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Three Dimensional Local Electrode Atom Probe Analysis of Microtips Fabricated on a Planar Specimen Utilizing a Broad Ion Beam

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The development of the Local Electrode Atom Probe (LEAP®) by Imago Scientific Instruments is making significant contributions to the speed and breadth of three-dimensional atomic structural and compositional imaging. Due to its localized electric fields, good mass resolution over a large field of view (up to ~100 nm diameter) and high data collection rate (>10,000 atoms/s) [1], the LEAP® is able to analyze microtips made from planar specimens with geometries similar to those first proposed by Nishikawa and Kimoto [2] (Fig.1). Formation of such microtips using ion beam mask etching (IBME) [3] was first realized by Liddle et. al. on an III-V epitaxial layered material [4] and later by Larson et. al. on a variety of specimens [5]. A similar protocol has been used here to fabricate microtips from a planar sample utilizing the technique of IBME. The compositional data collected in the LEAP® were then used to construct a three-dimensional image with approximately one million times magnification and near-atomic resolution.

The sample material used was a small planar section (4 x 4 x 0.85 mm) of a commercial aluminum alloy without heat treatment. The sample was first masked with 30µm polycrystalline diamond particles in a diluted solution and the diamond particles were manually arranged using an optical microscope so as to avoid clustering (Fig. 2). The masked sample was then aligned normal to a broad incident ion beam (~2 mm dia. at sample) by means of a multi-axis stage and milled for 20 hours at a beam setting of 6kV and 8mA. This was followed by a finishing mill of 1kV, 2mA for 10 minutes to minimize any artifacts of ion beam sputtering. Upon completion of the mill, the resulting microtip sample was examined in an SEM to evaluate necessary specimen height (>~50 µm) and tip radius of curvature (<~100nm) required for field ion evaporation.

A number of quality microtips were formed on the planar sample (Fig. 3), with average heights between 50-70µm and tip radii of curvature of ~50 nm (Fig. 4). In excess of four million ions were collected from one of the microtips and the resulting data were analyzed. 3-D reconstruction of the collected data showed a number of small Mg-Si precipitates within the aluminum matrix of the sample (Fig. 5). In future work a number of other milling techniques will be explored; including platinum deposition of arrays of masking dot and broad beam milling of multi-layered films.

[1] T. F. Kelly et. al., "First Data from a Commercial Local Electrode Atom Probe (LEAP)." *Materials and Microanalysis*, **10(3)** (2004) 373.
[2] O. Nishikawa and M. Kimoto, *Appl. Surf. Sci.* **76/77** (1994) 424.
[3] W. Hauffe, in: **Sputtering by Particle Bombardment III**, R. Behrisch and K. Wittmaack, eds. Springer, Berlin, 1991. 318.
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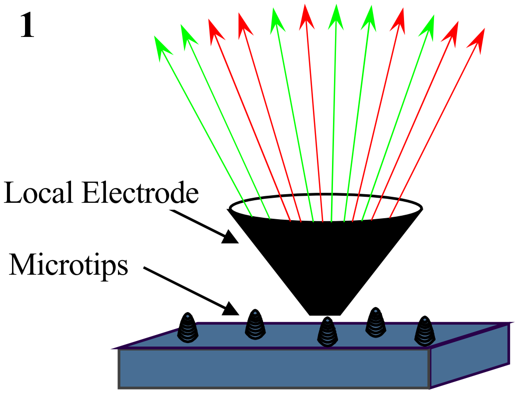
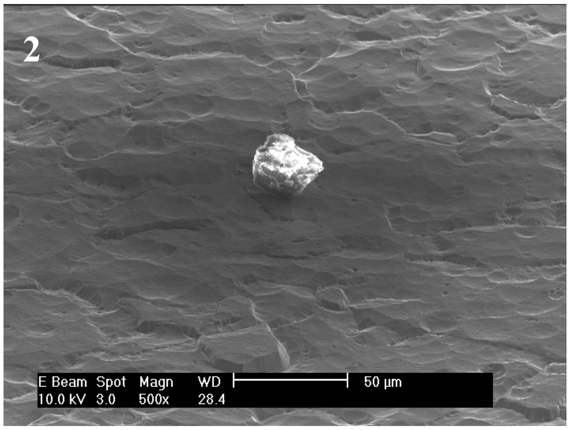
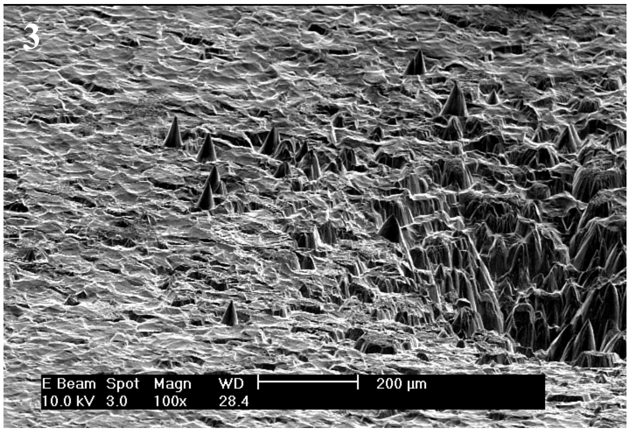
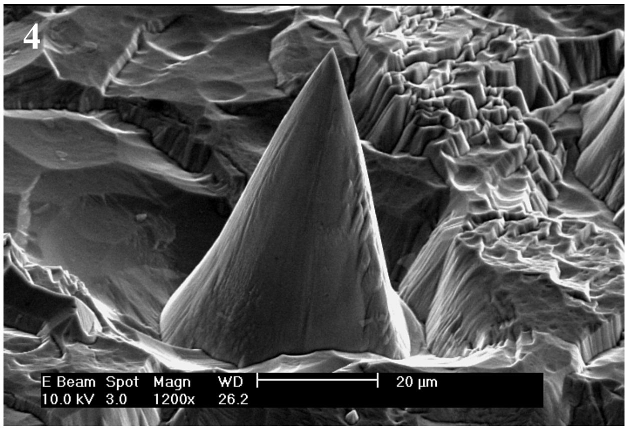
[5] D. J. Larson et. al., *Appl. Surf. Sci.* **87/88** (1995) 446.









