LA-UR- 02- 4929

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Title: Electron Backscattered Diffraction (EBSD) of Plutonium-Gallium Alloys

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Submitted to: to be presented at the TMS Annual meeting symposium titled Actinide Materials: Processing, Characterization, and Behavior to be held March 2-6, 2003 in San Diego, CA

Los Alamos
NATIONAL LABORATORY

LA-UR-02-4929

Electron Backscatter Diffraction of Plutonium-Gallium Alloys

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Abstract

At Los Alamos National Laboratory a recent experimental technique has been developed to characterize reactive metals, including plutonium and cerium, using electron backscatter diffraction (EBSD). Microstructural characterization of plutonium and its alloys by EBSD had been previously elusive primarily because of the extreme toxicity and rapid surface oxidation rate associated with plutonium metal. The experimental techniques, which included ion-sputtering the metal surface using a scanning auger microprobe (SAM) followed by vacuum transfer of the sample from the SAM to the scanning electron microscope (SEM), used to obtain electron backscatter diffraction Kikuchi patterns (EBSPs) and orientation maps for plutonium-gallium alloys are described and the initial microstructural observations based on the analysis are discussed. Combining the SEM and EBSD observations, the phase transformation behavior between the δ and ε structures was explained. This demonstrated sample preparation and characterization technique is expected to be a powerful means to further understand phase transformation behavior, orientation relationships, and texture in the complicated plutonium alloy systems.

Electron Backscatter Diffraction of a Plutonium Alloy

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Outline

- Background
- Experimental
  - Sample Preparation
    - Metallographic polishing + electropolishing
    - Auger surface characterization and ion-sputtering
    - Vacuum transfer of sample to SEM
- Results
  - Sputtering effects on microstructure
  - EBSD images
  - Microstructural Evolution
- Summary

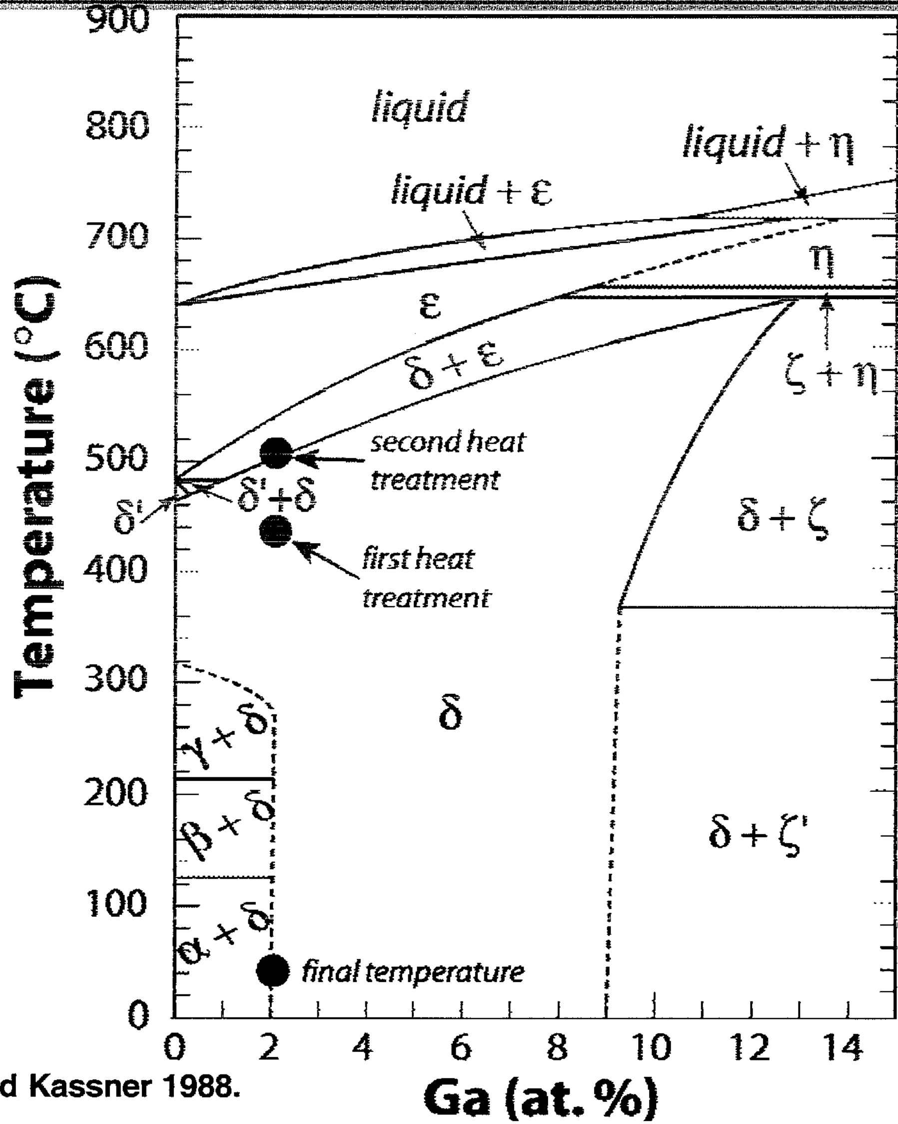
Background

- For Pu Alloys, microstructural stability is an important issu
- OIM is a non-destructive, high-resolution technique for identifying the distribution of crystallographic orientations i multi-phase, polycrystalline microstructures

Objectives

- Obtain EBSD patterns of Pu
- Use OIM for understand microstructural evolution, texture, and phase transformations

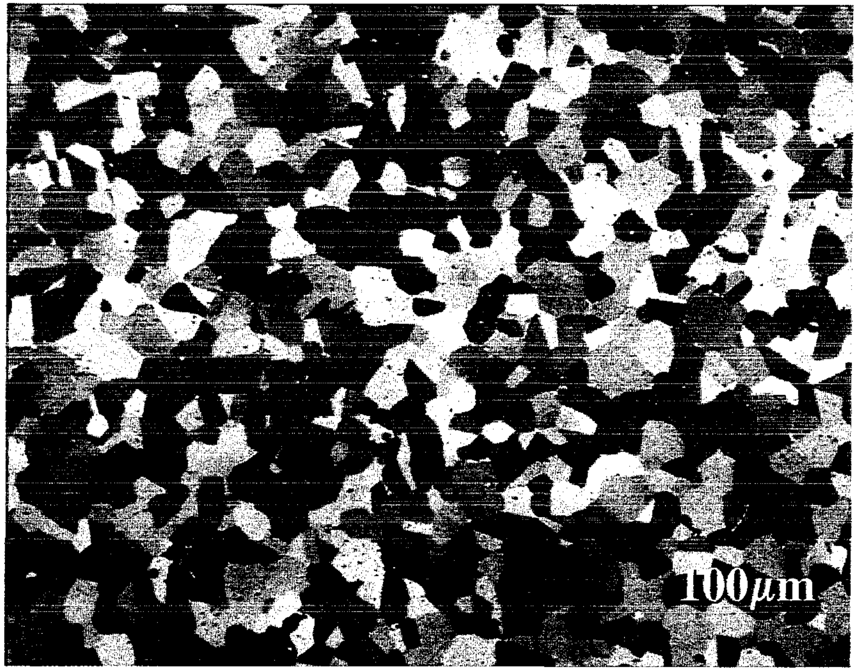
Pu-Ga Binary Phase Diagram

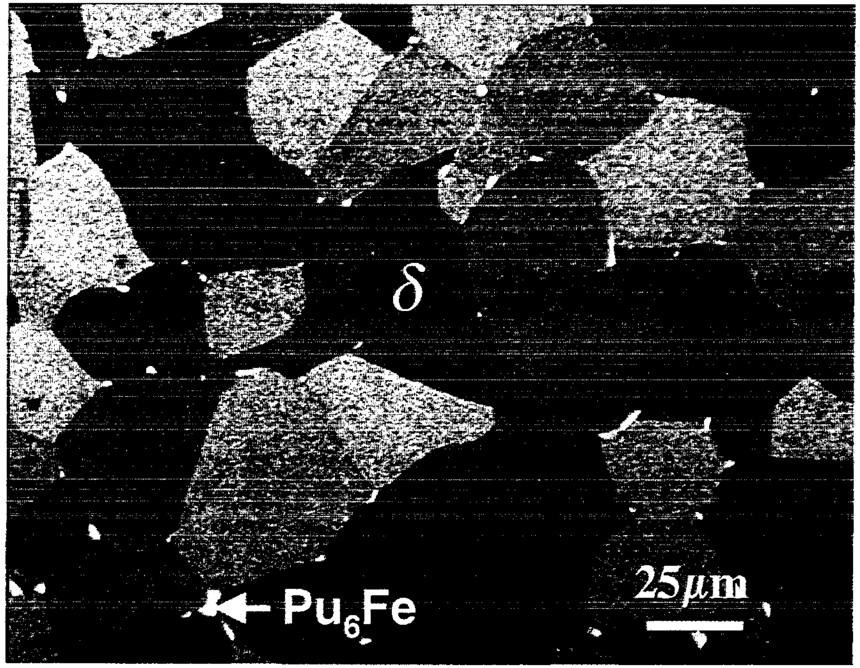


Peterson and Kassner 1988.

Small Ga additions of a few atomic percent form a solid solution that is retained to RT. The Pu-Ga alloy's thermal history is highlighted.

