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Hardening and softening analysis of pure titanium based on the dislocation density during torsion deformation

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ABSTRACT

The hardening and softening phenomena during torsion deformation are studied based on the Taylor dislocation model for pure titanium. The hardening and softening phenomena are observed through the hardness analysis during micro-indentation test and micro-hardness test. Besides, the variations of indentation size also verify the existence of hardening and softening phenomena during torsion. The variations of geometric necessary dislocations (GNDs) and statistic store dislocations (SSDs) state that the positions of high dislocation density and low dislocation density correspond to the positions of hardening and softening. The results from the microstructure, grain boundaries evolution and twins analysis indicate the twins play an important role in appearance of hardening and softening phenomena. The appearance of hardening and softening phenomena are attributed to the combination of different slip systems and twinning systems combining with the Schmid Factor (SF) analysis and the transmission electron microscope (TEM). The appearance of hardening and softening phenomena can be explained by the Taylor dislocation theory based on TEM analysis.

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1. Introduction

The pure titanium has been drawn extensive attention to aircraft and biomedical fields due to its excellent mechanical properties, high chemical stability, excellent corrosion resistance and biocompatibility [\[1](#page--1-0),[2\].](#page--1-0)

A higher plastic strain can be accumulated during torsion deformation compared with the tension and compression deformation, so torsion deformation is combined with other deformation modes to refine grains and improve strength [\[3\]](#page--1-0), such as high pressure torsion (HPT) $\left[4\right]$ and twist extrusion (TE) $\left[5\right]$. Therefore, torsion deformation has attracted many scholars' attention due to its enormous importance to improve the comprehensive mechanical properties of materials. Many scholars studied that the grains were refined and the micro-hardness was improved gradually with increasing torsion turns for pure copper [\[6](#page--1-0)–[8\].](#page--1-0) Meanwhile, the grain size became smaller and smaller and the micro-hardness increased gradually from rod center to the edge on transverse section. The hardening of deformed rods was attributed to the dislocation strengthening. Non-uniform distribution of microstructure and micro-hardness on transverse section was related to gradient strain introduced by torsion deformation. The similar phenomena were observed through analyzing the twisted pure

<http://dx.doi.org/10.1016/j.msea.2016.06.046> 0921-5093/© 2016 Elsevier B.V. All rights reserved. aluminum rods [\[9\]](#page--1-0). The compressive strength increased gradually while tensile strength exhibited a non-monotonous change with the increase of torsion angle for hexagonal close-packed (HCP) AZ31 manganese alloy $[10]$. Meanwhile, the hardening and softening phenomena are observed on transverse section during torsion using the HPT method for HCP metals [\[11,12\]](#page--1-0). The appearance of softening phenomenon during torsion deformation indicated that the strengthening mechanisms were attributed to many factors.

The plastic deformation of metals is dominated by dislocation movements. Researches had shown that the hardening of deformed materials was attributed to dislocation proliferation during torsion deformation for pure copper and pure aluminum $[6,9]$. Guo pointed out that dislocation proliferation lead to hardening, but the generation of lamellae twin and the weakening of extrusion texture would result in appearance of softening during torsion deformation $[10]$. Xin indicated that the grains subdivided by twins could largely affect strain hardening behaviors during the plastic deformation of magnesium alloy [\[13\]](#page--1-0). The occurrence of dynamic recovery, formation of shear bands would lead to the decrease of dislocation density and appearance of softening for pearlitic steel during torsion [\[14\]](#page--1-0). Many papers indicated that deformation twins played an important role in the hardening and softening mechanisms of pure titanium during plastic deformation [\[15](#page--1-0)–[17\]](#page--1-0). The mechanical properties of materials were affected by the different twins, which were determined by the relationship among dislocation slip, twinning and fracture for HCP metals

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[\[18,19\]](#page--1-0). Such as the compression twin {10-11} of magnesium alloy reduced the flow stress, improved the plasticity, and played as the softening role $[20]$. However, the tension twin ${10-12}$ played a hardening role in magnesium alloy [\[21\]](#page--1-0). Moreover, the interaction between slip and twinning had an important effect on hardening and softening for HCP metals [\[18,22,23\]](#page--1-0).

The dislocation can be classified into the geometry necessary dislocations (GNDs) and statistic store dislocations (SSDs) based on the Taylor dislocation model [\[24](#page--1-0),[25\]](#page--1-0). M. R. Staker stated that the strain gradient increased with the increase of strain. GNDs and SSDs increased with increasing strain gradient, and then resulted in apparent hardening effect [\[26\]](#page--1-0). The GNDs were directly related to the curvature of plastic deformation [\[27\]](#page--1-0), and the permanent geometrical changed at the surface of specimen [\[28\].](#page--1-0) However, the SSDs evolved from random trapping processes and its density increased through proliferation or interaction during the plastic deformation [\[29\]](#page--1-0). Many papers have stated that GNDs can be characterized by micro-indentation test [\[24](#page--1-0),[27\]](#page--1-0), and the SSDs can be characterized by Taylor dislocation model according to the strain gradient plastic theory [\[28,30\]](#page--1-0).

Numbers of studies have concentrated on the strain hardening characteristics of pure titanium during compression deformation and tension deformation [\[31](#page--1-0)–[36\]](#page--1-0). They indicated that deformation hardening characteristics were divided into three stages in simple compression and tension of pure titanium. However, scarce special research has been conducted on the appearance of hardening and softening during torsion for HCP metals although the hardening and softening phenomena are observed. On this background, this paper analyzes the hardening and softening phenomena of pure titanium during torsion deformation based on the dislocation density theory, and the explanations are provided according to the experimental results and finite element simulation.