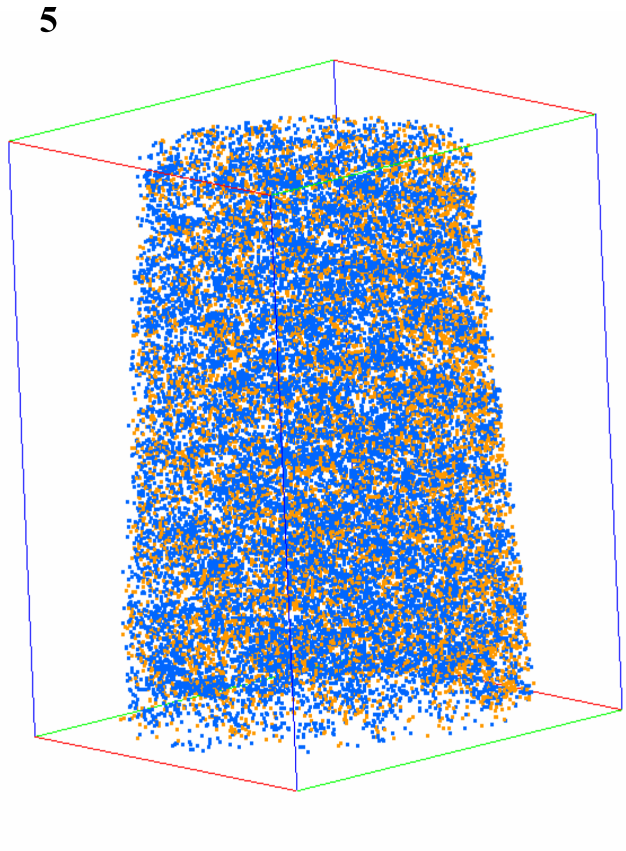


FIG. 1. Schematic of Local Electrode Atom Probe geometry in relation to a microtip on a planar sample.

FIG. 2. 30µm polycrystalline diamond positioned on planar alloy surface.

FIG. 3. Array of microtips upon completion of ion beam milling with sputtering crater caused by ion beam at right.

FIG. 4. Final form of a single aluminum alloy microtip.

FIG. 5. 3D reconstruction of analyzed Mg (blue) and Si (orange) data with a 40 x 40 x 60nm analysis volume.