Pu-Ga Alloy Microstructure

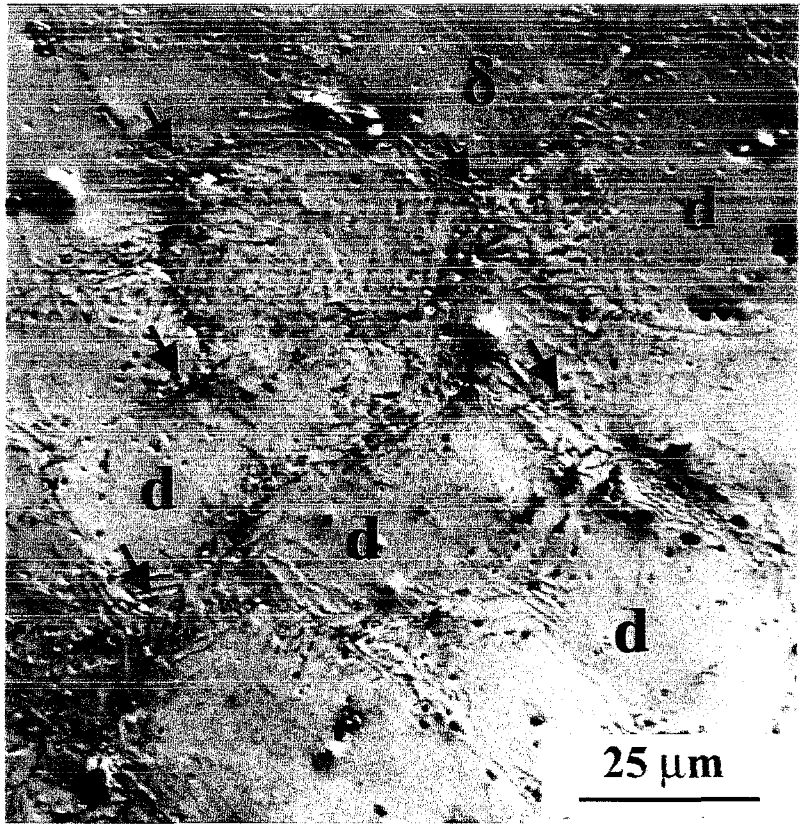




Courtesy of Ramiro Pererya, LANL

After homogenization, the δ-phase grain size was ~50µm. Pu_6Fe was primarily located at grain boundaries and triple points. Smaller contaminates were located throughout.

OM Image of Microstructure



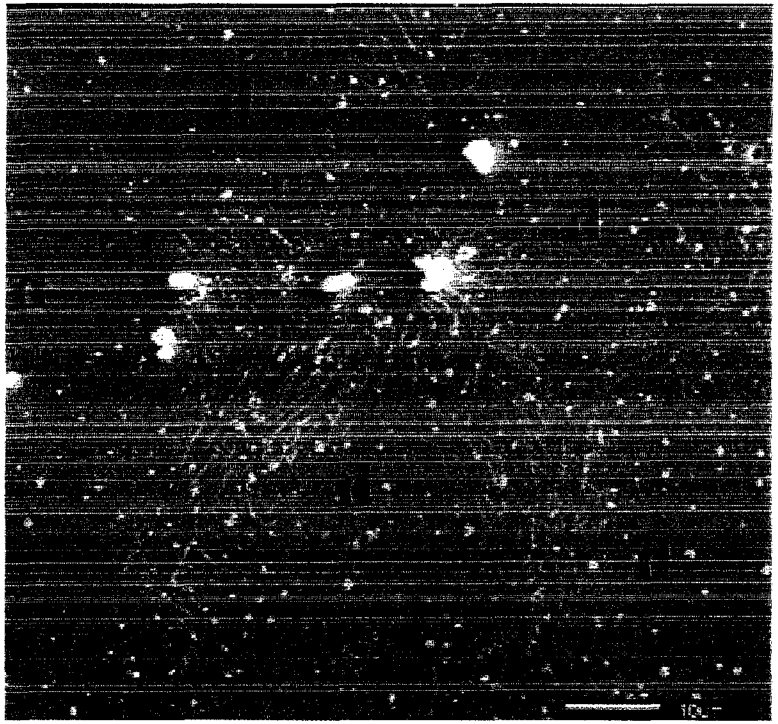
Courtesy of Ramiro Pererya, LANL

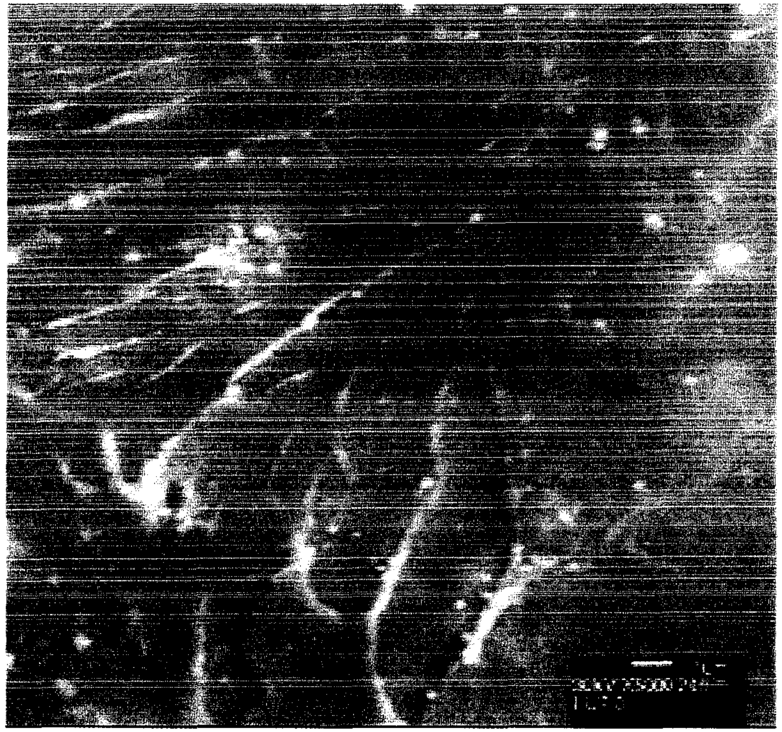
Arrows indicate transformed regios.

Metallography

- Sample diamond cut and mounted in epoxy
- Ground w/180-600grit SiC paper in PF-5070 lubricant
- Polished w/6 and 1 μm diamond paste in De-Solve-It
- Broken out of mount; 530μm thickness
- Electropolished to remove polishing damage layer
  - 10% nitric acid in dimethylformamide @ RT
  - 20Volts, 300mA
- Punched to 3mm diameter
- Placed onto specially designed puck in preparation for ion-sputtering

SEM Images

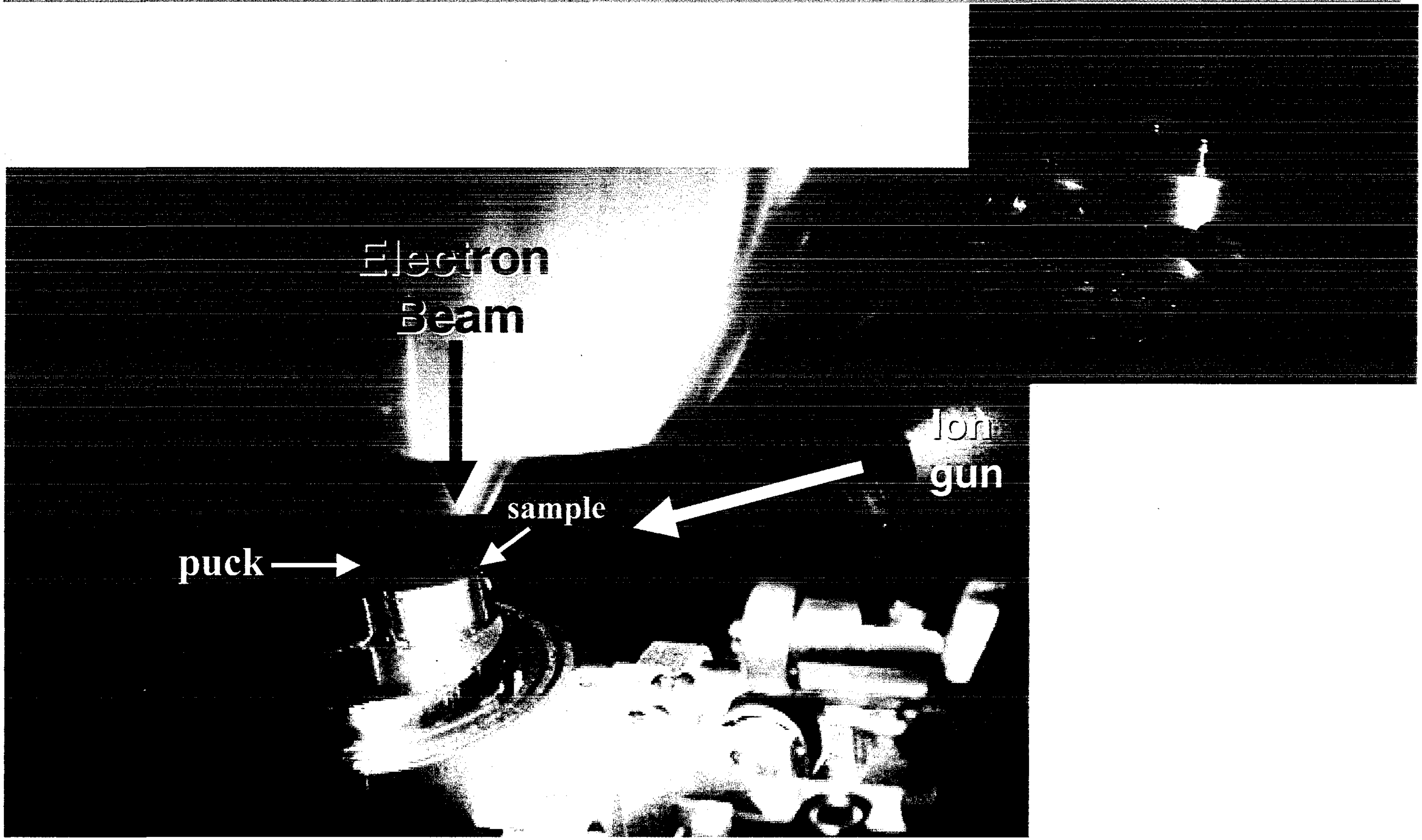




Low Magnification

High Magnification

Scanning Auger Microscope (SAM)

