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Mechanical Precision Preparation of Atom Probe Tips

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The ongoing trend towards engineering materials on a nano-level demands material characterization at the atomic level. For metals, semiconductors and ceramics, atom probe tomography (APT) is a frequently used technique. For many studies, however, much of the effort goes into liberating the region that will later comprise the APT tip from its surroundings or simply picking a region within a material. Precision mechanical grinding, originally developed for micron scaled tensile specimens [1], can be used for site-specific sample preparation reducing the time-per-sample and time-to-data.

The machining tool that provides the shaping is a "morphable" grinding wheel, while the sample is fixed in a rotating chuck. This provides lathe-like grinding, with arbitrary shapes. Figure 1 shows a schematic of the individual sample positioning and preparation steps. It enables site specific preparation at a level where the region-of-interest (ROI) in a microstructure can be targeted without the need of focused ion beam (FIB) milling. The ROI, or the later location of the APT tip, can be defined with a precision of < 50 µm (Figure 2a-c). This is especially useful where either only a small amount of material is available, making traditional electropolishing approaches difficult, or where a ROI such as a near-surface layer or a specific part of a microstructure is to be targeted. In the following, two typical use cases are presented.

Figure 2d-f shows a sample after the APT experiment prepared from a nanocrystalline Fe-based alloy by grinding and electropolishing and the acquired APT data. With the applied grinding geometry, the shape was well defined and reproducible, but slightly deviating from that of a simply electropolished sample with a shank angle close to the tip being consistently between 30-45°. By measuring the tip radius of the sample (Figure 2e), the field factor for the displayed specimen shape is estimated to be 3.17 ± 0.15 using Gomer's field based calculation [2], which is comparable to regular electropolished samples.

The grinding process can be also used to prepare thin films or coatings and materials which cannot be electropolished. Figure 3 shows the preparation steps of a hard coating (TiAlWN) on a tool steel substrate, where the FIB sample preparation time is ~15 min per tip. The intended geometry of the sample can be precisely controlled by grinding. For the hard coating, a truncated cone shape with a flat top of ~25µm was chosen (Figure 3b), which is dictated by the mechanical properties of the thin film. For this FIB prepared sample, an estimate of the field factor k, the image compression factor and the evaporation field of the material could be made by measuring the tip end radius after the APT experiment, the radius evolution and the presence of repeated layers of slightly lower W concentration from high-angle annular dark field images with a known distance of 50 nm. The calculation yielded an evaporation field of 35 V/nm, a field factor of 3.6 and an image compression factor of 1.8. It agrees well with a theoretical value from finite element calculations corrected for a local electrode setup [3,4] and is only slightly higher than the values of 2.9-3.3 often observed for electropolished samples, confirming that this kind of sample geometry does not drastically lower the field at the tip apex.

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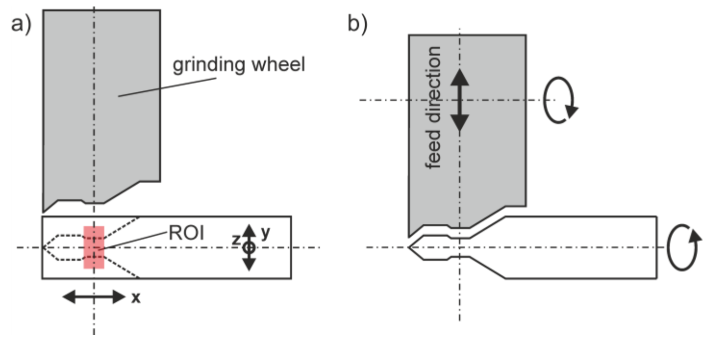

Figure 1. The ROI marked by the red rectangle is aligned to the rotational axis of the sample holder unit in the y-z direction and positioned with the axis of the grinding wheel by a movement in the x-direction. The later shape of the sample after grinding is indicated by the dashed line (a). The tapered section is situated at the exact position of the previously defined ROI. The feed direction of the grinding wheel and the rotational directions of the wheel and the sample holder are further marked (b).

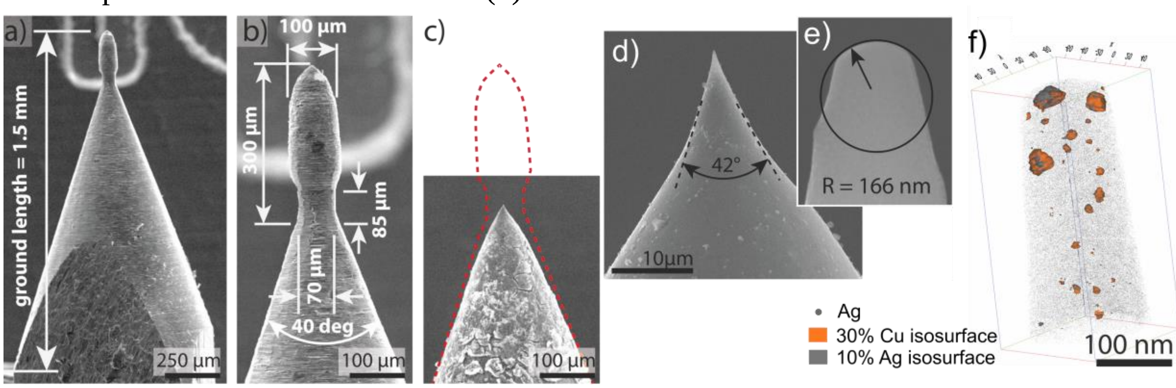

Figure 2. Electropolishing of pre-shaped sample: shape before (a, b) and after electropolishing (c). In (c), the shape of the original sample is marked with a dashed line. Electrostatic environment of the electropolished sample: sample and tip radius (d,e) after the APT experiment and acquired APT data (f).

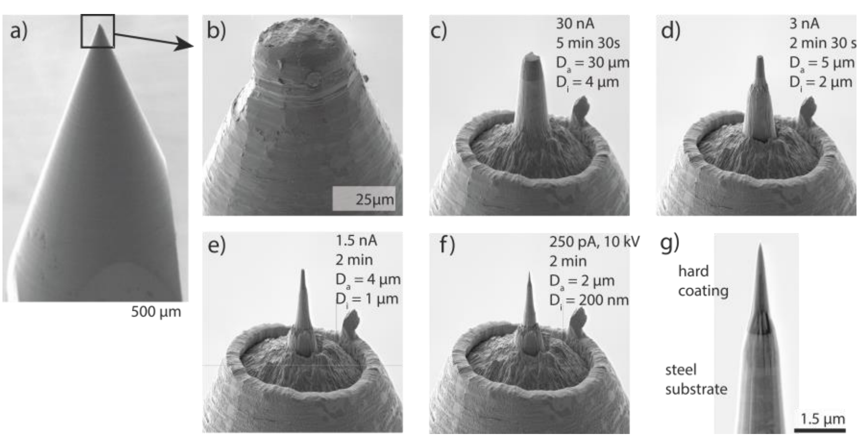

Figure 3. FIB based milling of a pre-shaped sample: Initial shape after machining (a). End shape of the truncated cone (b); note the presence of the coating. Coarse milling steps (c – e). Fine milling step with low accelerating voltage (10 kV) (f). Final tip shape after fine milling (g).

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