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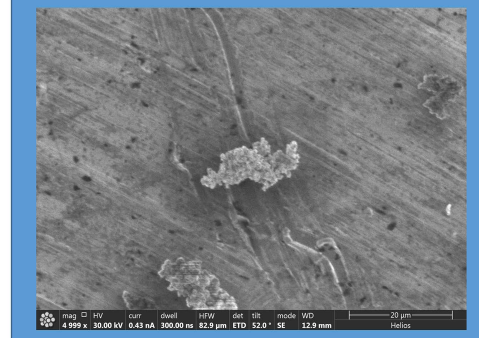
A Method for FIB Liftout of Particles in Epoxy Resin

Warren L. York[1], David Robinson[1] and Joshua D. Sugar[1]
[1]Sandia National Laboratories, Livermore, CA, USA

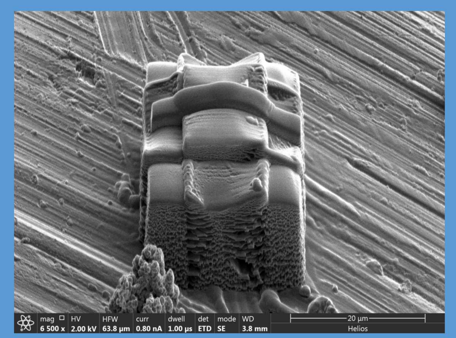
Introduction

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" 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. 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.

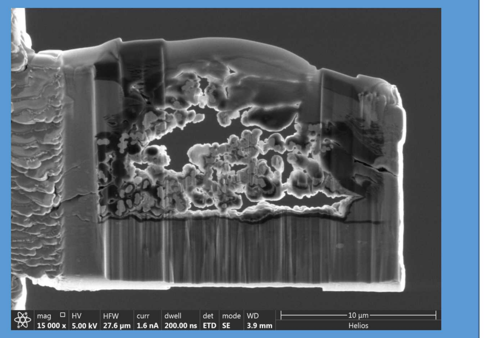
The sample chosen was a Pd powder. The sample required TEM analysis to understand the internal structure of the ~100 nm particles in the powder so a lamella was prepared with FIB/SEM. The first preparation attempt involved encapsulating a small clump of particles with C deposition in the FIB. A lamella was them lifted out from the encapsulated particles and prepared using traditional FIB thinning steps with a 30kV beam and currents from 9.3nA-0.23nA and a final polish step of 5kV and a current of 41pA .



SEM image of the powder before encapsulation in FIB C dep.

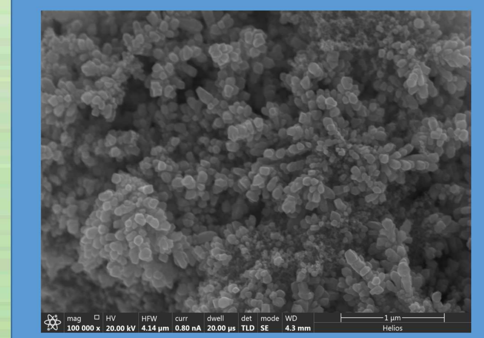


SEM image of the powder after encapsulation in FIB C dep.

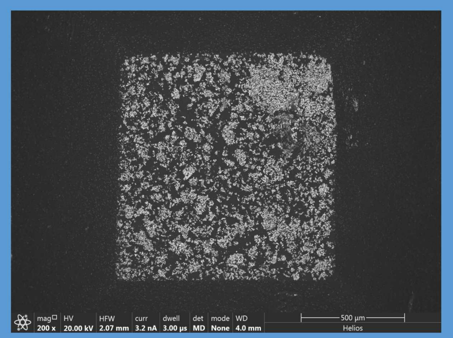


SEM image of the powder after FIB thinning.

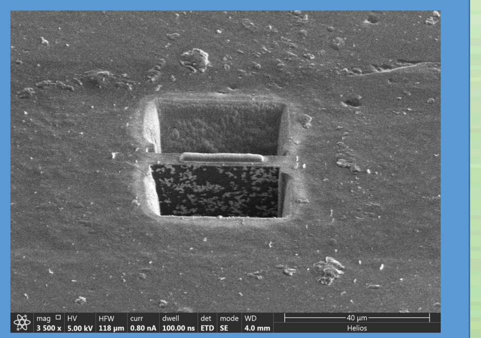
Several voids appeared during the thinning steps in the FIB and the sample soon lost integrity and was unusable for TEM imaging. The next attempt was to use epoxy and vacuum impregnation in order to obtain a starting material with no air pockets that would lead to voids in the sample.