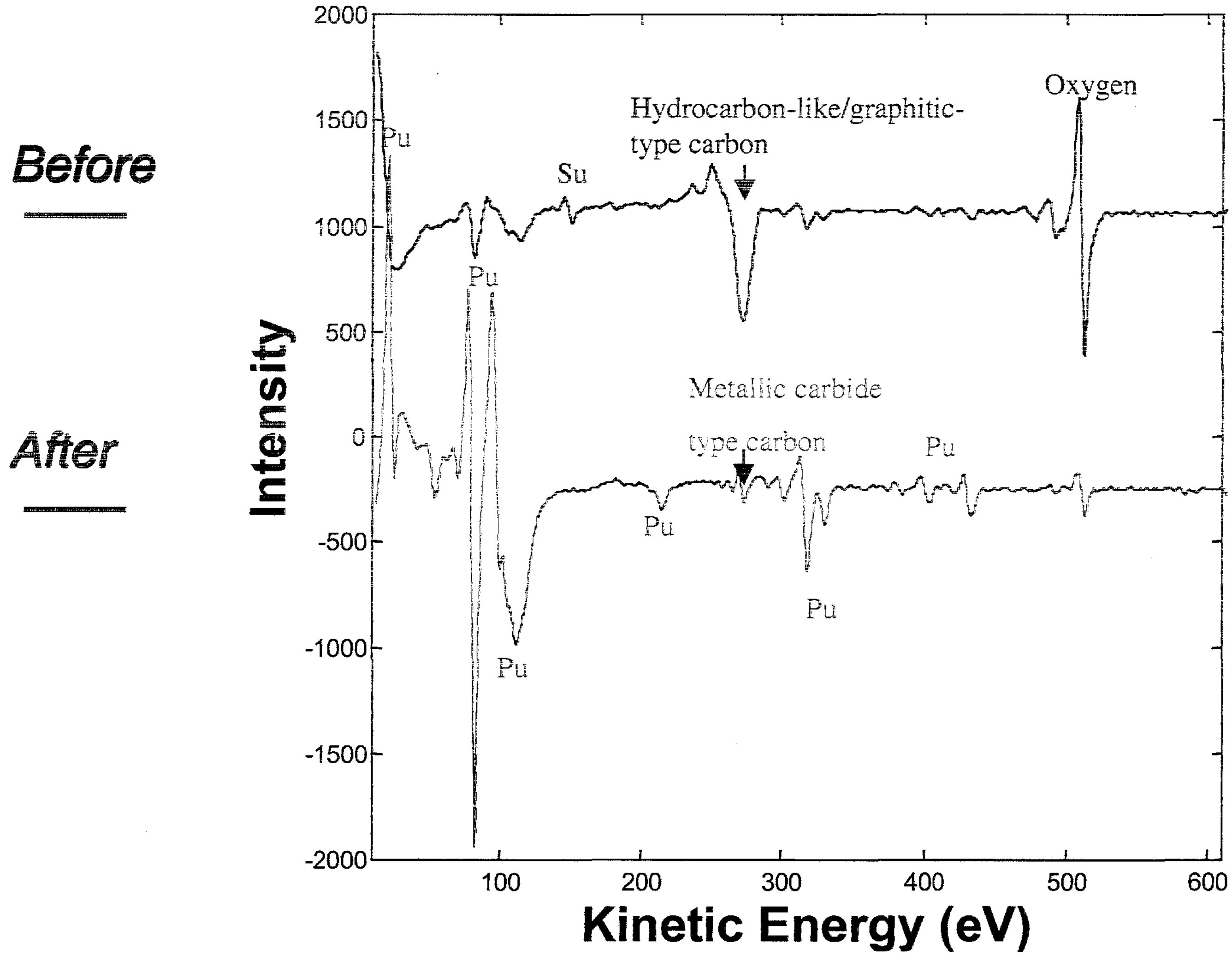


In the SAM, the sample is tilted so that it can be viewed in SEM mode and also sputtered at a variety of angles and conditions.

Sputtering Procedures

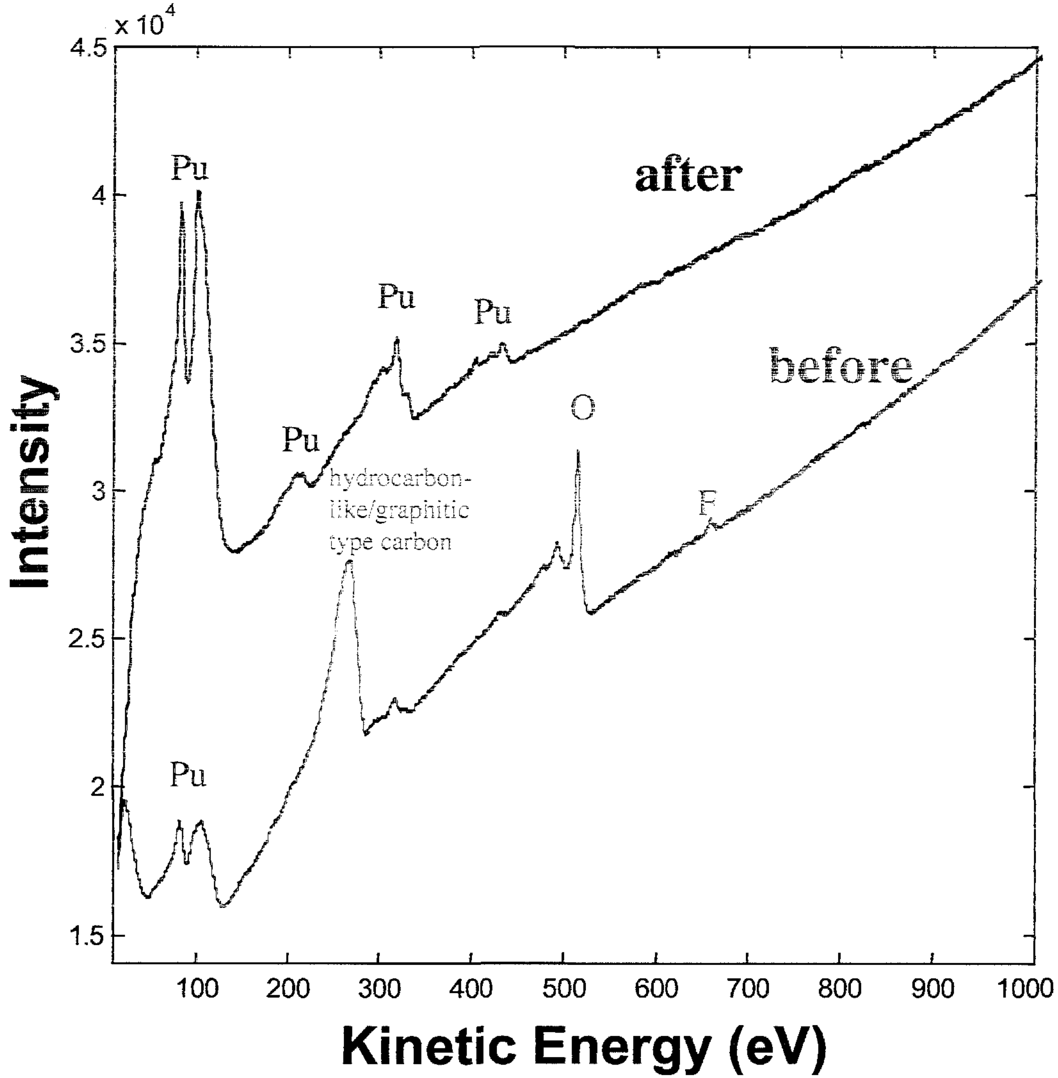
- Monte-Carlo based simulations were used to estimate sputter time required and damage depth
  - 4KeV Ar ions, 40° : ~5nm damage depth
  - 200eV, Ar ions, 20°, 1/3hr:~1nm damage depth
- Flux: 9-55μA/cm^2
- 2x10^{-9} torr atmosphere
- 15° wrt sample surface
- Initial run: 4KeV, 1hr, 3mm x 3mm, 5.5μA
- Finishing step: 500eV, 1hr, 2mm x 2mm