2. Experiments and methods

The pure titanium Ti-GR2 (ASTM) rods are subjected to torsion deformation. Detailed dimensions about the specimen are shown in Fig. 1. The torsion test is conducted on the XC-10 wire torsion testing machine with the torsion speed of 30r/min. The rods are designed to twist 3.14, 6.28, 9.42, 12.56, 15.70, 18.84 rad respectively at room temperature, corresponding to the maximum equivalent plastic strain of 0.091, 0.181, 0.270, 0.361, 0.443, 0.526 at the specimen surface. The mechanical properties of deformed specimens are obtained based on uniaxial tensile test. The uniaxial tensile tests are carried out at room temperature with a constant strain rate of 10^{-3} s⁻¹ using Instron 3382 (Instron Inc., USA).

The micro-indentation test is conducted at a constant load of 400mN and rate of 9.6841 mN/s using MCT-W501 in order to eliminate the micro-indentation scale effect [\[37\].](#page--1-0) Eight indentations distribute equally on a circle, and the radii of the circles are 0.6, 1.2, 1.8, 2.4, 3.0 mm, as shown schematically in [Fig. 2](#page--1-0)(a). And the indentation depth is the average of the eight values in order to ensure the reliability of test data. [Fig. 2](#page--1-0)(b) shows the loading and unloading curves of micro-indentation, and the micro-indentation hardness can be obtained using the method of Oliver and Pharr [\[27\].](#page--1-0) Besides, based on the Taylor dislocation model, the GNDs and

SSDs can be characterized according to micro-indentation experiment [\[38\].](#page--1-0) The framework of GNDs has been given a physical basis for strain gradient dependent material behavior. The variation trends of micro-hardness from center to surface on transverse section can be explained from the perspective of GNDs and SSDs [\[25\]](#page--1-0). The degree of hardening and softening of deformed rods can be reflected by the variation of maximum indentation depth. Meanwhile, micro-hardness test is conducted at the corresponding positions in [Fig. 2](#page--1-0)(a) at the load of 100 g and dwell time of 15 s using HXP-1000TM tester. As the hardening and softening are caused by plastic deformation, the finite element simulation is conducted to study the plastic strain variation during torsion.

Microstructures are characterized along the transverse direction (TD) of deformed rods by an optical microscope (OLYMPUS/ PMG3). The chemical etchant used on the specimens is a solution of 5 ml HNO₃, 10 ml HF, and 85 ml H₂O. The indentation surface morphology is observed by a scanning electron microscope (SEM, MIRA3 TESCAN). The specimens with the gauge dimensions of Φ 6 mm \times 3 mm are prepared for electron back scattering diffraction (EBSD). The samples are first mechanically polished and then electropolished in a solution of 60 ml perchloric acid, 300 ml methanol and 640 ml n-butyl alcohol at 30°C using a DC power supply with 30 V and time 30 s. The variation of SF and distribution characteristic of the grain boundary are obtained by the electron back scattering diffraction (EBSD) in order to analyze the mechanics of hardening and softening. The coordinate system and the selective local areas are shown in Fig. $2(c)$. X0, Y0 and Z0 refer to the radial direction (RD), TD and normal direction (ND). The A area is close to the specimen center and B area is close to the specimen surface. Finally, TEM samples are punched from the billets and mechanically polished to thicknesses of $70 \mu m$. After perforation, TEM samples are polished by use of a twin-jet polishing unit with a solution of 5% perchloric acid, 35% butanol and 60% methanol at an applied potential of 40 V and a temperature of 233 K.

The stress state of the sample transforms from elasticity to the elastic-plasticity with increasing torsion angle during torsion. When the surface shear stress achieves the shear yield stress, ω_ρ can be calculated using the following formula

$$
\omega_e = \frac{\tau_s}{\mu R} \tag{1}
$$

where ω_e is torsion angle of unit length at the elastic limit state (deg), $\tau_{\rm s}$ is shear yield stress (MPa), μ refers to shear modulus of pure titanium (μ =40.067 GPa), R is radius of the specimen $(R=3$ mm).

The critical shear yield stress can be calculated based on the Mises yield criterion.

$$
\tau_s = \frac{1}{\sqrt{3}} \sigma_s \tag{2}
$$

where σ_s is yield stress, $\sigma_s = 428.61$ MPa. Then $\tau_s = 247.465$ MPa, and ω can be calculated according to the Eq. (1).

$$
\omega = \omega_e \mathcal{L} = \frac{\tau_s \mathcal{L}}{\mu \mathcal{R}} \times \frac{180}{\pi} = \frac{247.465 \times 60}{40670 \times 3} \times \frac{180}{\pi} = 6.976^{\circ}
$$
 (3)

where ω is plastic deformation torsion angle, L is scale length $(L=60$ mm).

The plastic deformation will occur after the specimen is twisted a small angle from Eq. (3) . It can be seen from Fig. $9(b)$ that volume fraction of elastic deformation area is very small and can be neglected. So the equivalent strain can be regarded as the equivalent plastic strain. The relationships between flow stress, dislocation density, indentation depth and torsion radian, torsion radius are built in [Fig. 3](#page--1-0) with the purpose of analyzing the hardening and Fig. 1. Dimensions of torsion specimen (Unit is mm). softening phenomena [\[4,24](#page--1-0),[27,28,30](#page--1-0),[39](#page--1-0)-[45\]](#page--1-0). The letters in [Fig. 3](#page--1-0)

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