SEM image of the powder before encapsulation.

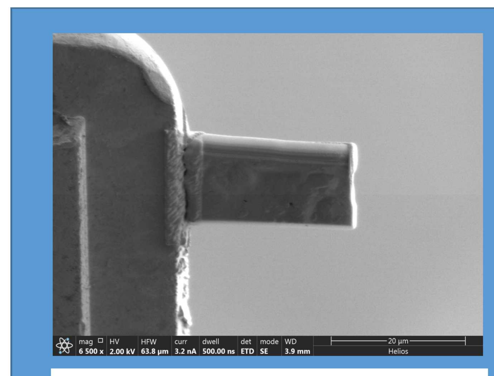


SEM image of a polished cross section of the encapsulated powder showing size and morphology of the near surface particles.

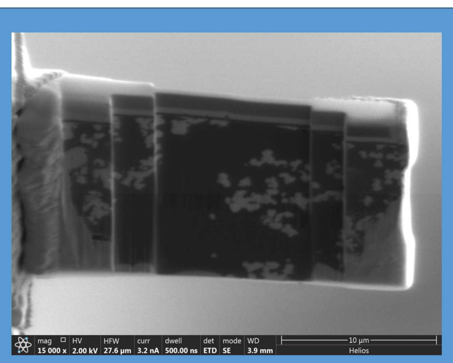


Trenched out areas before liftout of the lamella. Several particles can be seen in the lamella.

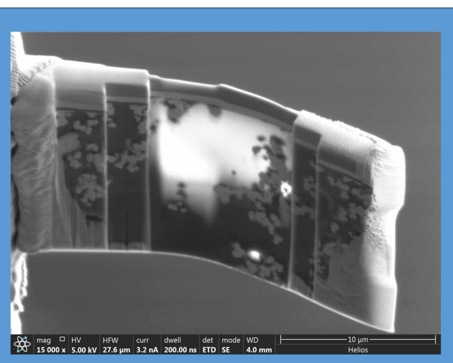
Using our typical and conventional FIB thinning protocol at 30 kV, a sample was thinned down to electron transparency. However, the result was unacceptable because the entire lamella warped and thinned unevenly. This led to unwanted holes in the exact region of interest for TEM imaging, the location of particles.



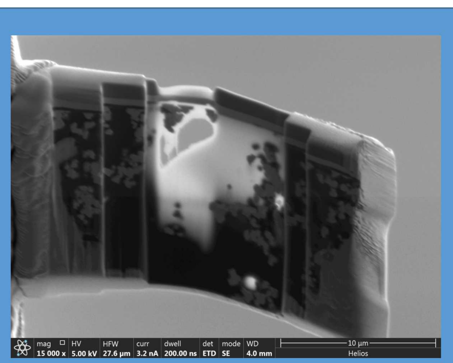
SE image of sample after grid placement.



Sample after the first few thinning steps at 30kV (2.5nA-0.43nA)



Sample after final 30kV thinning step of 0.23nA.



Sample after final 5kV thinning step.

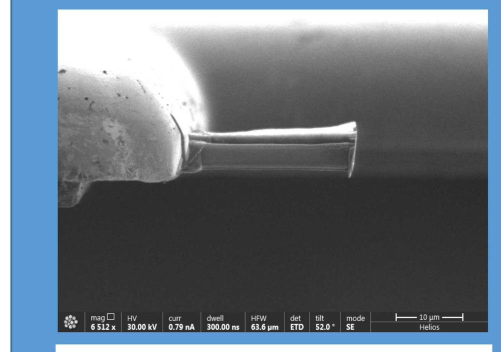
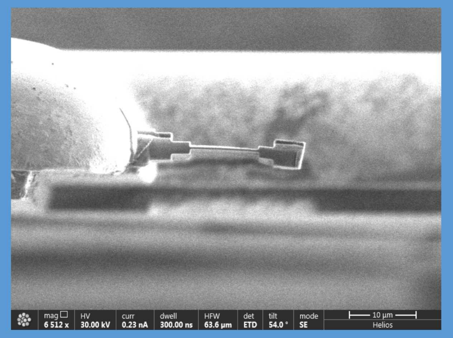
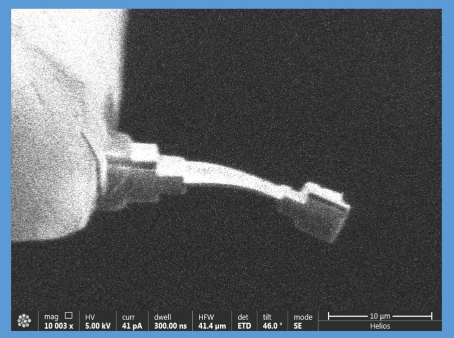