Auger Spectra



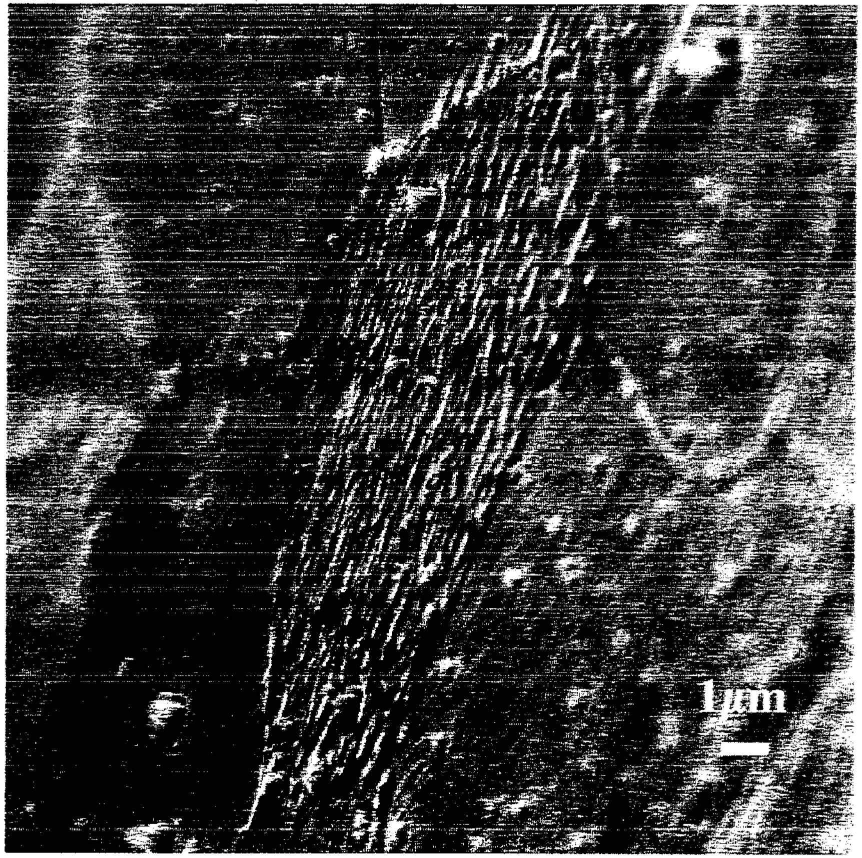
The large C and O peaks observed in the as-electropolished condition were minimized during the Ar ion-sputtering procedures.

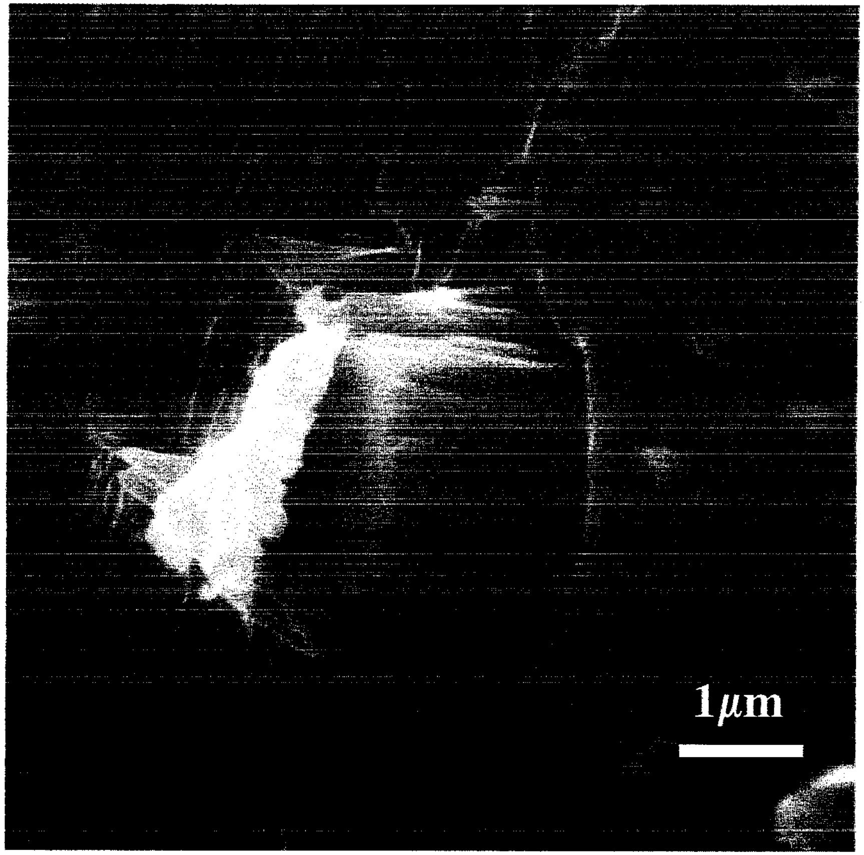
Auger Spectra



The large C and O peaks observed in the as-electropolished condition were eliminated during the Ar ion-sputtering procedures (10min@4KeV followed by 10min@500eV).

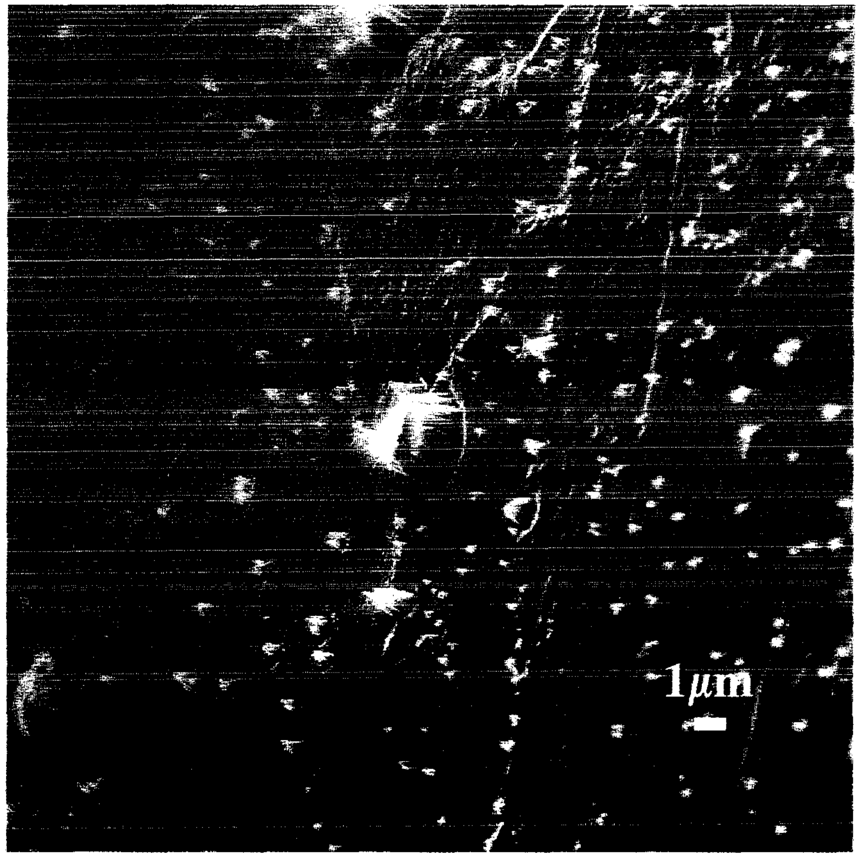
Ion-Sputtered Microstructures



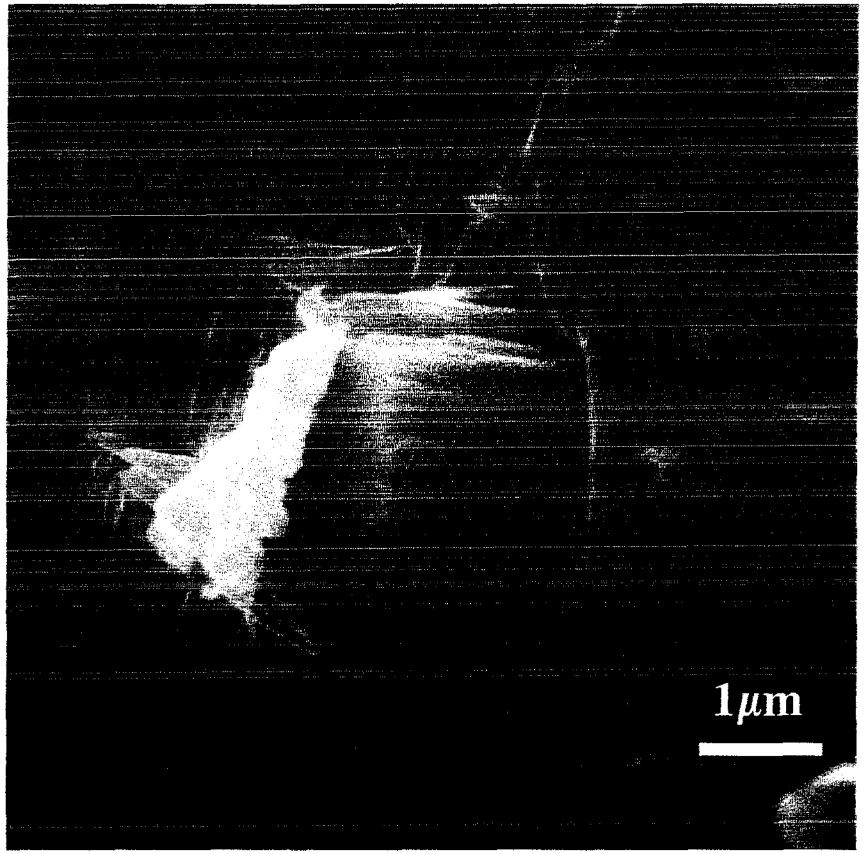


Sputtering increased the grain boundary contrast and the distribution of protrusion heights suggested that subsurface impurities were exposed as the depth of metal removed increased.

