Electron Emission as a Probe of Plastic Deformation in Single Crystal Metals

DE-FG03-02ER45988—April 1, 2003 to August 31, 2007

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Funds Remaining: $0.

Work under this grant focused on the use of photoelectron emission as a probe of deformation processes in metals, principally single crystal and polycrystalline aluminum. Dislocations intersecting the surface produce patches of low work function metal which emit electrons when illuminated with the appropriate ultraviolet radiation. We have shown that changes in the photoemission signals during deformation can be used to identify the onset of strain localization. In some systems, the photoelectron kinetic energy distribution reflects the distribution of surface orientations, which depends on the competition between grain rotation and slip. The statistics of photoemission intensity fluctuations is under analysis.

**Equipment.** In collaboration with Drs. Lyle Levine and Dave Pitchure at the National Institute of Standards and Technology (NIST), a UHV-compatible, precision tensile stage was designed and constructed to achieve a strain rate of \( \sim 10^{-3} \text{ s}^{-1} \) for tensile deformation of single crystal aluminum in vacuum. The stage design is based on a tensile stage used for x-ray studies of deformation in single crystal aluminum, facilitating comparison with previous work. Molds for the growth of single crystal aluminum samples were also constructed and sent to NIST for crystal growth and sample cutting. The cut samples were then returned to Washington State for photoemission/tensile deformation experiments.

Photoelectron emission microscope studies of shape memory alloys were carried out in collaboration with Wayne Hess, Kenneth Beck, Alan Joly, Timothy Droubay, and Gang Xiong at the DOE Environmental and Molecular Science Laboratory in Richland, Washington, using the EMSL Elmitec PEEM III instrument.

**Onset of strain localization.** During the initial stages of tensile testing, polycrystalline aluminum deforms rather homogeneously. At some point, deformation localizes along a band of grains. Further deformation is largely confined to a band which then moves along the gauge section. Although this important process has been the subject of extensive modeling efforts, quantitative real-time measurements are desired to constrain the models. We take advantage of the fact that the fraction of dislocations intersecting each of the four sides of the gauge section is changed by strain localization. This changes the rate of photoemission growth as a function of total strain. As expected, the photoemission data show curvature at the onset of strain localization.

**Effect of oxide on photoelectron emission intensities.** To reduce the noise in our measurements, it is convenient to be able to distinguish electrons emitted from bare patches of aluminum exposed by dislocation motion from those which pass through the native oxide of undeformed material. We observe two time-of-flight peaks in the photoemission signal from oxidized aluminum under 248-nm laser irradiation, where the oxide thickness was measured by X-ray photoelectron spectroscopy. For thin oxides, the intensity of the slower electrons
increases as the oxide thickness increases, consistent with energy loss due to inelastic scattering in the oxide. Numerically subtracting the intensity of these slow electrons from the total electron signal improves the correlation between photoelectron intensities and strain events. This work also establishes the optimum oxidation pre-treatment for the observation of deformation-related photoemission.

**Effect of surface orientation on photoelectron energies.** The work function of the (100), (110) and (111) surfaces of aluminum are 4.41, 4.01, and 4.24 eV, respectively. Thus changes in the area of (111) surfaces, due to slip, should alter the photoelectron energy distribution in different ways than changes in the area of (110) surfaces, due to grain rotation. By comparing photoelectron energy distribution measurements with electron backscattered diffraction measurements on cube-textured aluminum, we confirm that the changes in photoelectron energy distributions are consistent with slip on (111) planes and grain rotation on (110) planes.

**Single-crystal measurements.** Theoretical work by Robb and Levine predicts that the emergence of dislocation structures during the deformation of single crystal FCC metals displays stochastic properties similar to earthquakes. Photoemission from high purity, randomly oriented, single crystal aluminum during tensile strain show evidence of discontinuous strain events. The statistics of these events are being analyzed.

**Shaped memory alloys.** Shaped memory alloys exploit a temperature sensitive phase change to change from one shape to another. Metal work functions are typically quite sensitive to the crystalline phase. Thus one expects a significant change in photoelectron intensities to accompany the phase change. PEEM observations performed in collaboration with researchers at the DOE EMSL facility in Richland, Washington, show large changes in photoemission intensity as bulk CuZnAl and thin-film NiTiCu shape-memory alloys are heated through the phase change temperature. The NiTiCu films were deposited onto single crystal silicon. PEEM images of NiTiCu thin films show rapid changes in surface topography and intensity during cooling that lag the overall phase transformation determined from thin-film strain measurements. Since the photoemission signals are surface sensitive, this observation suggests that the transformation during cooling initiates along the silicon-NiTiCu interface and propagates to the free surface.

**Confocal microscopy observations.** The growth kinetics of photoelectron intensities during tensile testing show a marked change at the onset of strain localization. Scanning laser confocal microscope images of polycrystalline aluminum after various degrees of change show that the surface height distribution also grows more complex after the onset of strain localization. After localization, the height distribution becomes significantly nonGaussian. Since the analysis of surface roughness data often assumes Gaussian character, this observation indicates that more general techniques are required to characterize surfaces in the later stages of deformation.

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Publications Acknowledging this Grant
(Publications marked with asterisks are appended to this report.)


**Manuscripts in Preparation.**

Tribochemical wear of single crystal aluminum in NaCl solution studied by atomic force microscopy: M. Cai, F. Stevens, R. E. Ricker, and J. T. Dickinson.