The influence of laser-induced nanosecond rise-time stress waves on the microstructure and surface chemical activity of single crystal Cu nanopillars

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An apparatus and test procedure for fabrication and loading of single crystal metal nanopillars under extremely high pressures (>1 GPa) and strain rates ($>10^7$ s⁻¹), using laser-generated stress waves, are presented. Single-crystalline Cu pillars (~ 1.20 μm in tall and ~ 0.45 μm in diameter) prepared via focused ion beam milling of Cu(001) substrates are shock-loaded using this approach with the dilatational stress waves propagating along the [001] axis of the pillars. Transmission electron microscopy observations of shock-loaded pillars show that dislocation density decreases and that their orientation changes with increasing stress wave amplitude, indicative of dislocation motion. The shock-loaded pillars exhibit enhanced chemical reactivity when submerged in oil and isopropyl alcohol solutions, due likely to the exposure of clean surfaces via surface spallation and formation of surface steps and nanoscale facets through dislocation motion to the surface of the pillars, resulting in growth of thin oxide films on the surfaces of the pillars. © 2013 American Institute of Physics. [<http://dx.doi.org/10.1063/1.4793646>]

I. INTRODUCTION

There has been a keen interest in recent years to understand material behavior under extreme conditions of pressure (10–200 GPa) and strain rate (10^6 – 10^8 s⁻¹), as experiments have revealed new deformation mechanisms that do not follow the conventional dislocation-barrier models.^{1–7} These models assume that dislocations already exist in the material, that they are initially pinned, and that their motion in response to the applied strain rate is controlled by two key phenomena: (1) thermal activation over pinning barriers and (2) phonon drag during motion in-between barriers. Kiritani and co-workers^{8,9} found thin face-centered cubic (fcc) metal (Au and Cu) samples to be dislocation-free and composed of stacking-fault tetrahedra and nanoscale voids when subjected to tension and compression loads at high strain rates. Remington *et al.*¹⁰ found that in fcc metals with low stacking fault energy (such as copper), shock compression initiated the twinning-slip transition and a new $\alpha \rightarrow \epsilon$ (bcc to hcp) phase transformation in iron during plastic deformation at sub-nanosecond time scales. Large-scale molecular dynamics (MD) simulations have been carried out to explain these observations, for example by Li and Ghoniem,¹¹ showed that in pure Cu there is a transition from dislocation- to twinning-dominated deformation because dislocation barriers do not exist *a priori*, and the energy differential between these two deformation modes is rather small. Bringa *et al.*¹² performed simulations with pre-existing dislocation sources in the form

of prismatic loops in shocked Cu single crystals. Their simulations indicate that once the transient pressure wave exceeds the threshold for either activating the source (~ 10 GPa) or homogeneous nucleation of dislocations (~ 30 GPa), the dislocation density relaxes by a factor of three resulting in the onset of three-dimensional plastic relaxation behind the shock front with the formation of a high density of dislocations and stacking faults. The present investigation aims to explore new modes of deformation in materials subjected to strain rates higher than those previously studied (10^5 s⁻¹), and to uncover atomic-scale mechanisms (defect formation or annihilation, dislocation movement, twinning, etc.) that allow materials to rearrange themselves when subjected to extreme conditions. An additional objective of this work is to use the experimental observations to validate our on-going MD simulations.

In the present work, we report on the fabrication and shock-loading of 100-oriented, single-crystalline, Cu nanopillars, with pillar axes aligned with the wave propagation (compression) direction. To this purpose, we developed an apparatus and related test procedures for the compression of Cu nanopillars at pressures and strain rates greater than 1 GPa and 10^7 s⁻¹, respectively using laser-generated stress waves of 1–2 ns rise-times and 16–20 ns total duration. Deformation mechanisms are identified by transmission electron microscopy (TEM) characterization of the residual microstructure in shock-loaded samples. The experimental approach presented here is general and applicable to shock-loading of other materials. The experimentally measured stress wave profiles and the sample microstructures can be used as input for large-scale MD and finite element simulations to help elucidate the underlying microscopic dislocation dynamics and the atomistics of deformation mechanisms.

