**A novel double-liftout method for site-specific preparation of specimens for correlating TEM and APT in fine grained materials**

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Atom probe tomography (APT) has been used extensively to study grain boundaries in metal alloys. Initially, all specimens were prepared by electropolishing samples to form the appropriate tip geometry, but with the development of the focused ion beam (FIB) instruments it became possible to make site-specific APT specimens from grain boundaries and other regions of interest. Materials with a grain size in the 5-40 micron range are difficult to prepare using either method, as a large number of specimens will be required to locate a boundary using electropolishing, and the standard lift-out techniques require a straight, several micron long section of grain boundary to lift out to make the specimen.

A technique has been developed to target specific grain boundaries in these materials using a combination FIB and TEM, and the method was validated by targeting a prior austenite grain boundary in a martensitic steel. The technique involves first preparing a TEM sample perpendicular to the grain boundary, then transferring the sample to an electropolished stub compatible with a Fischione on-axis tomography holder. The TEM sample is then rough cut to position the grain boundary near the end of the sample, then annular milled to shape the atom probe specimen. This procedure produces samples with a grain boundary near the tip, on a sample stub suitable for use in an on-axis tomography, and has provided a higher success rate than experienced with traditional techniques for materials with grains in this size range.