Available online at www.sciencedirect.com

SciVerse ScienceDirect

Acta Materialia 61 (2013) 1421–1431

Acta MATERIALIA

www.elsevier.com/locate/actamat

The effect of crystal orientation on the stochastic behavior of dislocation nucleation and multiplication during nanoindentation

I. Salehinia, S.K. Lawrence, D.F. Bahr *

School of Mechanical and Materials Engineering, Washington State University, Pullman, WA 99164, USA

Received 9 June 2012; received in revised form 6 November 2012; accepted 12 November 2012
Available online 20 December 2012

Abstract

Current methods to measure the theoretical shear strength of metals using nanoindentation often present a stochastic view of the applied stresses needed to nucleate dislocations. In this study a combination of molecular dynamics simulations and experimental nanoindentation tests were used to explore the coupled effects of indenter size, crystallographic orientation, and the presence of internal structural defects on the resulting distribution of loads at the onset of plastic deformation in face-centered cubic metals. In this case stacking fault tetrahedra have been selected as a representative structural, rather than chemically distinct, defect. The sensitivity of the crystal to the presence of internal structural defects depends strongly on its crystallographic orientation. Simulations of indentations in the presence of a stacking fault tetrahedron show the highest reduction in the pop-in load for the (1 1 1) orientation, while experimentally the effect of orientation is dependent on the size of the indenter used, and hence the volume of material under stress. The simulations suggest that indenting near a defect can cause small, sub-critical events to occur which then lead to a large "pop-in" at higher loads, and thus the first event observed experimentally may not correspond to the first plastic deformation event. As internal defects are almost inevitable in materials, a defect-based model can be used to explain the stochastic pop-in loads in nanoindentation tests.

Keywords: Dislocation nucleation; Molecular dynamics; Stacking fault tetrahedron; Indentation size effect; Crystallographic orientations

1. Introduction

Nanoindentation is widely used as a tool to sample the mechanical responses of small volumes of materials [1]. The transition from elastic to plastic deformation, often referred to as the onset of or incipient plasticity, manifests itself in nanoindentation load–displacement curves either as an excursion in displacement under load control or a load drop in displacement control tests [2]. However, it is not always true that a yield point at this "pop-in" event is indicative of only incipient plasticity. Kiely et al. [3] noted that this event can be a result of events such as the sudden propagation to the surface of dislocations already nucleated in the material. They also showed that these major events happen after some minor events, including the nucleation and also propagation of dislocations under the indenter. Similar observations of "pop-in" after plastic deformation events have been noted by other researchers [4,5]. Minor events are almost unobservable in experimental load–displacements curves, however, molecular dynamics (MD) simulations have shown that nucleation, propagation and entanglement of dislocations are possible below the observable pop-in point [3,6].

One important observation in nanoindentation testing is an increase in hardness for either smaller indentation depths or smaller indenter radii [7,8]. Shim and co-workers [9] and Salehinia et al. [10] reported a different type of indentation size effect of nanoscale indenters based on the nucleation of dislocations at the onset of plasticity (pop-in) for nanometer size indenters. Morris et al. [11] also carried out a study on the indenter size effect for indenter radii in the range 115 nm–700 μm. Using the cumulative event

* Corresponding author. Present address: Materials Engineering, Purdue University, West Lafayette, IN 47907, USA.
E-mail address: dfbahr@purdue.edu (D.F. Bahr).

1359-6454/$36.00
http://dx.doi.org/10.1016/j.actamat.2012.11.019

frequency distribution of pop–in events in load–displacement curves they proposed a defect density (mainly dislocations) based model to explain the observed behavior on changing the indenter size. In that study a narrow distribution of shear stress at the onset of plasticity for small radii, a wide distribution for medium radii and again a narrow distribution of shear stresses for radii of 17.5 μm and larger supported the effect of structural defects on the onset of plasticity. Although their proposed model works very well for radii larger than 1.5 μm, for smaller radii there were significant deviations from the experimental data. Several researchers have used atomistic and multiscale simulations to address the effect of indenter size on the mechanical properties of different types of materials (these studies often use tip radii in this smaller size regime) [12–14].