Ion-Sputtered Microstructure



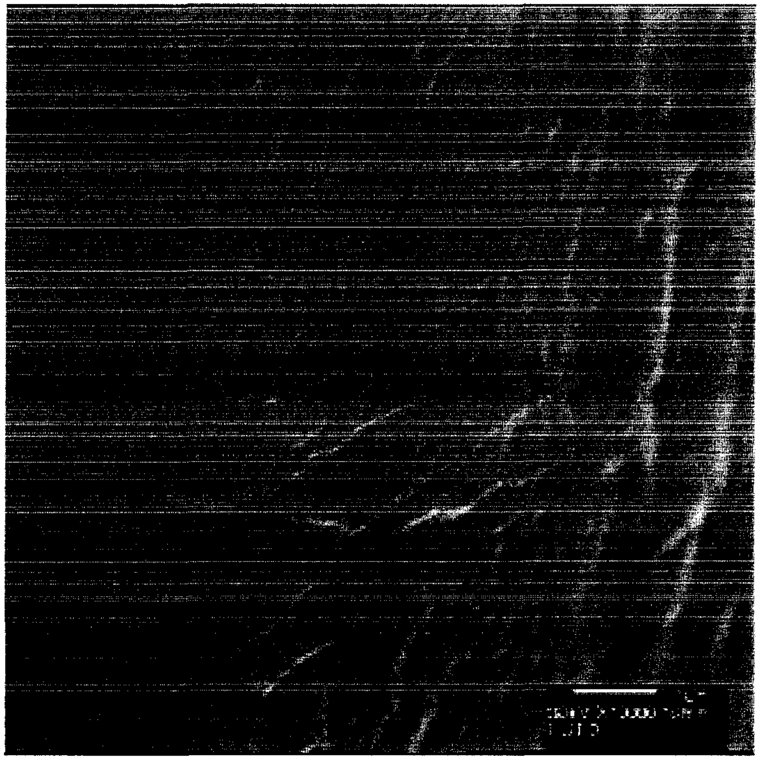
Low Magnification

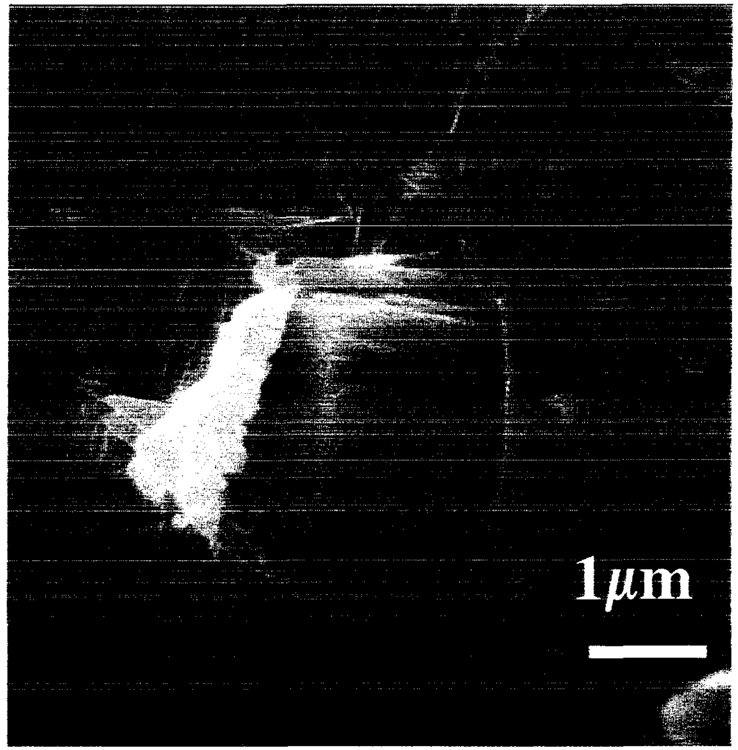


High Magnification

Sputtering increased the grain boundary contrast and the distribution of protrusion heights suggested that subsurface impurities were exposed as the depth of metal removed increased.

Ion-Sputtering



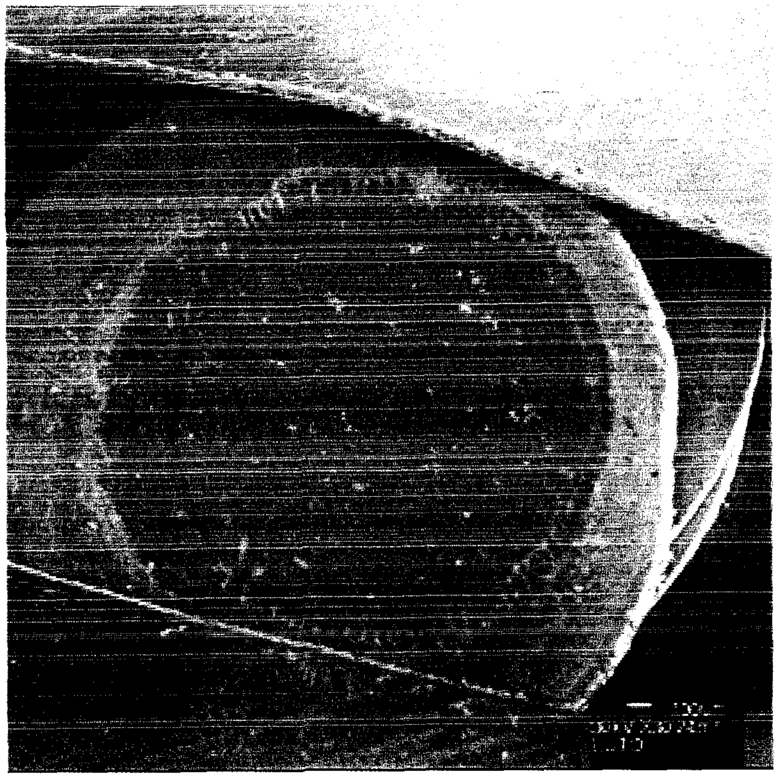


20 minutes

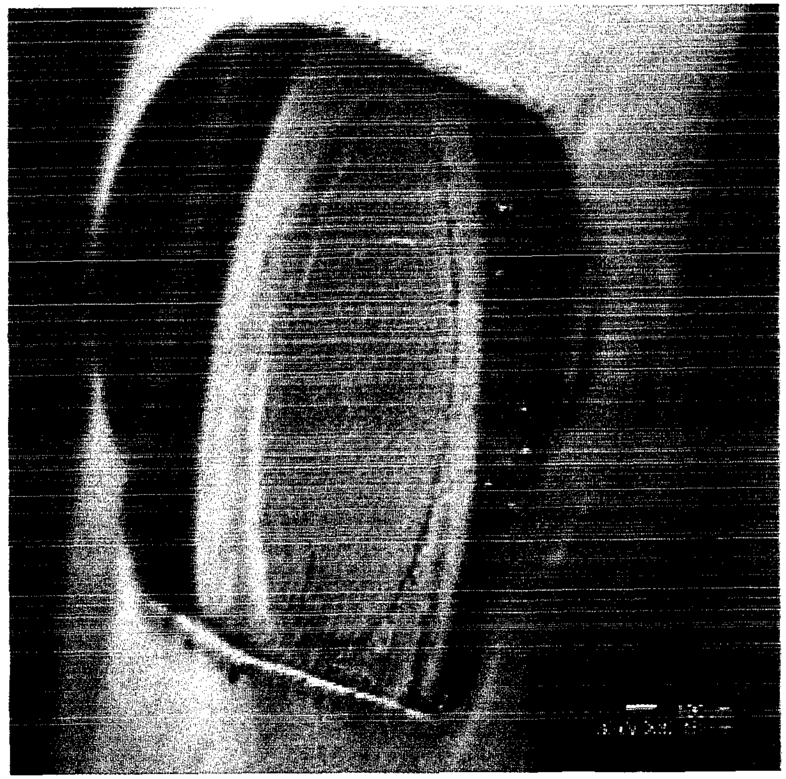
2 hours

Minimizing sputtering reduces surface topography and improves EBSD Orientation map quality.

