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A Method for FIB Liftout of Particles in Epoxy Resin

Warren L. York[1*], Dave Robinson[1], and Joshua D. Sugar[1]

[1]Sandia National Laboratories, Livermore, CA, USA.
* Corresponding author: wlyork@sandia.gov

Preparation of electron transparent samples from small particles can be extremely challenging, even when using the Focused Ion Beam (FIB). In particular, when the dimensions of the particles are less than a typical liftout (<10um) and are loosely packed, a uniform thin area of several particles can be difficult to achieve. One way to overcome this is to use the carbon deposition available in the FIB to encapsulate particles. However, in our case, this caused a variety of problems because the C did not penetrate into all of the pores between particles. This caused severe "curtaining" (fig. 1c) and redeposition of material on the particle surfaces. Epoxy resin is a good alternate because the voids between the particles can be removed through proper vacuum encapsulation procedures, but the differential milling rates of the powder and resin material can cause additional problems. Presently there are several small particle TEM preparation techniques described in the literature, for example reference [2]. Here, we investigate how to minimize Ga-induced beam damage and prepare a site-specific specimen of a particle cluster or individual particle in a cold setting epoxy resin we had on hand.

The sample of interest was a Pd powder and cold setting epoxy resin. In our case, the resin we used caused challenges in milling because it would bend while thinning and cause non-uniform milling of the powder material. When the usual recipe for thinning was followed at 30 kV with the usual current and window size combinations, the sample bent like a "potato chip" (fig. 1e-f). Multiple less aggressive conditions were investigated to try to eliminate these unwanted effects. Parameters that were varied included kV, current, beam overlap and thin window size. We found that lower initial kV such as 16 kV and a smaller window were the best combination for this type of beam sensitive sample. (fig. 2). The sample was then thinned down to the appropriate (near transparent) thickness using the 5kV beam on the FIB for TEM.

The resulting thin sample is shown in figure 2. Figure 2c) shows a 30 kV STEM-dark field image taken in the FIB. The grain structure and individual dislocations are clearly visible in the image. The particles were thin enough for TEM analysis and each of the particle structures are clearly visible in the image. A set of 300 kV STEM images are shown in Figures 2d-f), where the particles are easily visible. A high-quality result is highly dependent on stopping the thinning process as soon as the protective layer has been removed and removing any high-kV beam damage. This technique provides high-quality TEM results and can be used to study particles in epoxy resin [3].

References:

[1] LA Giannuzzi and FA Stevie, Micron **30** (1999), pp. 197-204.
[2] JM Cairney and PR Munroe, Materials Characterization **46** (2001), pp. 297-304.
[3] Sandia National Laboratories is a multimission laboratory managed and operated by National Technology and Engineering Solutions of Sandia LLC, a wholly owned subsidiary of Honeywell International Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525