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II. EXPERIMENTAL PROCEDURE

All of our experiments were carried out using single-crystalline Cu(001) pillars in the Laser Spallation Facility, developed previously to measure tensile strengths of interfaces.¹³ Schematics of the experimental setup are shown in Figures 1 and 2. The Cu pillars ($\sim 1.20\ \mu\text{m}$ in tall and $\sim 0.45\ \mu\text{m}$ in diameter) spanning approximately a $10\ \mu\text{m}$ diameter were prepared out of Cu(001) substrates ($10\text{ mm} \times 10\text{ mm} \times 0.5\text{ mm}$ purchased from MTI Corporation, Richmond, CA). Pillar dimensions were chosen so that the morphologies of the as-prepared and shock-loaded pillars could be determined using electron microscopy. Four groups of six identical pillars were prepared in a Nova 600 Dual Beam focused-ion-beam/scanning electron microscope (FIB/SEM) system using gallium ions at 0.5 nA current and an accelerating voltage of 30 kV . Each of the four groups were separated from its neighbors by at least 5 mm so that each group of pillars could be independently loaded by the shock wave. This enabled us to carry out multiple tests on the same substrate.

In a typical experiment, the Cu sample was sandwiched between a 1-mm-thick glass substrate and a 9-mm-thick brass plate as shown in Figure 1. The back surface of the glass substrate was coated with a $0.5\text{-}\mu\text{m-thick}$, laser absorbing Al film, which was constrained from the top by a transparent $20\text{--}50\ \mu\text{m}$ thick layer of waterglass. The thicknesses of the Al and the waterglass layers were optimized by Yuan *et al.*¹⁴ and Gupta *et al.*¹⁵ to generate stress waves with the fastest rise times ($< 1\text{ ns}$) and shortest duration so that Cu pillars could be loaded under extreme conditions. Brass was chosen to minimize the wave reflection effects and maintain a uniaxial compressive state of stress in the copper nanopillars since it has a low acoustic impedance mismatch with the Cu substrate ($3.7 \times 10^7\text{ Ns/m}^3$ for brass vs. $4.1 \times 10^7\text{ Ns/m}^3$ for Cu). Moreover, in order to preserve the deformation caused by the incoming stress wave, the brass plate was made very thick to geometrically disperse the waves and therefore decrease the possibility of any reloading of the pillars by the reflected waves. The glass substrate, copper sample, and the brass plate assembly were mechanically joined, sandwiched between two plexiglass plates and submerged in an oil bath to ensure proper coupling and continuity between

FIG. 1. Cross-sectional view of the experimental setup, showing two sets of nanopillars on a copper sample, oriented such that the axes of the pillars are aligned with the stress wave propagation direction.

FIG. 2. Schematic of the laser spallation test set-up along with that of an optical interferometer used to generate and record stress waves. Adapted from Youssef and Gupta¹⁶ and Youssef.³²

all the interfaces so that stress waves could transmit across them. A window was cut in the plexiglass plate to allow access to the YAG laser pulse, as shown in Figure 1.

In this setup, a nominal 8 ns long Nd:YAG laser pulse was focused over 2.22 mm diameter on the Al film. Upon absorption of the laser energy, the Al film melts and exfoliates. This process results in the launch of a pressure wave with a $1\text{--}2\text{ ns}$ rise-time and $16\text{--}20\text{ ns}$ duration toward the sample mounted on the front surface of the substrate disc.^{17,18} Prior to the laser shot, the axis of laser beam was aligned with the center of one of the pillar groups. This configuration allowed for the stress waves to propagate along the axes of each of the nanopillars and transmit into the brass plate.

Each group of pillars was shock-loaded once using laser shots of different energies. All the pillars within a group were uniformly loaded as they were located 1.5 mm from the laser heating area and within $50\ \mu\text{m}$ of the stress wave propagation axis. This can be concluded based on the calculations of Lev and Argon,¹⁹ who showed that plane wave conditions are maintained over 95% of the initially stressed area at a wave propagation distance equal to the diameter of the laser heating spot.

In order to obtain the stress state in our copper sample, a state-of-the-art optical interferometer (see Figure 2), capable of recording fringes with 0.2 ns resolution in the single shot mode, was used to measure the free surface displacement. In prior studies, where thin film samples were subjected to tensile loads, optical interferometry was used to calculate the peak interfacial stress using a wave mechanics simulation.¹³ Utilizing this setup, interfacial strengths of 0.1 to 2.5 GPa have been reported for a variety of interfacial systems, involving metals, ceramics, and polymers.^{13–15,20–25} In this study, it was not possible to measure the incident time-dependent stress wave profile $\sigma(t)$ generated in the glass plate during the deformation of the Cu pillars. Instead, $\sigma(t)$ was obtained in a separate experiment in which an isolated glass plate was shock-loaded under the same stress wave generation conditions (laser fluence and thicknesses of glass substrate, Al film, and waterglass layers) as those used for

