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Anup Sharma1,2,*, Levi Tegg3, Aristide Djoulde2, Deepak Marla1, and Jing Fu2

1Department of Mechanical Engineering, Indian Institute of Technology Bombay, Mumbai, Maharashtra, India
2Department of Mechanical and Aerospace Engineering, Monash University, Melbourne, VIC, Australia
3School of Aerospace, Mechanical and Mechatronic Engineering, University of Sydney, Sydney, NSW, Australia
*Corresponding author: anup.sharma@monash.edu

Resin embedding has been widely used for electron microscopy of biological samples and recently has found application in atom probe tomography (APT) analysis of soft matters including nanoparticles [1]. Focused ion beam (FIB) lift-out is the most prevalent method for preparing site-specific TEM lamellas and needles for APT specimens. Although widespread, FIB lift-outs are time-consuming and require a highly skilled operator [2]. Additionally, during probing, the chances of failure at the platinum joints in lift-out are also considerable. Directly preparing needle specimens from bulk sample enhances structural integrity, minimizes fracture risk during APT data acquisition and facilitates easier handling. Laser machining can provide a quick way to produce micrometer-scale features for most materials and hence can be ideal for performing coarse machining of microscopy samples [3]. This article presents a novel workflow to rapidly prepare APT specimens from bulk resin samples using laser ablation for coarse machining and FIB for final nano machining.

Figure 1 depicts the proposed sample preparation approach, which can be summarized as follows: The resin mixed with the hardener is left to cure for around 24 hours at 60°C in a gelatine capsule. 150-200 µm thick slices are precisely cut from the capsule shaped resin block using a cut-off machine (Accutom-100), producing the sections shown in figure 1(a). A laser micro-milling machine (3D Microprep) equipped with a picosecond pulsed laser cuts half-grids of multiple pillars from these thin resin slices. Figure 1(b) illustrates the half-grid placed in an upright position to perform laser annular milling of the pillars to reduce the diameter to 50 µm.

The half-grid laser cut (figure 1a) is performed at 0.3 W of average laser power with a pulse energy of 60 µJ. The annular milling of the pillars (figure 1b) is performed using a lower power of 0.05W. The laser process parameters are selected to reduce heat diffusion into the bulk resin to avoid thermal damage to the core of the specimen. To enable precise machining with low heat affected zone, the number of repetitions of the patterns is kept high with low laser power. Figure 2(a) depicts the laser cut half-grid of a resin sample with nanoparticles embedded. FIB machining is performed using a FEI Quanta SEM-FIB instrument with the half-grid positioned upright as shown in figure 2(a).

The APT of the resin nanoneedles is performed on a Cameca LEAP 4000. A Cr coating of 10 nm is applied after the final machining to enable electric field delivery to the apex of the specimen during APT analysis. Around 8 million detections were made of the sample with region clearly identified as conductive coating, nanoparticle embedded and resin. The 3D reconstruction of the specimen is shown in the figure 2 (b). This novel workflow provides a seamless method for producing nanoscale specimens from bulk resin which paves the way for easier and time-saving characterization of samples like cells, and nanoparticles with resin embedding. A similar procedure with minor modifications can be applied for preparing Transmission Electron Microscopy (TEM) specimens from resin and accelerate the sample preparation process.

Fig. 1. Workflow for preparing APT specimen of resin. (a) 200 µm thick resin slices are prepared with Accutom precision saw from resin block. (b) Laser machining is used to cut-off half grids from the resin from specific location. Laser annular milling is used to prepare pillars of around 50 µm diameter. (c) FIB machining prepared the final nanoneedles for APT. The diameter of the half-grid is 3 mm with a total height of 2.1 mm.
Fig. 2. (a) SEM image of resin half-grid cut by laser with three micro-pillars, (b) 3D Reconstruction obtained from the APT of needle prepared from resin with ferrite nanoparticles embedded (blue and magenta) and chromium coating on top (orange).

References