Twin induced Strain Hardening, Grain Fragmentation, and Texture Evolution during Cold Compression of CP-Ti

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Abstract. This work is a systematic investigation of the twin induced strain hardening behavior, grain fragmentation, and texture evolution during titanium's cold compression. The compression tests were stopped at intermediate strains for quantitative microscopy by electron backscattered diffraction. The microstructural examination indicates the evolution of \(\{10\bar{1}2\}\) extension twins (ET), \(\{1\bar{1}22\}\) contraction twins (CT) and the interaction between them. The strain hardening rate and its derivative plot could be corroborated with the microstructural features and distinguishes regions of slip and twin dominating zones. The texture evolution indicates that dislocation slips were active apart from deformation twins during deformation. Texturally hard ET evolves, broadens, and interacts in texturally soft grains at low and intermediate strains. The ET regions are suitable for the nucleation of texturally soft CT with further strain. During deformation, ET-ET \(~56.8^\circ\langle 1010\rangle\), CT-CT \(~77^\circ\langle 1010\rangle\), ET-CT \(~87^\circ\langle 4\bar{2}21\rangle\) interactions boundaries and CT-ET \(~44.5^\circ\langle 5\bar{1}4\rangle\) double twins develop.

Keywords: Titanium; Strain hardening; Grain fragmentation; Extension twinning; Contraction twinning;

1. Introduction

Commercially pure titanium (CP-Ti) is widely used in various aviation, chemical, sports, and biomedical applications due to its high specific strength, corrosion resistance, and biocompatibility [1]. Many researchers have improved strength and ductility in low-density hexagonal close-packed (HCP) materials via microstructure and texture engineering through thermo-mechanical processing [2-4]. An in-depth understanding of deformation behavior is required for all-around improvement in ambient mechanical properties. Owing to the HCP crystal structure with a low \(c/a\) ratio, Ti possesses complex deformation behavior compared to cubic crystals [5,6]. To accommodate the imposed stress at ambient temperature, deformation twins get activated subsequently after micro-yielding via two independent prismatic slip to satisfy the Von-Mises criteria [7]. The activation of each slip and twin system requires a critical resolved shear stress [8]. Mainly, two types of twin systems \(\{10\bar{1}2\}\) extension (ET) and \(\{1\bar{1}22\}\) contraction twins (CT) occur in Ti at ambient temperature [5]. The evolution of twins occurs in sequences such as ET-ET or CT-CT-ET, depending on the initial texture and direction/type of external loading [9]. The activation of ET and CT reorients the parent matrix by \(~85^\circ\) about \(\{1\bar{1}20\}\) and \(~63.5^\circ\) about \(\langle 1010\rangle\), respectively. Each twin system has six twin variants, and their activation is governed by the angular relation between crystallographic orientation and loading direction. In the separate investigation by Chouhan and Shina et al. [8,10] showed the three different stages of twinning, namely nucleation, propagation, and lateral thickening during plastic deformation. It is reported that activation of multi twin variants and the intersection of propagating twin fronts enhance the grain boundary strengthening [10]. Hence, the grain fragmentation, crystallographic orientation changes, and textural hardening/softening are accelerated mainly by deformation twinning instead of dislocation slip...
in Ti. There are few investigations on the grain refinement via twin-twin intersection, splitting of twins during consecutive deformation, and twin growth in CP-Ti.

In this work, uniaxial compression tests on CP-Ti were carried out at ambient temperature to observe ET and CT sequential evolution and their effect on strain hardening behavior, texture evolution, and microstructure refinement. Multiple samples were collected at the intermediate strains for quantitative microstructure and texture analysis. The various grains were cropped from deformed samples. Here, we present the grain by grain analysis to link the twin and its morphology, such as lamella, intersecting, and lateral broadening with strain hardening behavior and flow curve feature followed by texture evolution.