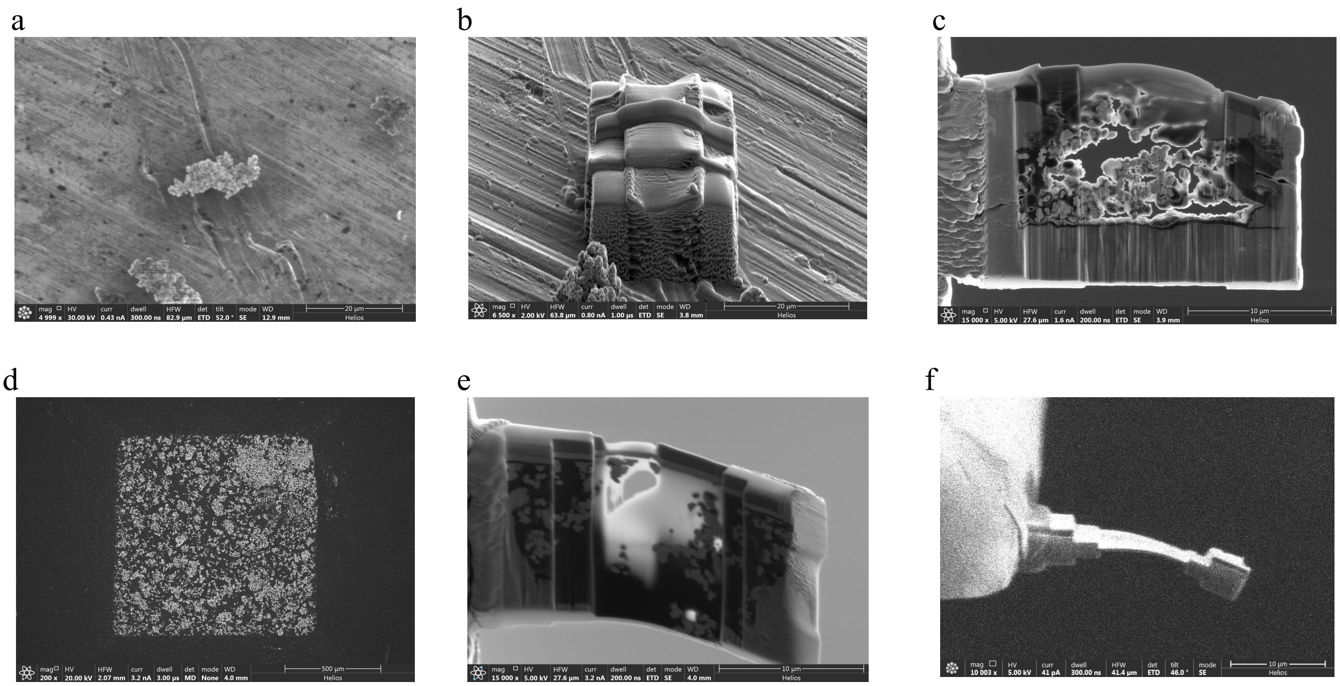

Figure 1. (a) SEM image of powder before encapsulation in Carbon dep. (b) SEM image of powder encapsulated in Carbon dep. (c) SEM image of thinned sample after liftout from Carbon encapsulated sample. (d) SEM image of powder in epoxy resin. (e) SEM image of sample after final thin. (f) FIB view of sample showing "potato chip" shape

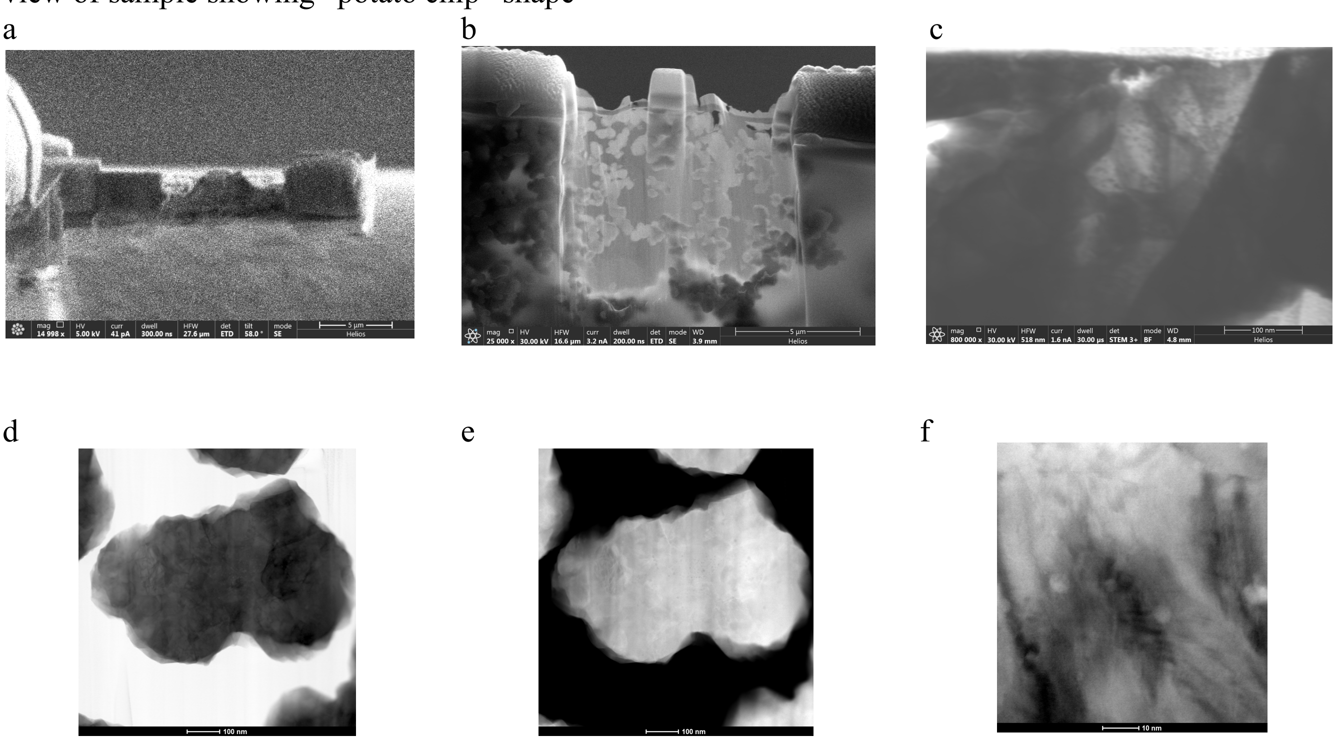

Figure 2. (a) FIB view of final thinned sample using 16kV. (b) SEM view of final thinned sample using 16kV. (c) STEM BF image of thinned area. (d) BF TEM image of thinned particle. (e) HAADF TEM image of thinned area. (f) BF TEM image of thinned particle.

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