size of the cells varied over a wide range from a hundred nanometers to a few micrometers. Individual subgrains with a size of 100-200 nm and thin clear boundaries were also observed. Rolling to ε=93% at room temperature led to a considerable refinement of the microstructure and simultaneously to an increase in dislocation density (Fig. 1d). The size of cells was found to be approximately 200 nm. In addition, grains or subgrains with clear thin boundaries and a mean size of ~150 nm were also observed in the microstructure.

Along with EBSD analysis [3] TEM investigation of cold-rolled titanium revealed three stages of microstructure evolution. At the first stage the microstructure evolved mainly via twinning. Being one of the main modes of deformation, twinning refined the microstructure considerably (Fig. 2a). Further microstructure evolution was associated with development of substructure within initial grains and twins. At this second stage the rate of microstructure refinement with strain decreased noticeably. Thus, the second stage may be envisaged as an incubation period for the accumulation of a sufficient level of dislocation density for the subsequent formation of high-angle boundaries. The third, and final, stage in microstructure evolution (Fig. 2a) occured at ε above ~40% and was most probably associated with the formation of high-angle deformation-induced boundaries and gradual decrease in grain size.

Microstructure evolution during cryo-rolling at liquid nitrogen temperature (-196°C) was qualitatively similar to that found at room temperature [4]. However, cryo-rolling to ε = 93 resulted in formation of the microstructure with smaller grain/subgrain size ~80 nm (Fig. 2a). In addition the kinetics of the microstructure refinement at lower temperature was found to be noticeable faster (Fig. 2a). The latter phenomenon was most likely associated with more intensive microstructure refinement at the initial stage of deformation, i.e. at the stage where twinning was active. Since twinning can promote formation of ultrafine-grained or nanostructure in CP titanium, it is important to determine the major factors and to quantify their influence on deformation twinning.



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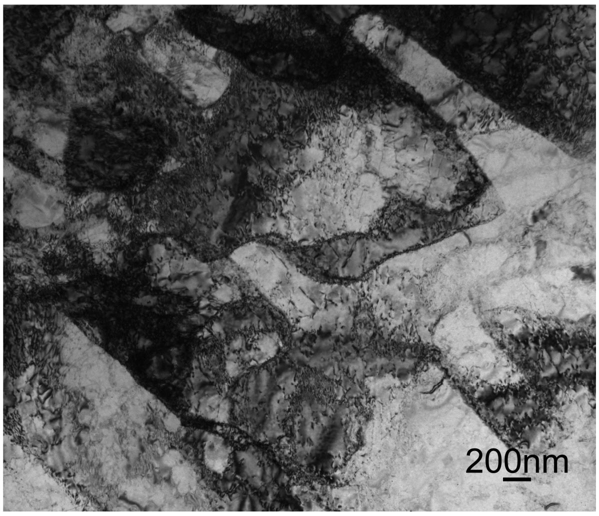

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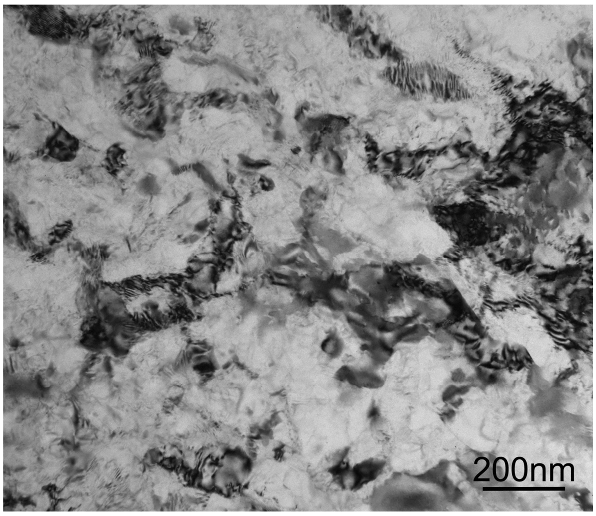

