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Laser-induced photoemission as a probe of slip band formation in single crystal and polycrystalline aluminum during uniaxial deformation

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Abstract. We report measurements of laser-induced photoelectron emission (LIPEE) from single crystal aluminum (99.999%) and high purity polycrystalline aluminum (>99.9%) during uniaxial tensile deformation. A 248-nm excimer laser (5-eV photon energy) was used as a light source. Deformation was performed on a tensile stage in ultra-high vacuum at an initial strain rate of $1 \times 10^{-3}$ s$^{-1}$. Photoelectron intensities are sensitive to changes in surface morphology accompanying deformation, including slip line and band formation. In the single crystal material, LIPEE intensity initially increases linearly with strain followed by a monotonically decreasing slope at larger strain. In the polycrystalline material, LIPEE intensities increase linearly with strain in two segments. Slip bands on the deformed surfaces were characterized by atomic force microscopy (AFM).

1. Introduction
Aluminum and aluminum alloys have major applications in the construction, automotive and aerospace industries. The relationships between dislocation microstructures and mechanical properties in aluminum and aluminum alloys have been studied extensively, typically by observations on previously deformed samples. The ability of these measurements to trace the evolution of complex dislocation structures is limited. Since macroscopic elastic strains are small in deformed metals, almost all of the slip accumulated during deformation appears as patches of fresh metal on the sample surface. Therefore, techniques that reflect these area changes during deformation can provide real-time information on deformation that is otherwise difficult to obtain [1].

At normal temperatures and strain rates, bulk aluminum deforms via the $\{111\}<110>$ slip systems, producing fine slip lines and slip bands on the surface with a band spacing of several microns [1-3]. Deformation of polycrystalline aluminum is more complicated because of the strain constraints along adjacent grain boundaries. In aluminum, deformation is generally held to be consistent with Taylor’s full constraint model [4]. Current 2-D and 3-D models of dislocation transport and work hardening suggest that dislocation motion and interaction show strong temporal fluctuations in the later stages of deformation of single crystal material [5-6]. The high sensitivity of photostimulated electron emission (PSEE) to changes in surface morphology make it a potentially useful probe of these fluctuations [7].
PSEE has been previously shown to accompany oxide fracture and slip band formation in aluminum and copper at strains < 0.10 [8]. Initial work with an ultra-high vacuum (UHV) compatible tensile stage and a stable mercury lamp showed fluctuations that were consistent with recent percolation models of dislocation motion [6-7]. In this work, we report improved measurements using an excimer laser as a light source as well as an improved UHV tensile stage.

2. Experiment
Photoemission measurements were performed in a stainless steel vacuum system with a base pressure of $1 \times 10^{-7}$ Pa [7]. Pulsed laser radiation at 248 nm (5-eV photons) was provided by an excimer laser (KrF, 30 ns pulses) operated at a repetition rate of 20 Hz. The laser output was attenuated with interference filters to achieve a total fluence of about 50 µJ/cm$^2$ at the sample gauge. Electron emission was detected with a Channeltron electron multiplier operated at a gain of about $10^6$. The output was amplified (amplifier time constant typically 10 ns) and digitized with a digital oscilloscope.

High purity single crystal aluminum (>99.999%) tensile specimens were characterized by Laue X-ray diffraction; the tensile axis was within a few degrees of the [112] direction with a gauge surface normal of [011]. Polycrystalline aluminum (>99.9%) specimens with different grain sizes were used to monitor the effect of grain size. The gauge plane of all specimens was 3 mm × 9 mm. Specimens were electropolished to a mirror-like surface and air-oxidized for 20 minutes before mounting in the UHV chamber. The average sample thickness after electropolishing was about 0.75 mm.

Real-time load and sample elongation during deformation were recorded with a digital oscilloscope synchronously with the photoelectron intensity. The crosshead speed of the tensile stage was 7.3 µm/s.

Atomic force microscopy (AFM) images of the deformed surfaces were acquired with a multimode scanning probe microscope operated at room temperature in the contact mode.

3. Results and discussion
Figure 1 shows the total laser-induced photoelectron emission (LIPEE) charge as a function of strain and the corresponding true stress-true strain curve. At the onset of loading, the sample response is mostly elastic and the stress rises rapidly (strain 0 to 0.010). On the expanded scale of figure 1b, the LIPEE signal remains at background levels through this elastic region. Grip effects and the early onset of recovery usually complicate room temperature stress-strain curves for pure Al single crystals. Nevertheless, the three expected stages of deformation (after elastic loading) are observed in figure 1 [9]. Stage I (strain 0.01 to 0.04) is dominated by simple slip on the primary slip system. At the onset of Stage II (strain 0.04 to 0.16), the slope of the stress strain increases as further deformation involves multiple slip on less favored crystallographic planes. Finally, the slope decreases again in Stage III (0.16 to failure), where deformation takes place by dislocation climb and diffusion. The LIPEE data exhibit an initial linear response with strain that starts at the onset of plastic deformation and continues to a strain of 0.16. At higher strains, the slope decreases until failure.

