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# Creating Polymer Microtip Specimens for Atom Probe Tomography

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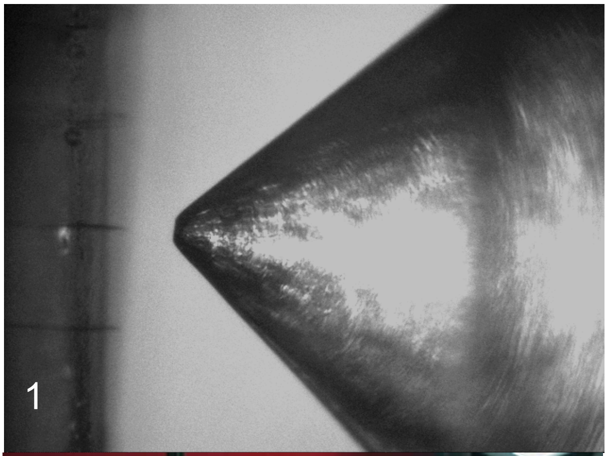
The local electrode atom probe (LEAP®) [1] geometry enables analyses of multiple microtip specimens fabricated from a planar surface, Fig. 1. However, the preparation of polymeric, biological and particulate specimens for such analyses remains a challenging task. One general approach to solving this problem is the use of mold-replication techniques. We report here the first successful efforts at forming microtip specimens from polymers.

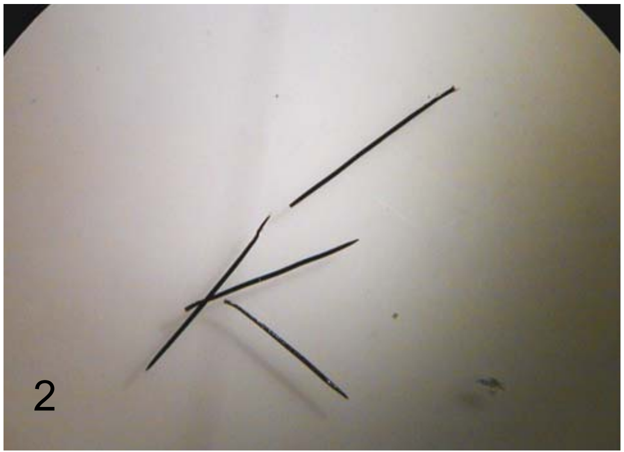
In addition to shape, a significant issue with analyzing organic material in atom probe tomography is electrical conductivity. A potential solution is to embed the organic material in a matrix of conductive material. Although most polymers are not electrically conductive, many intrinsically conductive polymers (ICP) are used in various industries for applications such as antistatic surfaces on television screens, flexible electrode materials and polymer light-emitting diodes. In this study, a conductive polyurethane dispersion (CPUD2 [2]) was chosen for its low viscosity and ability to form a thin, uniform coating on a surface. To form the specific specimen shapes required for LEAP, polydimethylsiloxane, a silicone rubber, was used to prepare a mold due to its ability to replicate fine features and to withstand the 50°C temperature needed to cure CPUD2. A micro-centrifuge tube was used to contain the silicone mold. Initial experiment molds were made from several sizes and types of needles. The polymer filled these molds well, but the needles of polymer were undesirably long (~1-2cm) and flexible, Fig. 2. The tip radii were relatively large (~3-10μm), which necessitated long milling times in a focused-ion-beam (FIB) tool. In addition, this process was time intensive and only created a few good specimens.

