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Laser Ablation: A New Approach to APT Specimen Preparation

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Laser ablation is a viable technique for atom probe tomography (APT) specimen preparation, and it has the potential to broaden the applications space to specimens which are not conducive to traditional sample preparation techniques. This method offers an alternative to the standard lift-out method of preparing APT specimens, and it provides advantages in time-to-knowledge, large area sampling, and potentially cryogenic applications.

Atom probe specimens must be needle-shaped, with an end radius of approximately 100 nm at the apex. A common method of preparing an APT specimen is the focused ion beam (FIB) lift-out method [1], a workflow almost identical to lamella preparation for transmission electron microscopy (TEM). In a standard FIB lift-out, a wedge of material is removed from the bulk; a micromanipulator is used to move the wedge as small sections are mounted to substrates and propagated, followed by annular milling to create a sharp needle shape. In contrast, the laser ablation method prepares the specimen in place by creating a central pillar of material within a crater. A micromanipulator and, in some cases, even a gas injection system (GIS) are unnecessary because the specimen is prepared by removing material around the region of interest.

ZEISS Crossbeam laser is a LaserFIB system that features a femtosecond laser module attached to a FIB-SEM. The laser allows rapid removal of large amounts of material – with milling rates up to 15×10^6 μm^3/sec for Si, over 20,000 times faster than milling with a Xe plasma FIB – while a Ga ion-based FIB allows precise milling of nanoscale specimens [2]. Laser parameters such as power, frequency, traverse speed, and line spacing can be optimized for either large amounts of material removal or for fine polishing steps [3]. The combination of the laser and FIB-SEM in one system allows for an efficient workflow for this method of APT specimen preparation.

Specimens from a polished Inconel® sample were prepared in the LaserFIB and quantitatively analyzed successfully in a CAMECA LEAP® 5000 XR. The specimens were prepared by first using the laser to form a crater with a central pillar on the order of 30 microns in diameter, shown in Figure 1a. A Ga ion beam was used for annular milling and final shaping at 30 kV and 5 kV, respectively. Figure 1b shows the final prepared specimen ready for atom probe analysis. Data acquisition was performed in both voltage and laser pulsing modes in the LEAP. Figure 2 shows the reconstruction and mass spectrum from a laser pulsing experiment of the laser-prepared specimen.

Laser ablation offers multiple advantages over the traditional lift-out process. Initial craters require less than one minute of laser milling, over 100 times faster than using a plasma-based ion source, providing substantial time savings over current in-place methods. Specimens can quickly be prepared from different areas of the sample, in contrast to the lift-out technique where specimens are typically from the same 10-20 µm long wedge. This is useful in large area sampling for quality control monitoring, for example.

Laser ablation could also provide new advancements for cryogenic applications. Preparing and analyzing specimens under cryogenic conditions is important for the investigation of hydrogen embrittlement [4], aqueous materials [5], and other applications. Cryogenic specimen preparation is challenging with a traditional lift-out due to uncontrolled gas precursor condensation when mounting specimens to posts. Laser ablation prepares the specimen directly in the bulk sample without requiring a GIS, so this challenge is avoided.

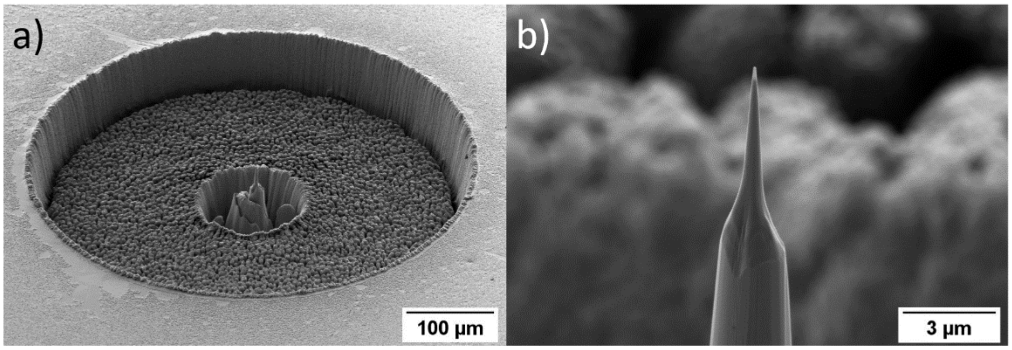

Figure 1. a) Laser ablated crater of Inconel with an APT specimen sharpened with Ga ion milling. b) Inconel APT specimen shown at higher magnification.

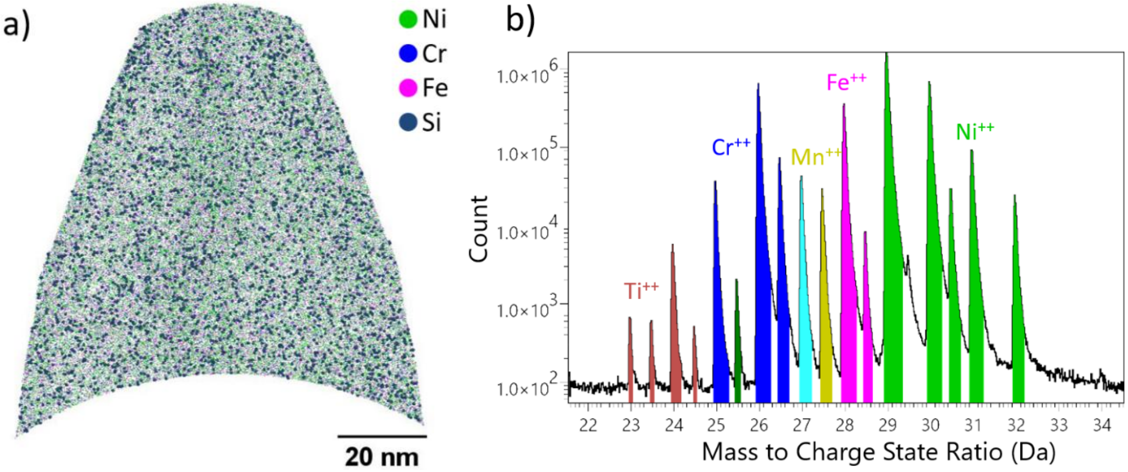

Figure 2. a) Reconstructed data and b) mass spectrum of laser prepared Inconel from laser pulsing experiment in LEAP 5000.

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