2. Materials and Method

Compression tests of hot extruded grade-2 Ti bar with length to width ratio of ~1.5 as per ASTM E09 were performed at a strain rate of 0.005s\(^{-1}\) on the Instron 1344 Universal Testing Machine. Samples were collected at various intermediate true strains from ~0.05 to 0.69 to carry out quantitative microscopy by electron backscattered diffraction (EBSD) in the AURIGA field emission gun scanning electron microscope. In this regard, the samples were prepared by mechanically polished out up to 1\( \mu \)m SiC abrasive paper, followed by electropolishing in LectroPol-5 (STRUTS) at a voltage of 18V for 15s at 25°C via electrolytic solution containing 700ml Ethanol, 120ml distilled water, 100ml 2-butoxyethanol, 80ml HClO\(_4\). The EBSD data were analyzed using ATEX software [11].

3. Results and Discussion

3.1. Flow Curve and strain hardening Behavior

The microstructure of the as-received material in figure 1(a) shows twin free equiaxed grains with an average grain size of ~ 20\( \mu \)m. During uniaxial compression, a significant grain size reduction takes place. Figure 1(b) shows the variation in grain size with increasing true strain. The grain size reduces to ~ 5\( \mu \)m after ~ 0.69 true strain. The experimental flow curve is shown in figure 1(c). A typical ‘S’ type flow curve depicts that the imposed plastic strain is accommodated by a profuse amount of deformation twins along with dislocation slip [7, 12]. The markers show the intermediate strains at which the samples were collected for microstructure and texture analyses.

![Figure 1](image1.png)

Figure 1. (a) Image quality map of the as-received material, (b) Average grain size for CP-Ti concerning true compressive strain, (c) Flow curve with markers representing the intermediate strains at which samples were collected for analysis, and (d) Strain hardening rate and it is derivative vs true stress curves for uniaxial compression along extrusion direction of CP-Ti.
The activation of \{10\overline{1}2\} \{10\overline{1}\overline{1}\} type ET [5,13] orient the part of parent grain by \(\approx 85^\circ\) about \{11\overline{2}0\} towards compression direction (CD). The formation of ET increases the hard orientations in the microstructure. It could be further corroborated with the strain hardening rate (SHR) and derivative of the SHR plot with respect to true stress, as shown in figure 1(d). After the micro yielding (at \(\varepsilon \approx 0.06\)), the decrease in the SHR denotes the material is deformed by slip along with deformation twinning. Whereas, at \(\varepsilon \approx 0.06\) derivative of SHR became zero and reached its minima at \(\varepsilon \approx 0.10\). From the \(\varepsilon \approx 0.06\) to 0.10, the twins are nucleated, and its activity increases. After that, an increase in strain (at \(\varepsilon \approx 0.22\)) the rise in strain hardening (figure 1c) and SHR (figure 1d) in a material due to the change in crystallographic orientation via ET. Therefore, in the strain range \(\varepsilon \approx 0.06\) to 0.17, the material is mainly deformed by deformation twinning. A horizontal line corresponds to zero value of derivative of SHR, divides the slip and twin dominating strain range, i.e., the strain ranges \(\approx 0.06\) to 0.17 and \(\approx 0.40\) to 0.50 are denoted the twin domination. The detailed description, along with the microstructure evidence, is reported by Chouhan et al. [8] in 2019.

3.2. Texture Evolution

Figure 2(a-d) shows the evolution of deformation texture using inverse pole figure along compression direction (CD). The as-received CP-Ti had axially symmetric \{10\overline{1}0\} \{1\overline{1}2\overline{0}\} prismatic fiber texture with a maximum multiple of a random distribution (MRD) of \(\approx 3.15\) along the compression direction (CD) shown in figure 2(a). After, uniaxial compression of \(\approx 0.05\) (figure 2b), basal texture fiber was initiated and intensified after the strain of \(\approx 0.22\) to MRD \(\approx 4.9\) MRD (figure 2c). With the further increase in the strain \(\approx 0.69\), the basal fiber shifts with an increase in its angular spread form \{10\overline{1}7\} \{1\overline{1}2\overline{7}\} axially symmetric fiber texture (figure 2d) along CD. The dotted line shows the position of the axially symmetric fiber in figure 2d.

