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Mechanical annealing under low-amplitude cyclic loading in micropillars

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ABSTRACT

Mechanical annealing has been demonstrated to be an effective method for decreasing the overall dislocation density in submicron single crystal. However, simultaneously significant shape change always unexpectedly happens under extremely high monotonic loading to drive the pre-existing dislocations out of the free surfaces. In the present work, through in situ TEM experiments it is found that cyclic loading with low stress amplitude can drive most dislocations out of the submicron sample with virtually little change of the shape. The underlying dislocation mechanism is revealed by carrying out discrete dislocation dynamic (DDD) simulations. The simulation results indicate that the dislocation density decreases within cycles, while the accumulated plastic strain is small. By comparing the evolution of dislocation junction under monotonic, cyclic and relaxation deformation, the cumulative irreversible slip is found to be the key factor of promoting junction destruction and dislocation annihilation at free surface under low-amplitude cyclic loading condition. By introducing this mechanics into dislocation density evolution equations, the critical conditions for mechanical annealing under cyclic and monotonic loadings are discussed. Low-amplitude cyclic loading which strengthens the single crystal without seriously disturbing the structure has the potential applications in the manufacture of defect-free nano-devices.

1. Introduction

Reducing the dislocation density at the submicron scale is important for obtaining multifunctional micro-devices, such as high-strength (Bei et al., 2008), improved dielectric properties (Alpay et al., 2004) and electrical and thermal conductivities (Watling and Paul, 2011). There is a considerable body of evidences that dislocation density tends to decrease in small submicron single crystal when subjected to monotonic loading (Greer and Nix, 2006; Shan et al., 2008; Uchic et al., 2009). This process named as "mechanical annealing" (Shan et al., 2008) is usually attributed to the massive dislocation escape from free surface (Greer, 2006; Greer and Nix, 2006; Wang et al., 2012). Generally, the occurrence of this phenomenon depends on the sample size (several hundred nanometers) and applied stress level (several GPa). Smaller sample size corresponds to higher attractive image force and more significant surface annihilation, while higher stress means that more dislocation junctions can be broken. However, the relative high applied monotonic loading (as high as several GPa) also

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triggers a significant amount of dislocation multiplication. The decreasing of dislocation density requires the annihilation of both the pre-existing and the multiplied dislocations. This leads to evident changes of the specimen shape, which is not anticipated in the practical applications. So it will be very attractive if there exists a new loading method (e.g., with relative low-amplitude) to drive out the dislocations and at the same time keep the specimen shape without significant change.

This raises the question whether the non-monotonic loading performed with relative low-amplitude can disengage the complex pre-existing dislocation structures without causing significant dislocation multiplication. Generally, under cyclic loading condition, dislocation accumulation by forming pattern structures such as well-ordered veins and walls (Aifantis, 1987), is usually expected even at the micron scale (Zhang et al., 2006). Whether the defect healing can be observed if the external size further decreases to several hundred nanometers? A convenient way to verify the aforementioned idea is to adopt in-situ TEM experiment and three-dimensional discrete dislocation dynamic (3D-DDD) simulation method. 3D-DDD has been proved to be an effective method to reveal the dynamic evolution of dislocations in small crystals (Csikor et al., 2007; Devincre et al., 2008; El-Awady, 2015; El-Awady et al., 2009; Groh and Zbib, 2009). In the current work, the cyclic loading tests with relative low-peak stress are carried out by both experiments and 3D-DDD simulations on Al micro-pillar with the diameter of a few hundred nanometers. It is somewhat surprising to observe that the initial high dislocation density significantly decreases within cycles. Particularly noteworthy is that there is no such pronounced shape change compared with relative high stress monotonic loading. This phenomenon makes it exhibit great promise for applications in obtaining high-strength crystal with low density dislocations.