The most significant feature of the LIPEE signal in the context of dislocation dynamics are small, but significant, fluctuations. Individual or multiple dislocation percolation events locally relax stress relaxation and sometimes appear as small drops in stress [6]. The strongest fluctuations in the LIPEE signal correspond to changes in surface area accompanying similar events. (The standard deviation for the averaged LIPEE data is less than 0.32 pC. Most of the larger fluctuations are significant.) The discontinuities in the LIPEE data are strong evidence for the temporal heterogeneity of the nucleation and growth of slip bands at the surface.

Figures 2a and 2b show LIPEE signals for two polycrystalline aluminum samples. True stress vs. true strain curves in both materials show typical Voce hardening behavior. Unlike the single crystal material, the LIPEE signals from both polycrystalline specimens exhibit two clear linear segments. In previous work, the intersection of these two linear segments was identified with the onset of shear band formation, where deformation becomes localized to a thin band within the gauge section [10]. As expected, shear bands form at lower strains in the coarse-grained material (strain about 0.13) than in the fine-grained material (strain about 0.25). In figure 2, the LIPEE continues to increase for a short
time after the onset of failure due to the continued production of surface area as the sample necks. As with the single crystal material, step-like increases in LIPEE signals from the polycrystalline material are clearly resolved.

Figure 1. Laser induced photoelectron intensity vs. strain and true stress vs. strain during tensile deformation of a (110)[001] single crystal. (b) shows the initial data from (a) on an expanded scale.

Figure 2. Laser induced photoelectron intensity and true stress vs. true strain during tensile deformation of high-purity polycrystalline aluminum with average grain sizes of (a) ~200 μm and (b) ~75 μm.

To probe the spatial heterogeneity of deformation, selected regions of deformed specimens were imaged by AFM after the samples were removed from the vacuum system. Figure 3 shows typical slip structures produced during tensile deformation. The [101](1̅1̅1) and [011](1̅1̅1) slip systems dominate the deformation with a starting Schmid factor of 0.4 in the single crystal. Both slip systems intersect the sample surface in the same direction although the vertical components of the Burgers vectors have opposite signs. In the single crystal material (figure 3a), the slip bands have an average height of 0.50 μm and a spacing of 7.5 μm. The AFM images from the polycrystalline samples are from individual grains of random orientation. The slip patterns depend both upon the local grain orientation (with respect to the tensile axis and sample surface) and the configuration of the neighboring grains. The imaged grain in the coarse-grained sample shows clear slip on multiple slip planes; the orientations of the two bands with the surface are labeled 1 and 2 in figure 3b. Figure 3b also reveals that, although the slip bands with Orientation 1 often shear those with Orientation 2, the reverse behavior is also observed. Thus, these two slip systems developed together. In contrast with the image of figure 3b, the imaged grain from the fine-grained sample (figure 3c) exhibits slip
predominantly on a single slip plane. In general, the slip bands in polycrystalline aluminum are less than 0.3 μm high—smaller than those in the single crystal material. The small step heights are attributed to inter-grain incompatibility stresses that cause the local Schmid factors to change more rapidly than in the single crystal. The existence of grain boundaries, multiple slip systems, and grain rotations in deforming polycrystalline aluminum complicate the temporal production of fresh area (from slip bands), and randomize the fluctuations in LIPEE signal at larger strains (figures 1 and 2).

![Figure 3](image)

**Figure 3.** AFM images of deformed surfaces of (a) single crystal aluminum; (b) coarse grained polycrystalline aluminum; and (c) fine-grained polycrystalline aluminum.

Surface microstructural changes, especially the formation of slip lines and bands (figure 3), are expected to be the principal mechanism for increased LIPEE intensity during deformation. Fluctuations in the LIPEE signal from single crystal aluminum presumably reflect the dynamics of the nucleation and growth of features similar to the steps observed in figure 3a. In polycrystalline material, the corresponding microstructural changes are strongly affected by constraints due to adjacent grains. These constraints are stronger for smaller grains, consistent with both the late onset of strain localization and the smaller slip steps in the fine-grained material.

4. Conclusions

Laser induced photoelectron emission is a sensitive technique for characterizing the formation and growth of fine surface features in aluminum. LIPEE is sensitive to microstructural changes and reflects macroscopic changes in deformation character, including the onset of strain localization in polycrystalline aluminum. Real-time LIPEE in both single crystal and polycrystalline aluminum shows step-like increases with strain, consistent with the predicted temporal heterogeneity in slip. Conversely, the spatial heterogeneity of the nucleation and growth of slip bands is revealed by AFM. Both LIPEE and AFM measurements strongly support percolation models of dislocation motion during deformation.

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