Electropolished TEM foil



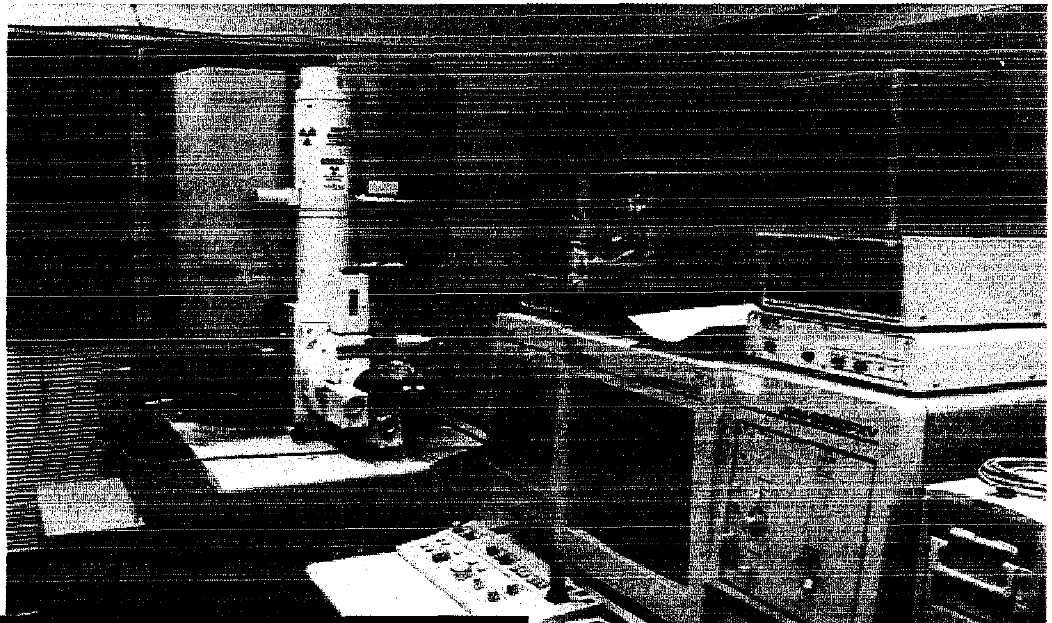
~ normal to beam



~ 70° tilt wrt beam

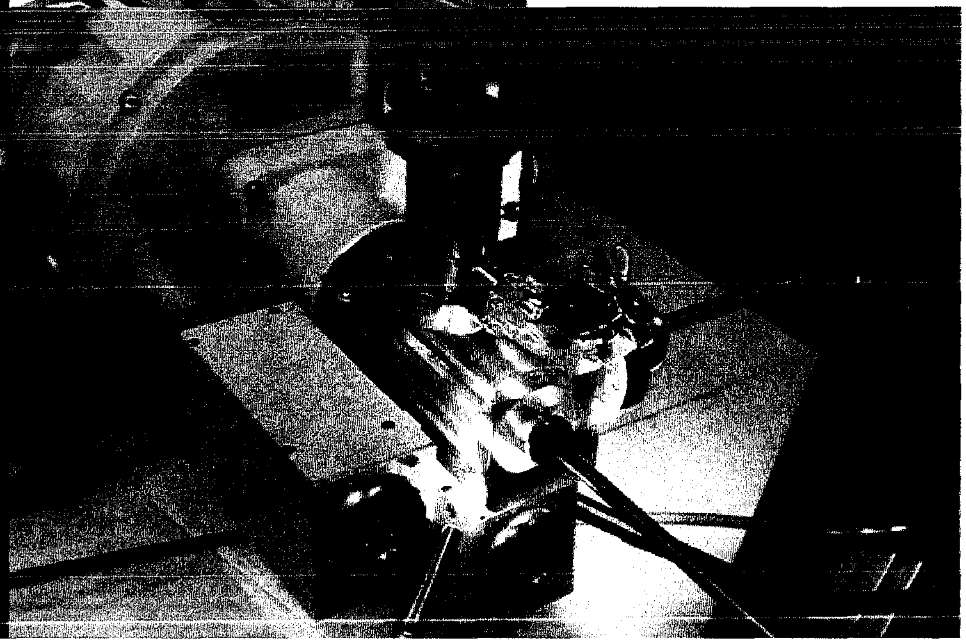
Electropolished TEM foils can be used for EBSD.