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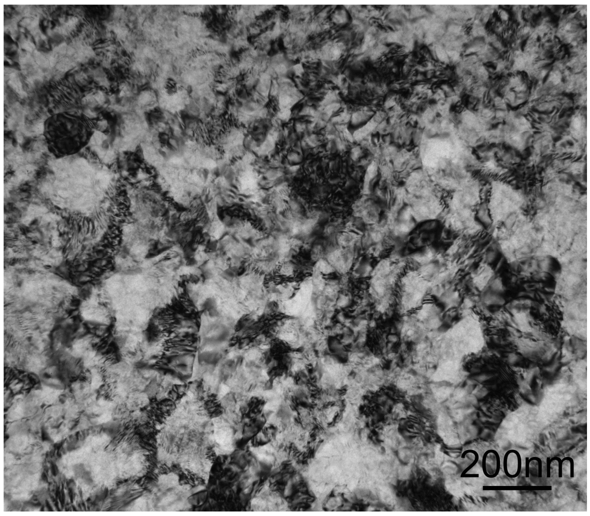
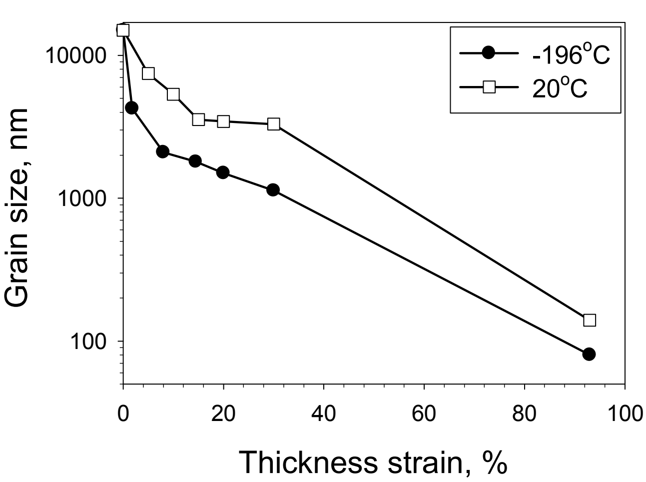


d

Figure 1 - Microstructure of CP titanium after cold rolling at T=20°C to thickness strain (a) ε=15%, (b) ε=30%, (c) ε=60%, (d) ε=93%.



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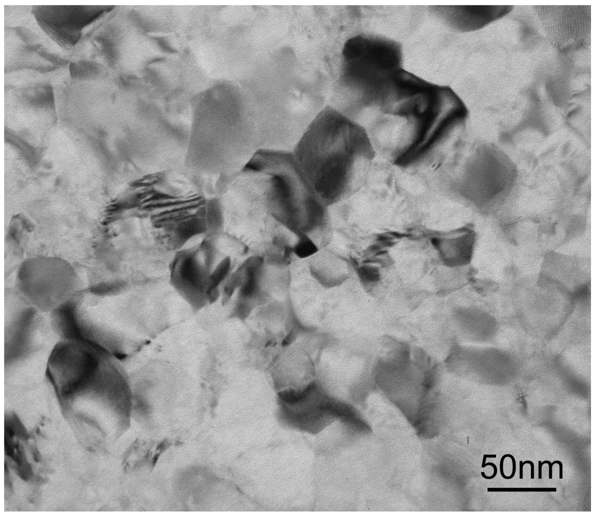

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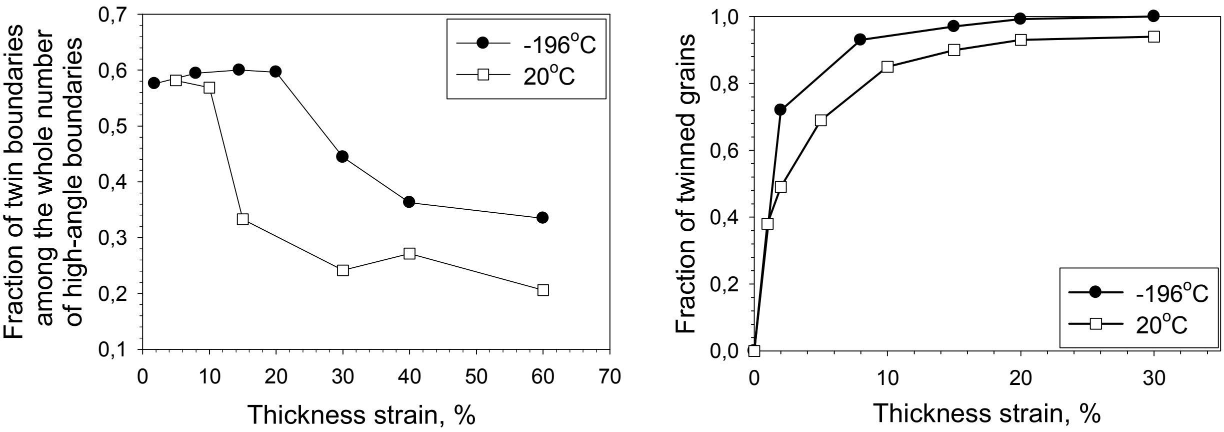
Figure 2 - (a) grain size of CP titanium as a function of strain during rolling at 20°C or -196°C and (b) microstructure of CP-Ti after cold rolling at T=-196°C to thickness strain ε=93%.