Naturally this intriguing observation raises several questions to us. Firstly, how could the low cyclic stress contribute to the decline of dislocation density? How will cyclic loading affect the dislocation annihilation and multiplication process? In addition, both the line tension model (Dupuy and Fivel, 2002) and atomic level analysis (Rodney and Phillips, 1999) indicate that the failure of dislocation junction often requires high enough applied stress. How can dislocation junctions be destroyed without high-stress under cyclic loading? Previous studies mainly focus on the stability of dislocation junction under monotonic loading (Dupuy and Fivel, 2002; Picu and Soare, 2010). Little attention is paid on the cyclic stability of dislocation

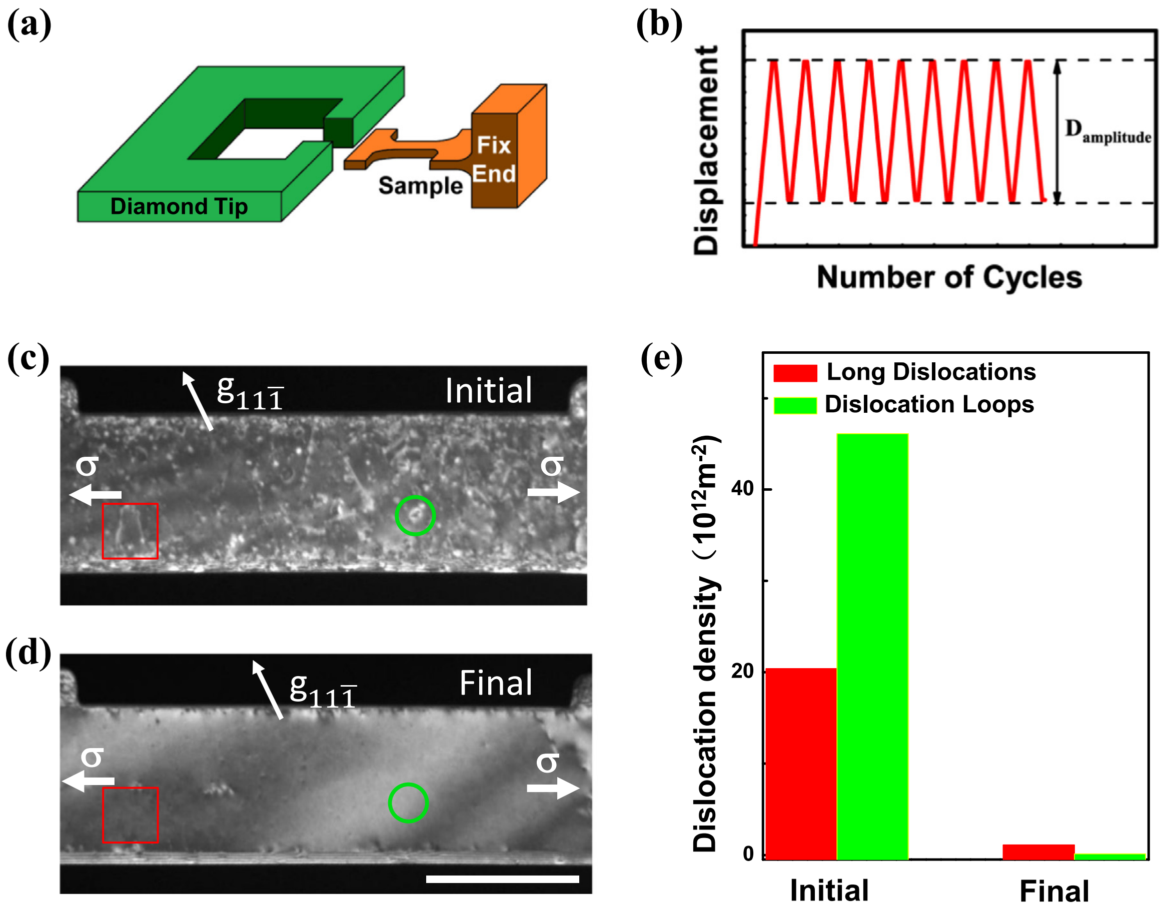

Fig. 1. A typical example of mechanical annealing of defected Al single crystal through low-amplitude cyclic straining. (a) Schematic of in situ cyclic tension loading test inside a TEM. (b) Cyclic loading displacement program. (c) Dark-field TEM image of as-fabricated Al single crystal. (d) Dark-field TEM image of Al single crystal after mechanical annealing. (e) The densities of the two types of dislocations before and after mechanical annealing. The scale bar in (d) represents 500 nm (Wang et al., 2015). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)