SEM Transfer



chamber
$\sim 4 \times 10^{-7}$ torr

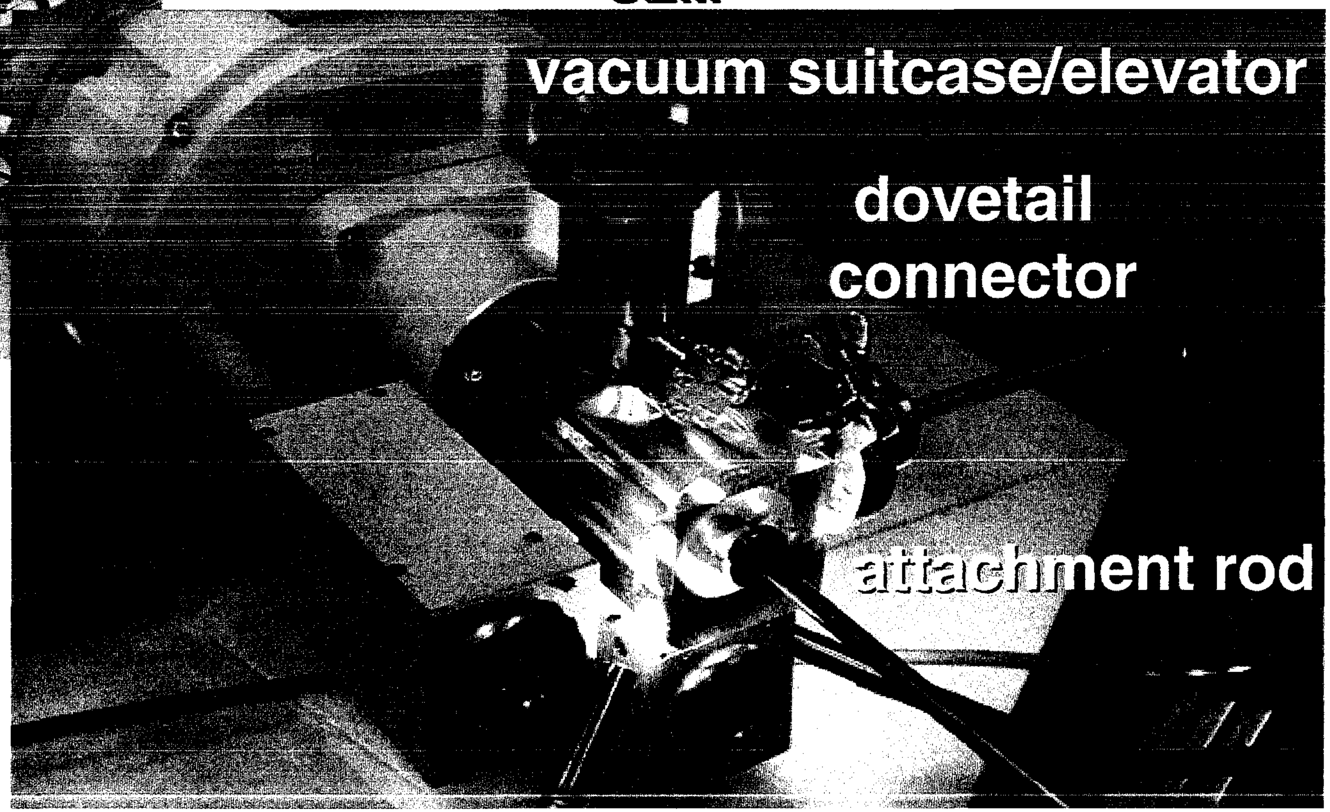
entry port
$\sim 4 \times 10^{-6}$ torr



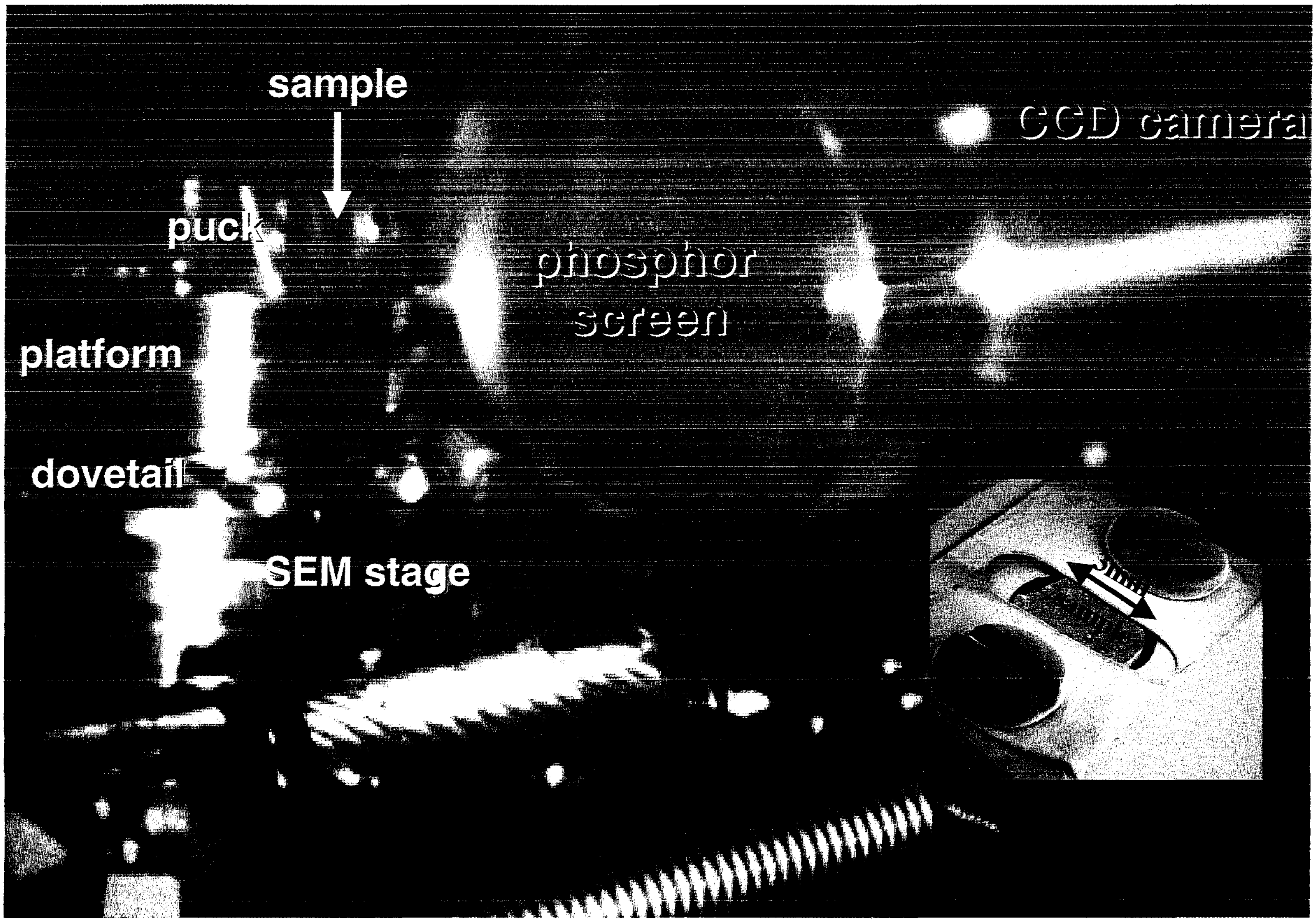
Vacuum specimen transfer device

Auger

SEM



Sample-puck-platform-dovetail assembly



OIM/SEM Parameters

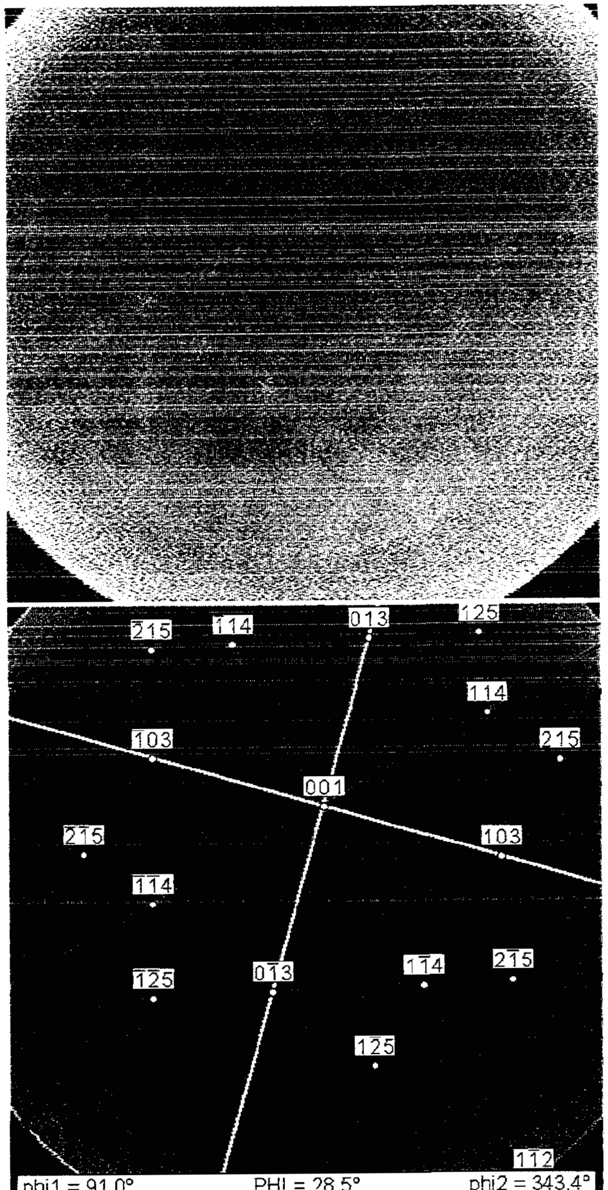
SEM

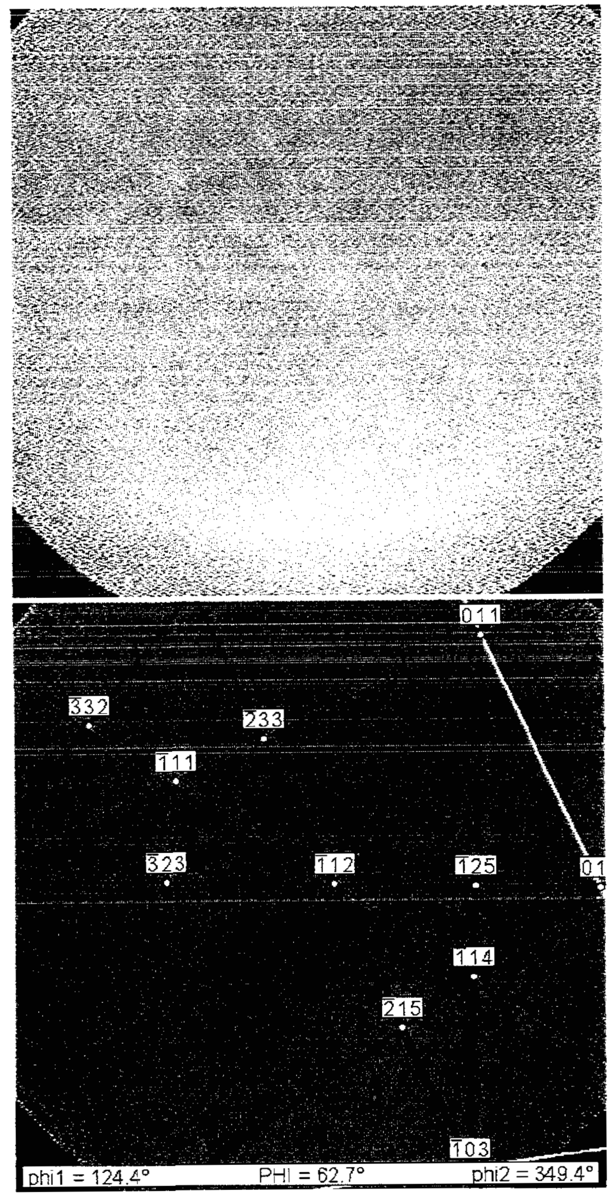
- JEOL 6300FXV cold FEG
- 30KeV, extraction voltage=5V, WD=16mm,
- 110μm obj. aperture, beam dia~2nm
- 12μA emission current; 1-2nA absorbed current