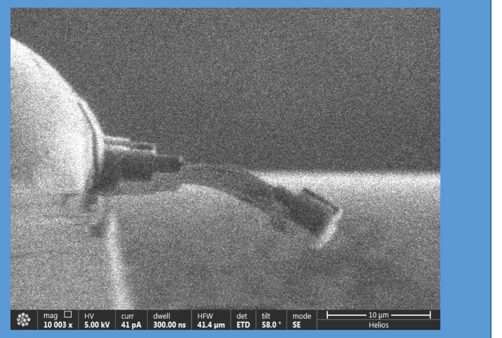
Image of sample after grid placement from FIB view.



Sample after the first few thinning steps at 30kV (2.5nA-0.43nA) from FIB view.



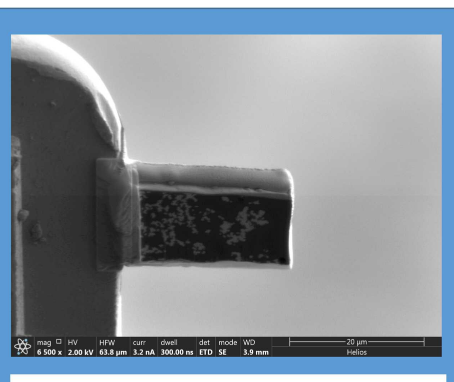
Sample after final 30kV thinning step of 0.23nA from FIB view.



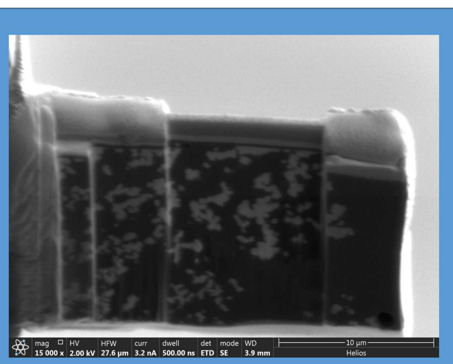
Sample after final 5kV thinning step from FIB view.

Notice the bending "potato chip" effect starts around 0.23nA and gets substantially more exaggerated during the final 5kV polish.

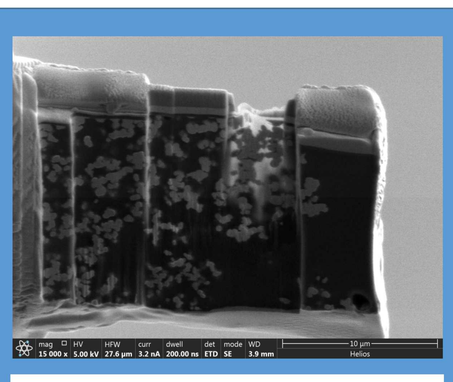
After experimentation, the following modifications were applied to the original procedure to improve the sample quality. 16kV was used for the initial thinning steps, and two smaller final electron transparent windows were used instead of one large window .



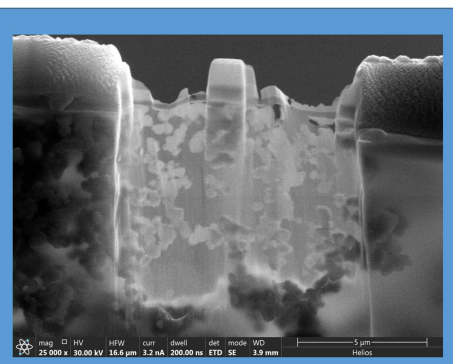
SE image of sample after grid placement.



Sample after first few thinning steps at 16kV (1.3nA-0.15nA)



Sample after final 16V thinning step at 0.15nA.



Sample after final 5kV thinning step.