Factors Intensifying Twinning. The intensity of twinning in HCP metals is controlled by a number of factors [7]. Among them deformation temperature (or strain rate), chemical composition and grain size have the most pronounced effect. However to use those factors for formation of nanostructure in CP-titanium their quantitative examination is needed.

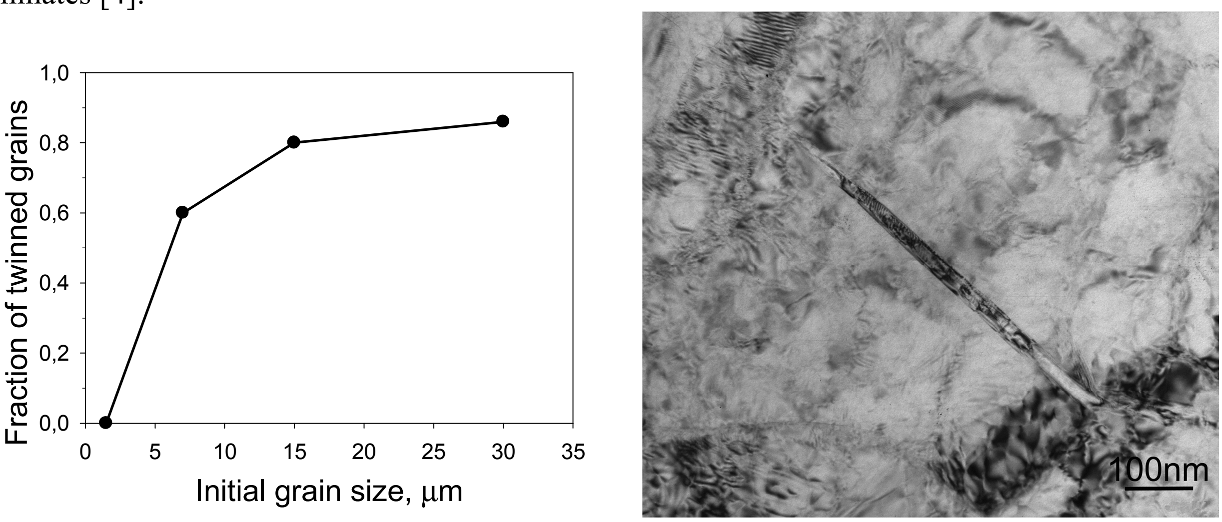
The beneficial influence of temperature decreasing on the twinning intensity was noted above (Fig. 2b). This effect is described in literature and is associated with a weak dependence (if at all) of the critical resolved shear stress for twinning on temperature compared to that for slip [8]. Detailed study by using light microscopy and EBSD analysis have shown that decrease in temperature from 20°C to -196°C resulted in: i) approximately twofold increase in the length of the twinning stage (Fig. 3a), ii) increase in the fraction of twinned grains (Fig. 3b) and iii) smaller twin thickness [4]. Formation of a very fine and rather homogeneous microstructure due to twinning gave rise faster kinetics of microstructure refinement in general.



a b

Figure 3 - Twin characteristics in CP titanium as a function of strain at 20°C and -196°C: (a) EBSD-determined fraction of high-angle boundaries corresponding to twins and (b) fraction of twinned grains determined by optical microscopy.

Another important factor influencing the intensity of twinning is initial grain size. With a decrease in grain size, the critical resolved shear stress for twinning increases more rapidly than that for slip [8]. As the grain size is reduced below a critical value slip becomes preferable and twinning terminates [4].



a b

Figure 4 - The influence of the initial grain size on the fraction of twinned grains in 10% cold rolled CP titanium (a) and the microstructure of titanium with the initial grain size of 1μm after rolling to 10% (b).