compression of the Cu pillars. The free surface velocity $v(t)$ of the glass plate was recorded using the optical interferometer (see Figure 2) and used to calculate $\sigma(t)$ by equation

$$\sigma(t) = \frac{\rho c_o}{2} \left[v \left(t + \frac{h}{c} \right) - v \left(t - \frac{h}{c} \right) \right], \quad (1)$$

where c_o ($=2289$ m/s), ρ , and h are the longitudinal wave speed in the medium (glass), its density, and thickness, respectively. Details of interferometer methodology can be found in Pronin and Gupta,²⁶ Barker and Hollenbach,²⁷ and Clifton.²⁸ The exact compressive stress-state inside each pillar can be determined using finite element simulations with $\sigma(t)$ as an input. Thus, local stresses obtained can be used for carrying out MD simulations.¹⁶

After shock-loading, the pillars were lifted off the substrate using FIB and mounted on TEM grids. Electron-transparent (<100 nm) cross-sectional samples were prepared by FIB milling of the pillars at a low accelerating voltage, 5 kV. TEM, selected area electron diffraction (SAED), scanning TEM (STEM), along with energy dispersive X-ray spectroscopy (EDS) measurements were carried out using a JEM-100CX (JEOL) TEM operated at 100 kV and a Titan (FEI) S/TEM operated at 300 kV.

III. RESULTS AND DISCUSSION

Figure 3(a) is a typical interferometric fringe record obtained at a laser fluence of 95.2 kJ/m² from the free surface of the isolated glass plate sample using the same stress generation conditions as those used to shock-load Cu pillars. Figures 3(b) and 3(c) show the corresponding velocity and stress wave profiles, $u(t)$, and $\sigma(t)$, respectively obtained from this fringe record, by using the well-established procedures of interferometry mentioned earlier. Using this stress wave profile as input, finite element simulations of the wave propagation under identical experimental conditions yielded a maximum compressive stress of 1 GPa and peak strain rate of 3×10^7 s⁻¹ inside the Cu pillars (with percent errors of $\pm 5\%$). In the following paragraphs, we focus on the effect of shock-loading on morphologies and microstructures of the pillars.

In order to investigate the effect of shock-loading on surface morphologies of the Cu pillars, each of the pillar groups were subjected to a different laser fluence and the resulting

morphologies were examined using SEM. Figure 4 is a representative panel of SEM images acquired from three different pillars each belonging to a separate group on the same Cu(001) sample. The images (Figures 4(a)–4(c)) in the top row show a reference pillar that was not shock-loaded. The middle (Figures 4(d)–4(f)) and bottom (Figures 4(g)–4(i)) rows show SEM images of two different Cu pillars that were shock-loaded under uniaxial compression by stress waves generated at laser fluence of 39.5 kJ/m² and 93.4 kJ/m², respectively. The time $t = 0$ h in the panel, i.e., the left column images (Figures 4(a), 4(d), and 4(g)), is defined arbitrarily and corresponds to the pillar morphologies prior to mounting the Cu sample for shock-loading experiments. Following loading, the sample containing these three different pillars was submerged in isopropyl alcohol (IPA) for ten minutes. It is assumed that IPA helps dissolve any oil that may be present on the sample, which was surrounded by an oil bath used as a coupling medium during loading. The sample was subsequently removed from the IPA solution and left air-exposed for 12 h at room temperature. SEM images were acquired from this sample and are shown in the middle column (see Figures 4(b), 4(e), and 4(h)). When compared to the reference pillar (Figure 4(b)), note the changes in surface morphologies of the shock-loaded pillars in Figures 4(e) and 4(h). On both the shock-loaded pillars, the images reveal: (1) increased surface roughness; (2) spalled regions near the top of the pillars likely due to loading by the shock wave; and (3) an outward growth of thin films with highest coverage around the base of the pillar.

Later, the same sample was submerged in IPA solution for additional 3 h, removed, and air-dried for 18 h at room temperature. Subsequently, SEM images were acquired from this sample and are shown in the right column (see Figures 4(c), 4(f), and 4(i)). The time $t = 36$ h indicated in Figure 4 is the total time elapsed between the loading of the sample and the acquisition of the SEM images shown in the last column. The images reveal extensive growth of thin films around the surface of shock-loaded pillars. In contrast, the surface morphology of the reference pillar changed little over the 36 h. Given that all the pillars were situated on the same Cu sample, submerged in the same set of solutions, and for the same duration, the observed film growth on the shock-loaded pillars strongly suggests that shock-loading enhanced their surface reactivities.