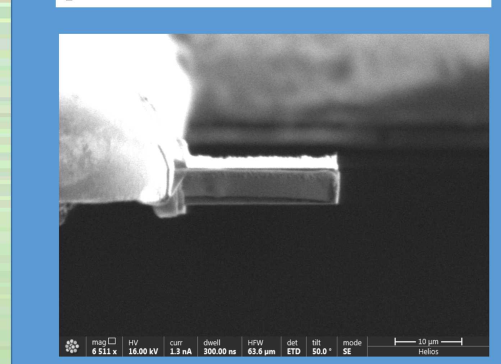
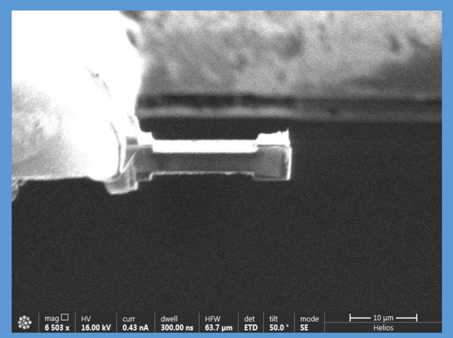
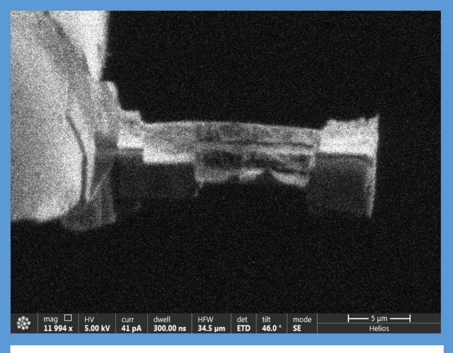


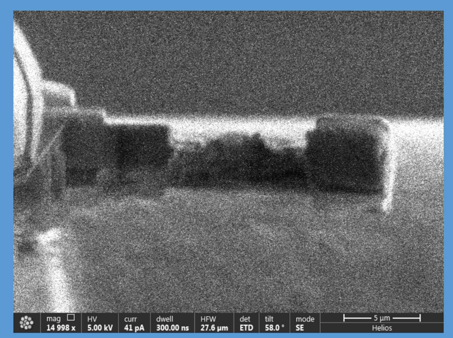
Image of sample after grid placement from FIB view.



Sample after first few thinning steps at 16kV (1.3nA-0.15nA) from FIB view.

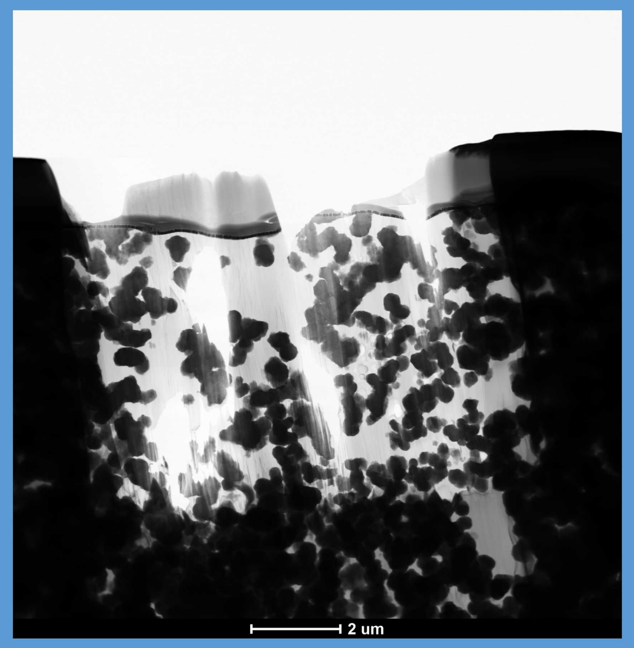


Sample after final 16V thinning step at 0.15nA from FIB view.

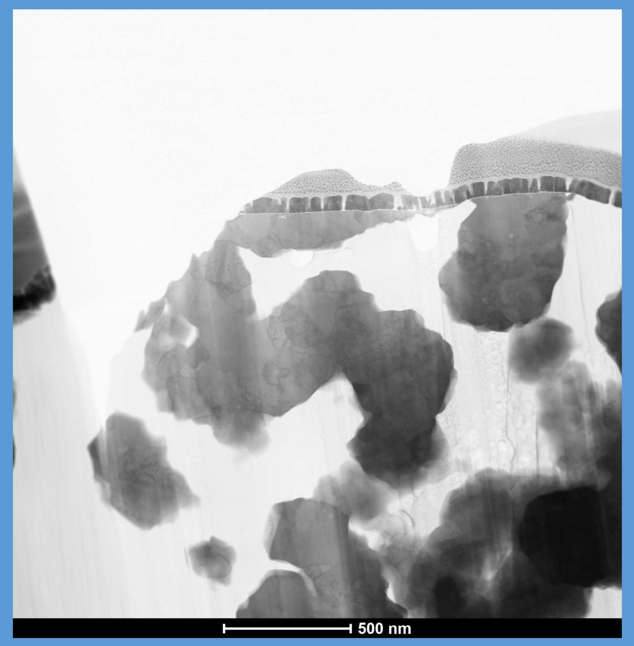


Sample after final 5kV thinning step from FIB view.