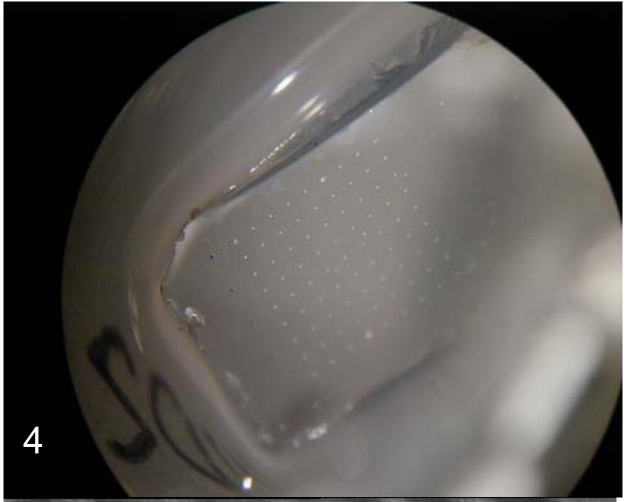
In the interest of making many identical specimens in parallel, a silicon substrate with a nine-by-nine array of atom-probe-sharp tips [3] was chosen to create a new mold. The microtip coupons, attached to copper stubs were slowly dipped into the silicone and held in place with alligator clips, Fig. 3. These were set aside to cure for twenty-four hours before removing the silicon substrate, Fig. 4. The CPUD2 was poured into the mold, capped and microcentrifuged for 30 minutes at room temperature and 3 RCF (relative centrifugal force). After curing, the CPUD2 replica was removed, trimmed and epoxied to a copper stub, Fig. 5. The extraction of the specimen from the mold must be done slowly and in a linear fashion. The resulting microtips' radii were ~ 2-3μm. With slight FIB milling using annular mill patterns [4], they were ready for attempted LEAP analysis, Fig. 6.

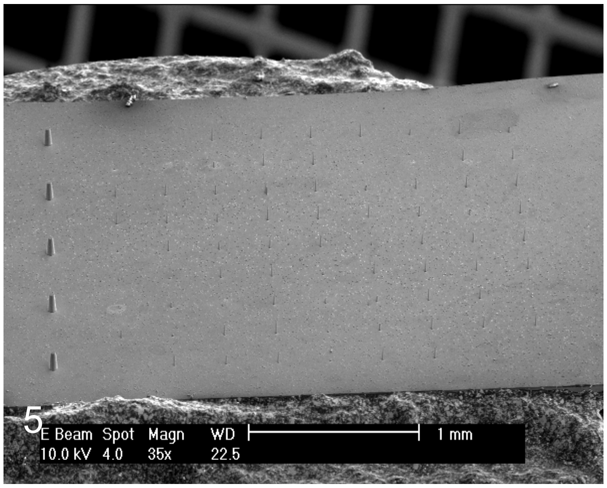
This specimen molding technique has the potential to be a relatively quick, simple and repeatable process for creating multiple specimens, enable the encapsulation of particulate specimens within a conductive matrix [5] and enable atom probe analysis of these soft materials.

[1] T. F. Kelly et al., *Microsc. Microanal.*, 10 (Suppl 3) (2004) 373.
[2] H.C. Starck Baytron® CPUD2
[3] K. Thompson, D. J. Larson, R. M. Ulfig, *Microsc. Micronal.*, this volume.
[4] D. J. Larson et. al., *Ultramicroscopy* **79** (1999) 287.
[5] S. L. Goodman et al., *Microsc. Microanal.*, 9 (Suppl 3) 1186CD.









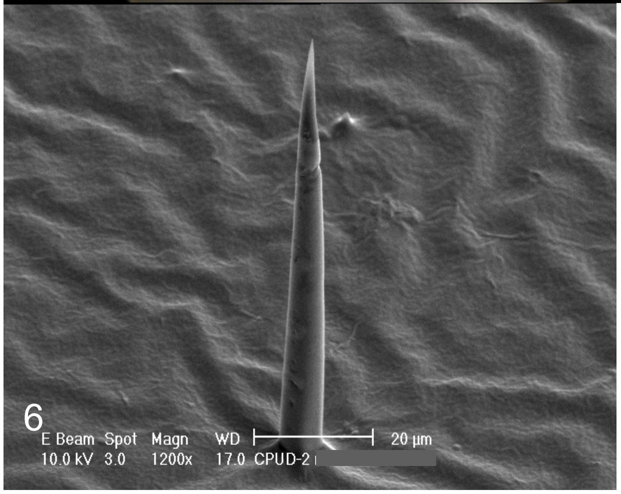


Fig. 1. View of local electrode and planar specimen of molded microtip CPUD2. Tips ~ 100um tall.
Fig. 2. Macroscopic image of molded needles of CPUD2.
Fig. 3. Manufacturing of microtip coupon mold from silicone.
Fig. 4. Silicone mold post-removal of microtip specimen.
Fig. 5. SEM image of molded CPUD2 specimen.
Fig. 6. SEM image of molded CPUD2 needle from microtip coupon ready for LEAP analysis.