FIG. 3. (a) Fringe record obtained at laser fluence 95.2 kJ/m² using the interferometry shown in Fig. 2(b). (b) The reduced displacement profile derived from (a). (c) The stress wave profile derived from (b).

FIG. 4. SEM images of Cu pillars on a Cu(001) substrate ((a), (d), (g)) prepared via FIB milling prior to testing. ((b), (e), (h)) and ((c), (f), (i)) are obtained 12 and 36 h, respectively after loading. The images in the top, middle, and bottom rows show three different pillars tested at laser fluence of 0, 39.5, and 93.4 kJ/m², respectively. The scale bar in all the images is 400 nm.

In order to identify the chemical composition of these films formed around the Cu pillars, EDS, to obtain the elemental constituents, and SAED, to determine the compound, were used. Figure 5(a) is a typical STEM image of the pillar shown in Figure 4(f), shock-loaded at 39.5 kJ/m². Point mode data was acquired from the regions surrounding the pillar. The associated plot below Figure 5(a) shows the characteristic X-ray signals from the elements (C, O, Cu, Pt, Ga, and S) at the points labeled 1–3 in Figure 5(a). The EDS data from spot 2 in Figure 5(a), i.e., from the thin film, reveal relatively high concentrations of carbon and oxygen. (The Pt and Ga signals are due to FIB milling and S is likely due to the oil bath, respectively.) The plot associated with Figure 5(a) shows strong signal from the elements (Cu, C, and O) in spot 2 from Figure 5(a). Based upon these results, we suggest

that the thin films observed around the pillars are likely to be copper oxides or copper carbonate. Figure 5(b) is a representative SAED pattern obtained from the film at point 2 in Figure 5(a). The observed diffraction rings indicate that the film is polycrystalline. From the measured interplanar spacings, we determined the crystal structure to be cubic copper(I) oxide (JCPDS file No. 5-667). We attribute the observed thin film growth to the exposure of fresh Cu surfaces resulting from surface material spallation and the formation of surface steps and nanoscale facets via elimination of the dislocations from the bulk. This film growth via shock-loading is possible throughout the entire bulk but is more likely to occur where the surface area to volume ratio is high, as is found with the pillars. While the surface spalls are visible in the SEM images, the presence of surface steps and/

FIG. 5. (a) STEM image of the pillar shown in Fig. 4(f) loaded at 39.5 kJ/m². Elemental composition spectrum, as determined using EDS, at the points 1–3 are provided in the graphs below. (b) SAED pattern acquired from the region labeled 2 in (a).

or facets could not be confirmed. However, in the following section, we provide evidence for shock-induced dislocation motion, a strong indication of mechanical annealing.

Figures 6(a)–6(c) are typical bright-field TEM images of a reference pillar that was not shock-loaded (Figure 6(a)) and two pillars that were independently loaded with a laser fluence of 39.5 kJ/m² (Figure 6(b)) and 93.4 kJ/m² (Figure 6(c)), respectively. The SAED pattern in Figure 6(d) was obtained from the pillar in Figure 6(a) and is identical to the diffraction (not shown) from the shock-loaded pillars. That is, in our experiments, shock-loading did not affect the crystallinity of the pillars. All the TEM images show darker contrast cellular structures within the pillars. A majority of these features are dislocations while other features are Ga clusters which are common artifacts due to the FIB fabrication processes utilized.²⁹ The high density of dislocations observed in the bulk of the reference pillar (Figure 6(a)) could be attributed to the quality of the Cu crystal. And, the dark contrast present around the surface of the pillars is due to the FIB milling process used to fabricate the pillars, indicated by the depth of the damage.²⁹ Since all the pillars were milled out of the same Cu(001) crystal and used the same FIB parameters for both pillar and TEM sample fabrication, it is expected that the density and spatial distribution of radiation damage from FIB were similar in all the pillars. While the microstructures of shock-loaded pillars shown in Figures 6(b) and 6(c) are qualitatively similar to that of the as-prepared pillar (Figure 6(a)), it is possible that shock-loading can increase or decrease the dislocation density by nucleation of new dislocations or by removal of dislocations by motion from the bulk to the surface, respectively. The latter process results in the formation of new surface steps and/or nanoscale facets, which could explain the enhanced surface reactivity of the shock-loaded pillars (Figures 4(f) and 4(i)).

