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Laser Ablation Sample Preparation for Grain Boundary Analysis of H in Atom Probe Tomography

Martina Heller[1], Veronika Schon[1], Benedict Ott[1] and Peter Felfer[1]

[1.] Friedrich-Alexander-Universität Erlangen-Nürnberg, Materials Science & Engineering, Institute 1, Martensstraße 5, Erlangen 91058, Germany

Atom probe tomography (APT) is a powerful technique to obtain 3D chemical and structural information. The required sample geometry is a small needle shaped tip with a radius of 50-100 nm. Atoms at the apex of the tip are field evaporated by a voltage or laser pulse, accelerated through an electrode and hits a position sensitive detector. With time-of-flight measurement and the x- and y-coordinates of the detector event, a 3D reconstruction of the sample is possible. For the creation of a field emitter from a specific location such as a grain boundary in a poly crystal, usually FIB based lift-out methods are used. This method requires the access to a focused ion beam (FIB) in combination with a scanning electron microscope (SEM) and can take several hours for preparation of a few tips. Within this method, a bar or pillar is cut from the material and region of interest. A micromanipulator can lift the bar/pillar out of the material and place it at the top of a carrier tip. The connection between carrier tip and sample is made with electron or ion beam induced deposition of e.g. Platinum or Carbon. After the lift-out, the final tip milling is performed by the FIB [1], [2]. Although the approach is widely used, it has drawbacks since it consumes a lot of FIB/SEM operation time and additionally adds a possible fracture point in the weld between sample and carrier tip during the atom probe measurement. As a result, the specimens produced by this method have been found to yield little data when e.g. exposed to electrochemical charging of hydrogen into the specimen. Consequently, alternative routes have been pursued.

The combination of a rough sample preparation with a laser ablation system and a final FIB milling is one possible solution. Regions of interest can be located with an optical system. Starting with a rough volume cut pattern, the laser ablates the surrounding material ~500 nm in depth of the sample leaving behind a pillar that includes the target region. An adjacent fine volume cut prepares the tip on top of the pillar with a residual diameter of ~10 µm. White *et al.* showed the influence of heat introduced by the Laser, on the final pillar [3]. Therefore, laser ablation parameters must be chosen with great care and alter the shape of the final pillar. After Laser ablation, the heat influenced material can be removed by a FIB preparation that already includes the final tip milling step to reach the desired tip radius. For this method, no support structure is used, rather a small part of the sample is brought into the atom probe. In the present study we developed dedicated sample holders that facilitates fast and easy transfer between the the Laser cutter, the FIB and the atom probe. Laser and voltage mode measurements are possible if enough clearance is cut around the tips to allow positioning with the instrument microscopes.

This new method is a promising solution to investigate Hydrogen embrittlement in pure iron at grain boundaries. With the laser cutter a site specific preparation of a grain boundary is possible. After a previous surface polishing step, the grain boundary is visible within the laser ablation system microscope enabling exact positioning of the Laser pattern on the top of the sample. After the following

FIB milling of the heat affected zone, the sample is charged with Deuterium and is fine milled with the FIB until the desired radius is reached. With this method a high throughput of samples is produced.

Besides grain boundaries in iron, this method works also for other interesting material systems one of them are Hf-Si-B2 thin films deposited on a Si wafer. These coatings exhibit a good high-temperature oxidation resistance and are of interest for the aviation industry. With this sample preparation method, atom probe tips were milled directly in the Wafer and investigated with atom probe tomography revealing the cluster behavior of Si [4]. Figure 1a and b demonstrate a small part of a wafer with the coating on top and a more focused image on one tip after the laser ablation step. The surrounding heat-damaged material is removed with the FIB and the region of interest, see figure 1c, the Hf-Si-B2 film is visible. The proposed preparation strategy was also applied for polycrystalline cobalt based super alloys with a complex composition that are possible new alternatives for high-temperature materials [5]. To investigate these materials and the segregation behavior of elements, diffusion couples were prepared from two different alloy compositions. The laser ablation enables the efficient fabrication of homogeneously distributed pillars over the entire diffusion zone.

The combination of a Laser ablation system with a focused ion beam offers the possibility to speed up sample preparation while maintaining site specification. In this study we will discuss the advantages and drawbacks of atom probe tips prepared with a laser cutter and present first results of deuterium charged grain boundaries in iron. [6]

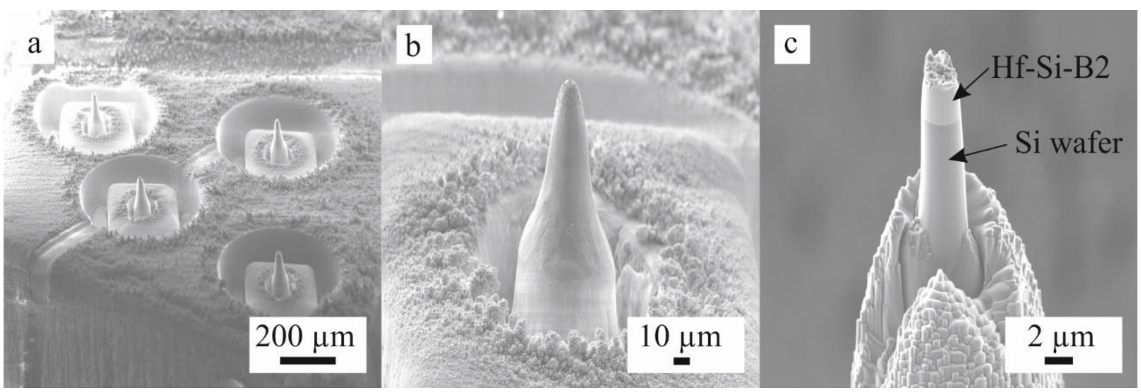

Figure 1. a) An Hf-Si-B2 coated wafer with 4 tips made by a laser cutter. b) Focused image of a pillar after laser ablation, no layer is visible in contrast to c) after FIB milling of the damaged region leaving a tip ready for fine milling of the tip.

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