Figure 2. Texture representation via Inverse Pole Figure along compression direction for the (a) initial and after the effective strains of (b) \(\approx 0.05\), (c) \(\approx 0.22\), and (d) \(\approx 0.69\)

3.3. Effect of Extension Twin

The effect of deformation-induced ET, four different individual grains containing prominent ET were cropped and shown in figure 3(a-d). The point to point misorientation across twin domain and angular inverse pole figure \(85^\circ\{1\overline{1}2\overline{0}\}\) are shown in figure 3(e-f). The unit cell of these grains and the ET domains are also superimposed in these figures. ET regions have orientated the basal poles towards compression direction. These ET domains have a higher Taylor factor and, therefore, texturally harder than the parent matrix [8,12]. The compression along \{10\overline{1}0\} and \{1\overline{1}2\overline{0}\} axes are deformed by the activation of two and four ET variants [12]. The evolution of one and two ET variants are shown in figure 3(a,b). Figure 3(c,d) shows the parent lattice's ideal crystal orientation for the evolution of two parallel ET variants. These grains are cropped from \(\approx 0.11\) and \(\approx 0.22\) deformed samples. ET lamellae
are broadened and consumed the parent matrix with further deformation. The newly formed ET domains have lower dislocation density concerning the matrix region. After the effective strain of $\approx 0.10$, twin domain growth occurred by strain-induced twin boundary migration toward the higher strain energy region of the parent matrix. Basinski-type hardening may take place due to the sessile nature of dislocation inside twin domains [8].

![Figure 3. Gran by grain analysis in the strain range of ~0.05-0.22 (a) Nucleation of primary extension twin, (b) ET-ET interaction, (c) ET lamellae formation, (d) Broadening of ET and remnant of the parent matrix, (e) point to point misorientation of ET, and (f) ~85° angular inverse pole figure along with IPF color key code triangle.](image)

### 3.4. Effect of Contraction Twin

The microstructure features formed due to CT are shown in figure 4. Compressive deformation along the (0002) axis is accommodated by activation of CT. Figure 4(a) shows that the matrix's basal axis (in green color) is oriented towards CD with a small angular deviation.

![Figure 4. Grain by grain analysis from ~0.11-0.69 (a) formation of secondary contraction twins at strain range ~0.15 – 0.22, (b) CT-CT interaction, (c) nucleation of tertiary ET lamellae inside CT domain, (d) ET-CT interaction, (e) point to point misorientation of CT, and (f) microstructure after ~0.69 effective strain.](image)
CT are nucleated in the primary ET domains and confirmed from remnant (in red color). The (0002) axis is orientated normal to the CD. Thus (red color) initial parent matrix deformed via activation of primary ET and oriented the (0002) toward the CD. Therefore activation of the CT in primary ET is designated as secondary CT. These are \((1\bar{1}2\bar{2})(1\bar{1}2\bar{3})\) type CT [5,13], reorients the matrix by ~63.5° about \((10\bar{1}0)\) axis confirmed via point to point misorientation and angular IPF shown in figure 4(e). CT's evolution could increase the texturally soft orientation in microstructure, confirms from the superimposed crystal lattices. It reorients the basal pole away from the CD. Further, an increase in the strain the interaction of CT-CT, ET-CT, and tertiary ET formed fixed angle axis pairs: CT-CT ~77°(10\bar{1}0), ET-CT ~87°(4221) interactions boundaries and CT-ET ~44.5°(5\bar{1}4\bar{0}) are shown in figure 4(b-d). The CT has evolved after the ~0.15 along with basal and pyramidal \((c + a)\) type slip systems [8].

4. Conclusions

Quasi-static uniaxial compression of Ti at ambient temperature is performed to investigate the role of deformation twinning on the strain hardening rate, texture, and microstructure evolution. Grain by grain analysis was carried out, and the strain hardening response is correlated at different strain stages. The major findings of this study are listed below:

1. As per Initial texture and loading direction, ET-CT-ET type sequential twinning was formed along with dislocation slip.
2. Slip and twin domination zones could be identified by the strain hardening rate curve and its derivative.
3. \((10\bar{1}2)\) \((10\bar{1}\bar{1})\) type ET increases the texturally hard domains; whereas, \((1\bar{1}2\bar{2})(1\bar{1}2\bar{3})\) CT evolved inside primary ET domains and produced texturally softer orientations.
4. With the increase in strain, the various twin reactions such as ET-ET ~56.8°(1\bar{1}0\bar{1}), CT-CT ~77°(10\bar{1}0), ET-CT ~87°(4221) interactions boundaries and CT-ET ~44.5°(5\bar{1}4\bar{0}) double twins develop

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