To quantitatively assess the effect of shock-loading on the microstructure of the pillars, dislocation densities and orientations were determined. For consistency, all the TEM images analyzed were acquired under identical imaging conditions, i.e., bright-field images of the pillars were taken in exact [001] zone axis. And, since the TEM samples were prepared using FIB, those dislocations near the pillar's

surface were ignored. From the TEM images the average dislocation densities were calculated. The calculated dislocation density decreased from $7.9 \times 10^{14} \text{ m}^{-2}$ in the reference pillar to $3.1 \times 10^{14} \text{ m}^{-2}$ and $2.1 \times 10^{14} \text{ m}^{-2}$ in the pillars loaded at 39.5 kJ/m² and 93.4 kJ/m², respectively. The decrease in dislocation density is most likely a result of dislocation motion out of the bulk.

In order to further explore the influence of shock-loading on dislocation density in the pillars, the orientations of projected dislocations visible in the bright-field TEM images (Figures 6(a)–6(c)) were measured with respect to the major axis of the pillars, [100]. Since detailed characterization of all of the dislocations present in the pillars is a non-trivial task requiring elaborate electron microscopy, analyses were limited to measuring *relative* changes in orientations of only those dislocations that are visible under specific imaging conditions. In particular, dislocations with $\frac{1}{2}[110]$ Burgers vectors are of interest, which are common in fcc crystals such as Cu.³⁰ When the [100]-oriented Cu pillars are viewed along the [001] zone axis in the TEM, the projections of these dislocation lines are expected to appear at 45° with respect to the pillar axis, provided the deviation parameter is small.³¹ In the reference pillar, the average angle subtended by the dislocations is $46.0 \pm 5.5^\circ$. In comparison, the angles are $41.5 \pm 8.6^\circ$ and $43.2 \pm 13.0^\circ$ in samples loaded with stress waves generated at laser fluence of 39.5 and 93.4 kJ/m², respectively. Each of these values was determined from 14 dislocation lines observed in each of the pillars. While the average angles are nearly the same within the measurement uncertainties, note the increase in deviations in angles from 5.5° in the reference pillar to 8.6° and 13.0° in the shock-loaded samples. That is, the orientational spread of dislocations increases with increasing laser fluence. These results provide further evidence in support of the hypothesis that shock-loading induces motion of the dislocations and/or formation of new dislocations.

The data presented above indicate that the density and orientation of dislocations depend on the laser fluence. These results can be explained as follows. The stress waves generated within the pillars due to the laser shot are transient and over a short duration (~20 ns). Therefore, the amplitude of

FIG. 6. Bright-field TEM images of three different Cu(001) pillars acquired (a) as-prepared and after shock-loading using laser fluence of (b) 39.5 kJ/m² and (c) 93.4 kJ/m². All pillars have [001] as their main axis and the samples were oriented along the [001] zone axis during TEM analysis. The scale bars in all images is 100 nm. (d) SAED pattern of the pillar shown in (a). The ring pattern observed in (d) is due to Pt thin film deposited as a protective layer prior to TEM sample preparation via FIB milling.

the stress and strain rate change continuously with a maximum stress of 1 GPa and strain rate of $3 \times 10^7 \text{ s}^{-1}$ for a laser fluence of 95.2 kJ/m^2 . At such high strain rates and short time scales over which the pillars were loaded at a given stress value, motion of pre-existing dislocations is limited because of the increase in the local shear strength of the slip systems. When dislocation motion does occur, it is limited as they are eventually pinned by their neighbors and additional stress is required for their continued motion. This dislocation pinning can also lead to nucleation of new dislocations. Those dislocations present near the surface, however, are likely to escape leaving behind surface steps and nanoscale facets. This is consistent with the TEM observations of a relatively low density of dislocations within the top 30 nm of the shock-loaded pillars. Consequently, dislocation motion is expected to be higher at higher laser fluence resulting in the observed lower dislocation density and wider orientational spread.

In summary, we demonstrated an experimental approach for ultra-high strain rate, uniaxial compression of metal pillars using laser-generated shock waves. FIB-prepared single-crystalline Cu(001) nanopillars were used as model materials. Electron microscopy was utilized to investigate the effects of shock-loading on surface morphology and bulk microstructure of the pillars. We found that shock-loading enhances surface chemical reactivity via a combination of surface spallation and bulk dislocation motion resulting in exposure of pristine surfaces and the formation of surface steps and nanoscale facets. Our results indicate that materials subjected to ultra-high strain rate loading undergo deformation and the underlying mechanisms can be different from those expected at conventional strain rates. The test procedures employed in our study are general and applicable to other crystalline materials. The sample geometry and the dimensions facilitate direct comparison of experimental results with molecular dynamics and finite element method simulations, which will provide valuable insights into the underlying deformation mechanisms.

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