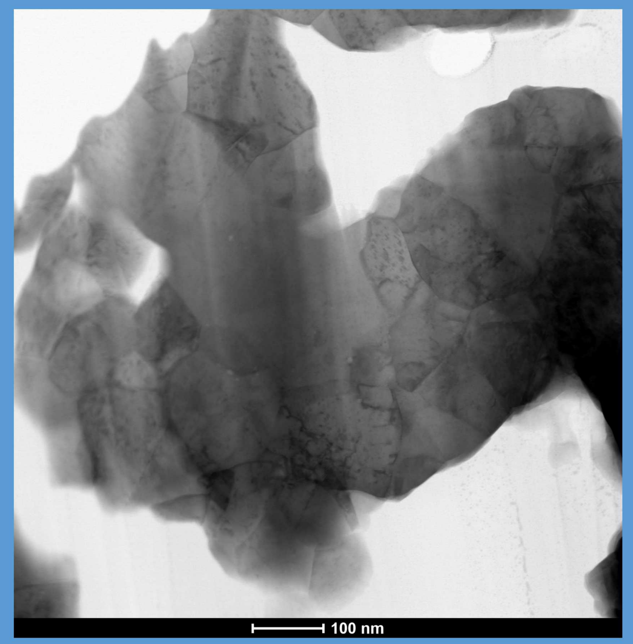
The resulting sample contains several thin particles for TEM investigation. The sample contains only slight warping and the particles are of a uniform thickness which is ideal for TEM imaging. A high-quality result is highly dependent on stopping the thinning process on either side of the individual particles so as to not remove to much material such that low-kV polishing can still occur and remove any high-kV beam damage.



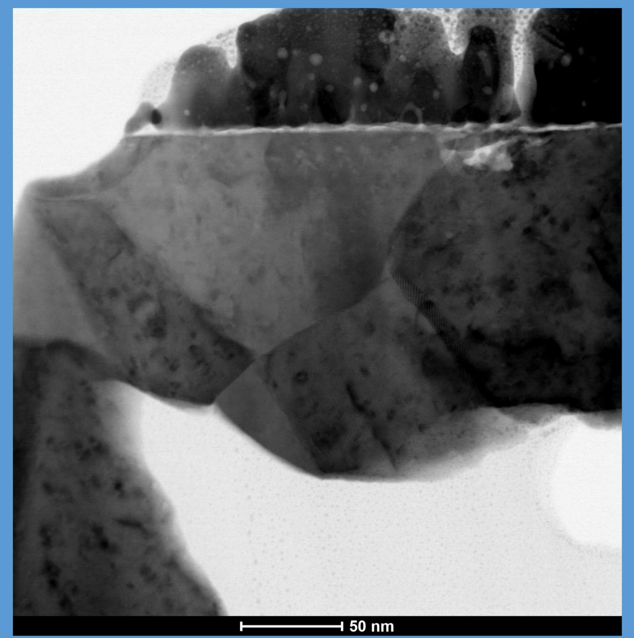
BF STEM image (300 kV) of sample after final polishing step.



BF STEM image (300 kV) of sample after final polishing step.



Higher magnification BF STEM image (300kV) of sample after final polishing step.



Higher magnification BF STEM image (300kV) of sample after final polishing step

A 300 kV STEM image of sample after final polishing step showing a high-quality sample has been prepared. The images reveal a thinned area with uniform thickness and clearly defined grain structure and crystalline defects within each particle.

Conclusion

Adjustments in FIB kV and final thin window size were essential to making successful high-quality samples. For particles that are too small for conventional liftout, these parameters enable successful TEM preparation when an encapsulation medium with different thinning properties is used. This technique provides high-quality TEM results and can be used to study small ~100 nm particles in epoxy. This technique can also be broadly applied to other materials and particle sizes.

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