The investigation of the microstructure of CP titanium with different grain size rolled at room temperature to ε=10% have shown that activity of twinning increased with increase in grain size reaching maximum fraction of twinned grains at grain size of 15÷30μm. Meanwhile almost no twinning was observed in CP titanium with grain size 1μm (Fig.4a,b). This observation for the critical grain size below which twinning is impossible (~1μm) is in good agreement with the results obtained earlier (0.9μm) [4]. It worth noting that the thickness of twins also increases with increasing grain size that results in a coarser twinned microstructure.

Variation in the chemical composition of titanium can also influence considerably on the twinning intensity. Interstitial elements increases twinning stress thereby suppressing twining [7]. Indeed, comparison the fraction of twinned grains for the program material (VT1-0) and that for CP titanium Grade4 with higher percentage of impurities shows much more intensive twinning in the former case (Fig 5a). From a practical point of view this result shows the importance of the optimal choice: the material for producing ultrafine-grained or nanostructure should have some impurities to be strong enough but a high level of impurities suppress twining thereby retarding microstructure refinement.

Strain path had a moderate effect on the twin intensity. Comparison of multipass unidirection rolling and rolling with changing rolling direction by 90° shows ~5% increase in the fraction of twinned grains. This effect is obviously associated with the activation of new twin families when the rolling direction changes. The contribution of strain path change is obviously dependent on the initial texture of the material and should be taken into account during ultrafine-grained structure formation.

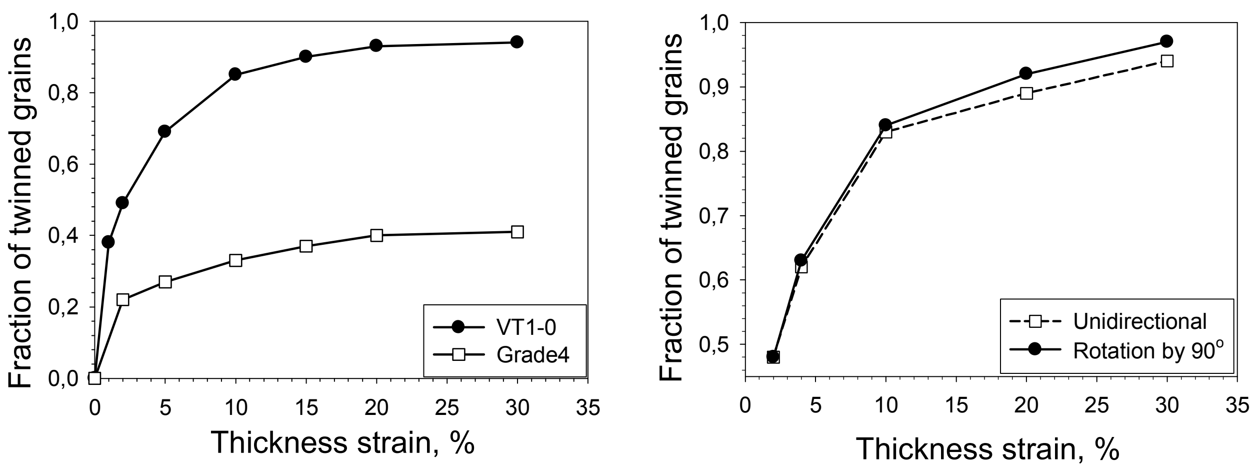


Figure 5 - EBSD-determined fraction of high-angle boundaries corresponding to twins: (a) in CP titanium VT1-0 and Grade4 and (b) in CP titanium VT1-0 during cold rolling via different strain paths.

Therefore the results obtained in the present work show a considerable influence of twinning on the formation of nano- or ultrafine-grained structure in CP titanium during cold rolling. To intensify the formation of the ultrafine-grained structure due to twinning, a CP titanium with relatively low level of impurities and with grain size ≥15μm should be used. Decrease in deformation temperature and change in strain path at the initial stages of working results in further intensification of twinning and thereby formation of microstructure with grain size of 80nm.

Summary and Conclusions

The evolution of microstructure in commercial-purity titanium during cold- and cryorolling to a thickness strain of 93% was quantified using TEM and EBSD techniques. The following conclusions can be drawn from this work:

1. The microstructure evolution of CP titanium during cold rolling is sequentially associated with twinning, formation of substructure and formation of deformation-induced high-angle boundaries. Deformation twinning promotes microstructure refinement.
2. The intensity of deformation twinning strongly depends on temperature, initial grain size and chemical composition of CP titanium.
3. Rolling to a thickness strain of 93% results in the formation of a microstructure with a grain size of ~80 nm or 200 nm at -196°C or 20°C, respectively.

Acknowledgements

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