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Plastic deformation in nanoindentation of tantalum: A new mechanism for prismatic loop formation

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Abstract

The mechanisms of deformation under a nanoindentation in tantalum, chosen as a model body-centered cubic (bcc) metal, are identified and quantified. Molecular dynamics (MD) simulations and indentation experiments are conducted for [1 0 0], [1 1 0] and [1 1 1] normals to surface orientations. The simulated plastic deformation proceeds by the formation of nanotwins, which rapidly evolve into shear dislocation loops. It is shown through a dislocation analysis that an elementary twin (three layers) is energetically favorable for a diameter below ~7 nm, at which point a shear loop comprising a perfect dislocation is formed. MD simulations show that shear loops expand into the material by the advancement of their edge components. Simultaneously with this advancement, screw components of the loop cross-slip and generate a cylindrical surface. When opposite segments approach, they eventually cancel by virtue of the attraction between them, forming a quasi-circular prismatic loop composed of edge dislocation segments. This "lasso"-like mechanism by which a shear loop transitions to a prismatic loop is identified for both [0 0 1] and [1 1 1] indentations. The prismatic loops advance into the material along ⟨1 1 1⟩ directions, transporting material away from the nucleation site. Analytical calculations supplement MD and experimental observations, and provide a framework for the improved understanding of the evolution of plastic deformation under a nanoindenter. Dislocation densities under the indenter are estimated experimentally ($\sim 1.2 \times 10^{15}\ m^{-2}$), by MD ($\sim 7 \times 10^{15}\ m^{-2}$) and through an analytical calculation ($2.6–19 \times 10^{15}\ m^{-2}$). Considering the assumptions and simplifications, this agreement is considered satisfactory. MD simulations also show expected changes in pile-up symmetry after unloading, compatible with crystal plasticity.

Keywords: Nanoindentation; Molecular dynamics; Dislocations; Shear loops; Prismatic loops

1. Introduction

The origins of hardness testing can be traced back to the 19th century [1–3]. This simple method to evaluate the strength of metals has been immensely successful, principally because of its simplicity and quasi-non-destructive nature. The number obtained, the "hardness", represents the resistance of the material to penetration, a reasonable measure of compressive strength under lateral confinement. Portable units to measure the hardness are available, and hardness measurement is also very useful as a research tool, providing a ranking of materials. The plastic deformation under the indenter is highly heterogeneous, and attempts to correlate the numbers obtained with fundamental materials parameters often fall short [4]. Nevertheless, the simple Tabor [5] relationship between yield stress and hardness ($\sigma_y = H/3$) is often used, in spite of its limitations.

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Nanoindentation testing has gained global acceptance as a tool to probe the mechanical properties of materials at the micrometer and sub-micrometer scale and the continuous load–penetration curve provides the elastic modulus and hardness through the widely used Oliver–Pharr analysis [6].

In parallel with plasticity analysis of the plastic deformation under an indenter [7–10], molecular dynamics (MD) simulations are becoming quite realistic and capture, albeit at a much smaller spatial scale and much larger indentation velocities, the features of the plastic deformation processes occurring under the indenter. There is a large number of studies carrying simulations for face-centered cubic (fcc) metals, due to the large number of experiments for such metals, but also due to the availability of empirical potentials for fcc metals, which behave reasonably well at large strains. Zhu et al. [11], Li et al. [12] and van Vliet et al. [13] performed MD and finite element calculations for fcc metals and showed that stacking-fault loops nucleated under the indenter, not at the surface but below it. Although there are several dislocation nucleation criteria [11], a simple criterion based on a threshold shear stress is often used. Indeed, the region of maximum shear stress under an indenter is below the surface. The Hertz lines of maximum shear stress in the compression of a flat surface by a cylindrical or spherical indenter show this clearly [14]. Zhong and Zhu [15] showed that nucleation, gliding and interaction of Shockley partial dislocations in fcc structures were involved in the early plastic stages of indentation, and recently detailed dislocation analysis was also carried out by Begau et al. [16] and Engles et al. [17].