OIM

- 16 frames averaged
- Expected depth of backscattered electrons $\leq$20nm

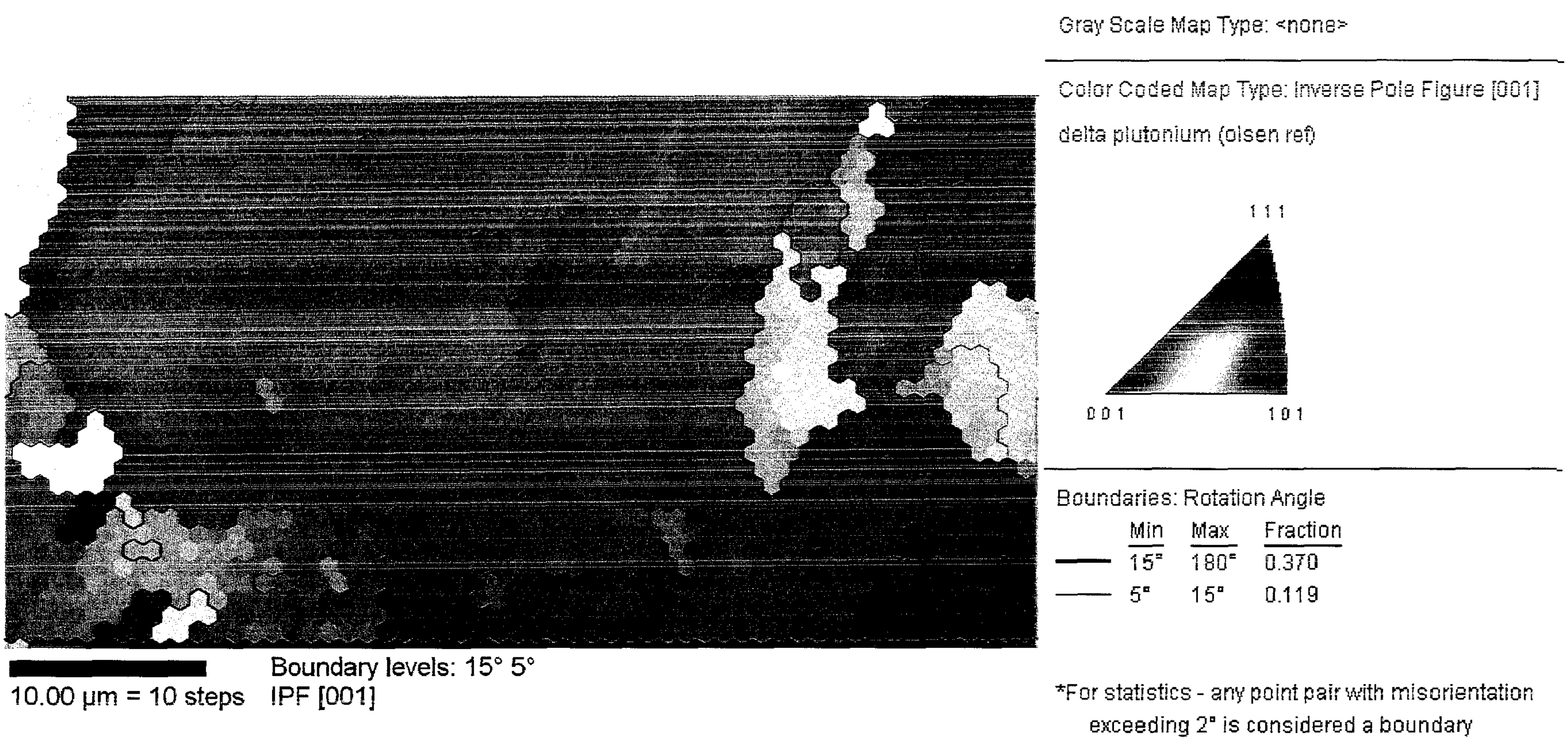
EBSD Kikuchi Patterns





EBSPs and their indexed orientations were captured for several δ-phase grains.

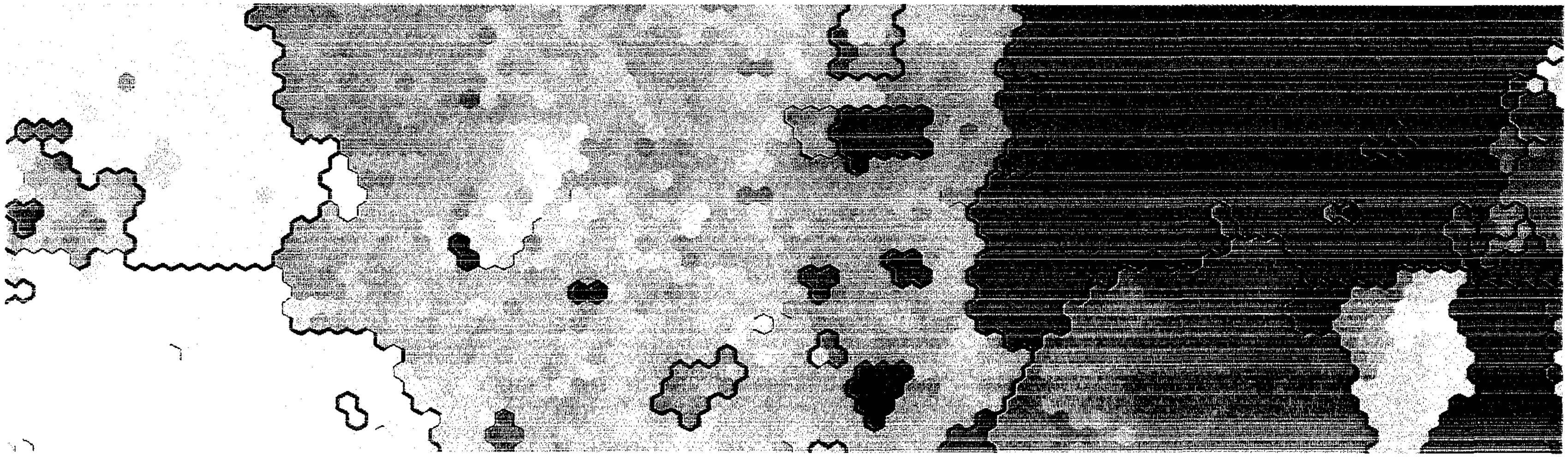
OIM Analysis of Pu



[001] inverse pole figure map

Normal-direction inverse pole figure maps for two different regions of the sample where the colors represent the sample's normal direction indexed to the fcc unit triangle

OIM Analysis of Pu

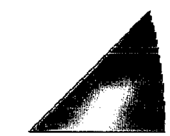


20.00 µm = 20 steps

Boundary levels: 15.0° 5.0°
IPF Map [001]

Gray Scale Map Type: <none>

Color Coded Map Type: Inverse Pole Figure [001]
delta



Boundaries: Rotation Angle

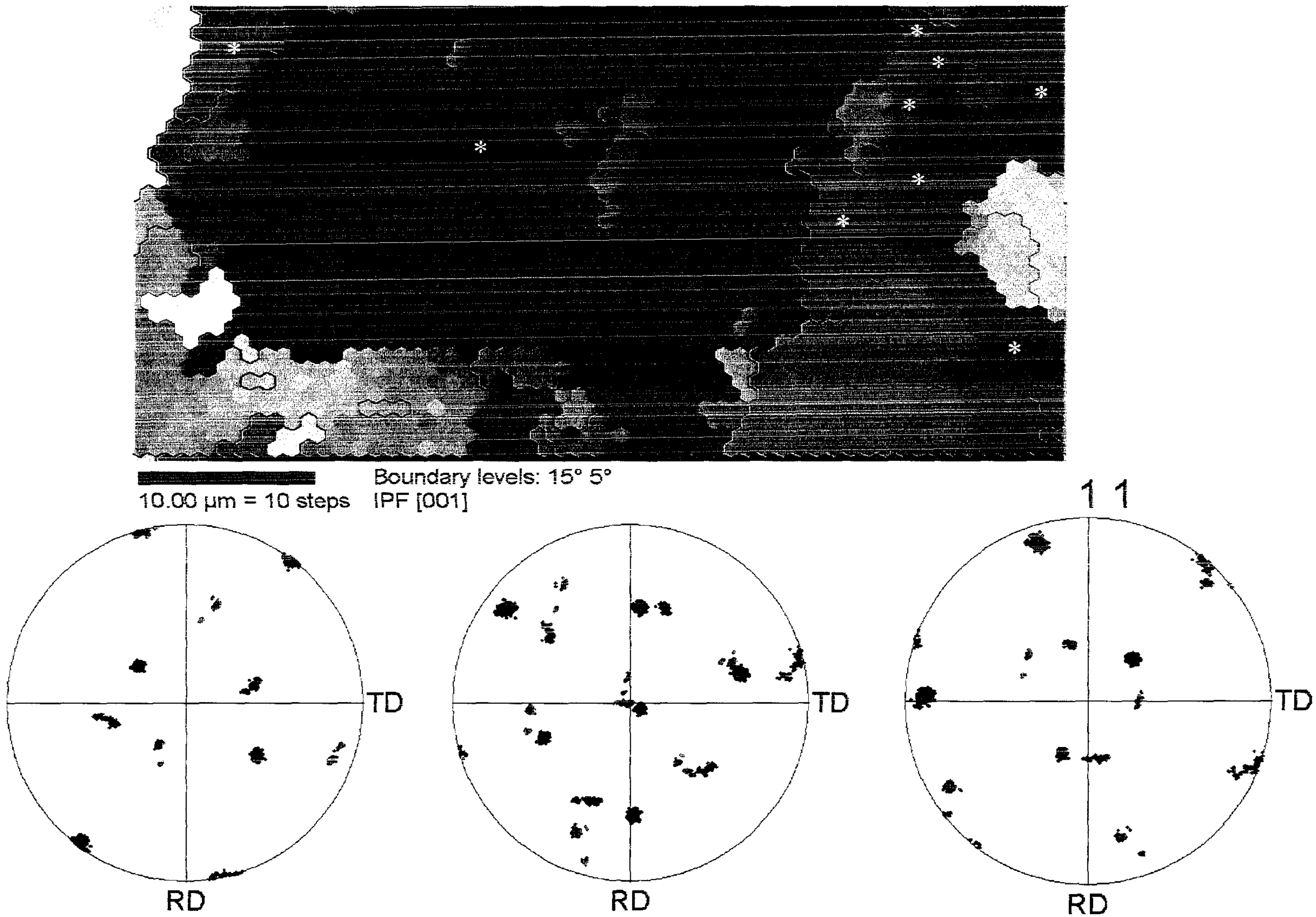
	Min	Max	Fraction
——	15°	180°	0.338
——	5°	15°	0.081

*For statistics - any point pair with misorientation exceeding 2° is considered a boundary

[001] inverse pole figure map

Normal-direction inverse pole figure maps for two different regions of the sample where the colors represent the sample's normal direction indexed to the fcc unit triangle

OIM Analysis of Pu



An orientation map where the orientation component of the marked grains (*) is highlighted by color according to the discrete pole figures.

Summary

- **EBSD Kikuchi patterns were captured for a Pu-Ga alloy. The rapid oxidation rate of Pu in air inhibits EBSD after normal sample preparation. To overcome this, the sample's surface was cleaned via ion-sputtering in a SAM and the sample was transferred to the SEM through a vacuum suitcase.**
- **The EBSD patterns identified the fcc δ-phase grain orientations and suggested a possible δ–ε orientation relationship exists.**