As the deformed volume of a material decreases the effect of crystal anisotropy on the mechanical response intensifies. Several studies have shown that for face-centered cubic (fcc) crystals the elastic moduli for the (1 1 1) and (1 1 0) orientations are about the same, and larger than that for the (1 0 0) orientation [12,15–21]. The majority of the literature examining the effect of orientation on pop-in behavior has shown that the pop-in loads in the (1 1 1) orientation are usually largest and those in the (1 0 0) are usually smallest [10,17–21]. Several works have tried to elucidate the stochastic nature of the pop-ins in nanoindentation tests for several crystal orientations. For instance, Li et al. [22] performed theoretical calculations and experiments to study the distribution of pop-in loads for nanoindentations on NiAl single crystals in several crystal orientations. For an indenter radius of 580 nm their results showed a narrow distribution of pop-in loads for the (1 1 1) and (1 1 0) orientations, and widely stochastic values for the (1 0 0) orientation. They linked the stochasticity of the data for the (1 0 0) orientation to the larger stressed volume for this orientation compared with those for (1 1 1) and (1 1 0), suggesting a higher chance of internal defects such as dislocations being influenced by the indenter stress field, hence contributing to the pop-in statistics. Recently a combined experimental and modeling study by Wang et al. [23] explored the orientation effect on the stochastic distribution of pop-in loads, and observed that for indentations in Cu single crystals in the (1 0 0), (1 1 0) and (1 1 1) orientations using indenters of 164 and 53 nm there was little to no effect of orientation on the yield behavior. Using Weibull statistics they suggested that bulk defects are more likely responsible for the displacement bursts for the (1 0 0) orientation, while surface defects are behind the pop-ins for the (1 1 1) and (1 1 0) orientations. MD simulations of nanoindentation tests then showed that for the (1 1 1) and (1 1 0) orientations the nucleation of dislocations was followed by a sudden load drop, while the (1 0 0) orientation exhibited homogeneous dislocation nucleation with only a small load drop, followed by the formation of a dislocation lock under the indenter tip.

Since as the test size decreases the effect of structural defects on the mechanical response of a crystal is enhanced, exploring different defects could be a reasonable way to tailor their possible effects on the onset of plasticity in nanoindentation experiments. Among different structural defects in single crystals, surface defects, such as steps [24–26], and internal defects, such as point defects (vacancies, interstitials, and impurities), and their clusters, like stacking fault tetrahedra (SFT) [27], have been of interest in the literature. Calculations have shown that the critical stress for dislocation nucleation at surface steps can be half that on a flat surface [24]. Salehinia and Bahr [27] studied the effect of vacancies, self-interstitial atoms, and SFT on the pop-in load in nanoindentation of (1 1 1) copper single crystals and showed up to a 50% reduction in the applied stress at the onset of plasticity in the presence of an SFT under the indenter. As structural defects in materials are common, to obtain a better understanding of the observed stochasticity in pop-in loads we believe simulations should include defects.

In this paper experimental and modeling studies are combined to explore the coupled effects of indenter size, crystallographic orientation, and the presence of structural defects on the distribution of pop-in loads during nanoindentation in commercially pure nickel, while corresponding MD simulations have been carried out for copper. The choice of two different materials was made to emphasize the strengths of each technique. The available embedded atom model (EAM) potentials for Cu are more reliable for dislocation nucleation than those for Ni, while experimentally Ni exhibits a thin oxide layer and less of an effect of solute oxygen than Cu. The load–depth curves and deformation mechanisms from MD simulations will be used to explain the different distribution of pop-in loads for different indenter radii and crystal orientations in the experiments.

2. Methods

2.1. Modeling

Molecular dynamics simulations were carried out using the LAMMPS code (http://lammps.sandia.gov) [28] applying the EAM potential developed by Mishin et al. [29] to model nanoindentation of copper with (1 0 0), (1 1 0) and (1 1 1) crystallographic orientations. The size of the simulation box was $20 \times 20 \times 20$ nm containing around 680,000 atoms. The side and bottom faces were fixed and the top face was allowed to move. The simulation box was large enough that the effect of boundaries on the onset of plasticity was negligible. The indenter was modeled as a rigid sphere of 15 nm radius with a quadratic repulsive potential. The time step was 1 fs and the indenter speed was 10 m s^{-1}. To verify that this velocity was not creating an artificial construct a velocity of 1 m s^{-1} was applied for a few tests; the deformation mechanisms and mechanical behavior of the material was basically unchanged. Energy minimization followed by dynamic relaxation for 50 ps was done before initiation of loading. The temperature of the system was