Scanning tunneling microscopy and atomic force microscopy (AFM) in ultrahigh vacuum have also been used to characterize indentation in gold (fcc structure) [18]. There are many related studies on Au nanoindentation [19]. Elastic and plastic indentations were identified both in the residual impression images and by features in their force–displacement curves such as the sink-in depth, pop-ins and hysteresis energy but there are still many open questions [20].

Fang et al. [21] performed MD calculations for aluminum and reported partial dislocations emanating from the indentation site, with stacking faults on {1 1 1} planes. Tsuru et al. [22] also investigated the behavior of aluminum, using both atomistic and microscopic models. In general, it is often found that the necessary critical resolved shear stresses for dislocation nucleation is higher than the theoretical shear strength because of the compressive stress state underneath the indenter, with load–displacement curves showing both elastic and pop-in displacements. Begau et al. [16] performed simulations for copper, allowing the dislocations to move larger distances from the indentation region, and observed leading and trailing partial dislocations and stacking faults between them in configurations fairly similar to the ones observed by Traiviratana et al. [23] in the growth of voids in copper. Ziegenhain et al. [24] investigated the effect of crystal anisotropy (in Cu and Al) on the generation of partial and perfect dislocations and observed emission of prismatic loops below the indenter.

In one of the first MD simulations for indentation of body-centered cubic (bcc) metals, tungsten was investigated using a Finnis–Sinclair potential with ~850,000 atoms, [1 1 1] and [1 1 0] surfaces, and a tip diameter of 10 nm [25]; penetration depths were limited due to the relatively small sample size, but allowed the observation of the earlier stages of plastic activity, often including emission of prismatic loops. Kumar et al. [26] recently simulated Fe, pure and with impurities, using ~1 million atoms, a 4 nm diameter indenter and velocity of 100 nm ps^{-1}, showing that slip occurs in {1 1 0}, {1 1 2} and {1 2 3} planes, as expected from bcc crystal symmetry.

Alcalá et al. [27] described dislocation structures under nanoindentations in tantalum crystals using the potential by Li et al. [28], and presented experimental loading curves for [0 0 1], [0 1 1] and [1 1 1] surfaces. They found that (indent depth)/(indenter diameter) ~ 0.2 gave a reasonable value for the first pop-in event in both simulations and experiments. They reported the generation of stacking faults and twins directly under the indenter as well as dislocation loops; twin nucleation and interaction as well as annihilation produced these loops. Their load–penetration curves showed a marked elastic–plastic transition in line with their experimental measurements for [1 0 0], [1 1 0] and [1 1 1], which exhibited an elasto-plastic pop-in corresponding to the emission of the first dislocations. The leading edges of the loops were of edge character and the trailing parts were screw dislocations. Twin annihilation was attributed to reduction of stacking fault energy for twin layers thinner than four atomic layers and found to be enhanced at higher temperatures and decreased loading rates. Sectional views showed the progressive development of dislocation loops and their interactions.

It should be mentioned that most indentation studies are either computational or experimental. The investigations conducted by Alcalá et al. [27], Lodes et al. [29] and Sadrabadi et al. [30] on CaF_2 are rare exceptions. Since this crystal is amenable to etch pitting, Lodes et al. [29] were able to quantitatively estimate dislocation densities under the indenter and compare them with MD predictions. The molecular dynamics predictions essentially confirmed earlier experimental results [30]: dislocation density decreased with penetration depth consistently with a decrease in hardness due to increasing load.

The objective of this contribution is to provide a combined computational–experimental–analytical study leading to a quantitative knowledge of the mechanisms of plastic deformation under the indenter for a model bcc metal, tantalum. Below we first describe experimental and computational methods, then present loading curves and AFM images of the indented surfaces. We present simulated loading and unloading curves, describing in detail the formation of prismatic loops during loading by a new "lasso" mechanism and present a dislocation-based model