Experimental simulation of defect nucleation during nanoindentation

Nanoindentation, *i.e.*, the quasistatic normal penetration of a surface to depths on the order of nanometers, is simulated using the Bragg bubble raft model[1] in which a close-packed array of soap bubbles represents the equilibrium positions of atoms within a crystalline solid. Systematic experiments reveal that homogeneous dislocation nucleation occurs within the crystal when its surface roughness is comparable to indenter radius, and that the depth of the nucleation site below the surface scales with contact half-width. These *in-situ* observations appear to provide a fundamental justification for the unusually high local stress required for defect nucleation in nanoindented face-centered cubic (FCC) crystals.

Nanoindentation of FCC metals shows a load versus displacement response separated into regions of elastic deformation and discrete displacement bursts[2-4] (Fig. 1a). The first displacement burst generally occurs when maximum shear stress generated under the indenter is on the order of theoretical shear strength[2,3]. This high local stress ostensibly causes homogeneous nucleation of dislocations beneath the surface, producing a displacement burst[3].

In order to rationalize these observations, we present the bubble raft as a model for nanoscale atomic contact. The bubble positions represent the equilibrium positions of atoms[1], enabling visualization of deformation, dislocations, adhesion and fracture[5-7]. Methods of bubble raft production are known[1,6,7]. Indentation along the <121> direction of the raft proceeds orthogonally to the <110> closed-packed direction in the {111} plane. The raft's contact edge is made flat by removing extraneous bubbles with a soldering iron. A completed raft, comprising more than 10^4 bubbles, measures approximately 250 mm x 250 mm, simulating semi-infinite boundary conditions. Each bubble – 1 mm in diameter – represents a typical atom diameter of 0.3 nm. All other relevant dimensions in the model are converted to atomic dimensions using this size scale analogy.

We indent a single-crystal, initially defect-free bubble raft in the plane of the raft along the <121> direction with indenters of simulated tip radii, R = 8 and 28 nm. The indenter, constructed of aluminum plate, is positioned in plane with the raft and slightly below the surface of the solution. The load is applied in displacement control, via a screw-driven mechanism (Fig. 1b). The in-plane shear stress beneath the indenter is maximum at a ratio of depth (z) to contact half-width (a) of 0.78 (Fig. 1c), as predicted by two-dimensional Hertzian indentation theory[8].

Figures 1c and 1d display dislocation nucleation events under atomically flat surfaces for two different indenter radii. In both cases, a dislocation dipole nucleates beneath the indented surface along the loading axis at a depth of 0.78a; this location was determined from direct observation of z and a. The dipole splits; one dislocation glides into the crystal and the other runs to the surface, creating a slip step. Burgers circuits confirm the dislocations as edge type, with Burgers vector $\boldsymbol{b}$ along the <110> direction. Dislocations nucleate at a constant depth of 0.78a; nucleation occurs farther below the surface with increasing indenter radius. Thus, continuum elasticity provides a valid assessment of stress distribution, down to the simulated atomistic dimensions. This finding suggests that such an approach would correctly describe elastic stresses in actual nanoindentation experiments, consistent with the elastic response seen prior to the first displacement burst during the nanoindentation of FCC metals[2,3] (*e.g.,* Fig. 1a).

In Fig. 1e, indentation of an “atomically rough” surface initiates plasticity at the contact surface. In Fig. 1f, indentation of a surface ledge, of width comparable to indenter radius, causes nucleation inside the crystal. This clear trend provides a fundamental justification for the possible role of roughness in inducing plasticity during nanoindentation of metals. That is, surface roughness (in the form of asperities and ledges) is expected to cause dislocation nucleation near the surface of the crystal if the width of the ledge is substantially smaller than the tip radius R. If the width of the ledge is greater than R, available heterogeneous nucleation sites are distanced far enough away from the maximum stresses beneath the indenter so as to sustain dislocation nucleation within the crystal. For typical nanoindentation with an indenter tip radius R = 50 nm, surface asperities larger than 50 nm should exhibit an indentation response similar to that for an atomically flat surface. As crystalline specimens are routinely polished to roughness of 50 nm or higher, this provides a rationale as to why the first displacement burst shown in Fig. 1 corresponds to stresses approaching theoretical shear strength.

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1. Bragg, W.L. and Nye, J.F. *Proc. Roy. Soc. A.*, **190**, 474-481 (1947).
2. Suresh, S., Nieh, T.G. and Choi, B.W. *Scripta Materialia* **41**, 951-957 (1999).

3. Gouldstone, A., Koh, H.J, Zeng, K.Y., Giannakopoulos, A.E. and Suresh, S. *Acta Mater*., **48**, 2277-2295, (2000).

4. Gerberich, W.W., Nelson, J. C., Lilleodden, E.T. Anderson, P. and Wyrobek, J. T. *Acta Mater*., **44**, 3585-3598 (1996).

5. Shi L.T. and Argon A.S. *Phil. Mag. A*., **19**, 255-274 (1982).

6. McClintock F.A. and O'Day, W.R. *Proc. Int. Conf. Fract*., **1**, 75-98 (1965).

7. Georges, J.M., Meille, G., Loubet, J.L. and Tolen, A.M. *Nature*, **320**, 342-344 (1986).

8. Johnson, K.L. *Contact Mechanics* (Cambridge University Press, Cambridge, 1985).