

집속 이온빔을 이용한 기계적 합금화 분말 재료의
3D Atom Probe 시편 제조

Preparation of 3D Atom Probe samples from mechanically alloyed
powder materials using Focused Ion Beam

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Three-dimensional atom probe (3D AP) analysis is a unique characterization technique which allows for spatially resolved chemical analyses of materials below nanometer scale. By applying a positive high-voltage to a tip-shaped sample, which has a radius of curvature smaller than 100 nm, surface atoms are field-evaporated and accelerated toward a position sensitive detector. A three-dimensional elemental map can be reconstructed from the impact positions of the atoms on the detector, using a simple point projection algorithm. The 3D AP used in this study has a resolution limit of 0.2 nm in depth and 0.5 nm in lateral direction.

Owing to this extremely high resolution, 3D AP has also become of great interest for the investigation of mechanically alloyed powder materials. However, a huge limiting factor of such analyses has been the difficult sample preparation. As extremely fine tips need to be prepared, 3D AP has been mostly applied to bulk materials, which can be cut into blanks and easily sharpened by standard electro-polishing methods.

In recent years, some researchers have explored the possibility of applying the Focused Ion Beam (FIB) technique to the preparation of 3D AP samples. In this work, a dual beam FIB was successfully applied to the preparation of 3D AP sample samples from nanocrystalline Fe-Cu and amorphous Ti-Cu-Ni-Sn powders. Applying the "lift-out" technique, known from the preparation of TEM samples, it was possible to prepare tips appropriate for 3D AP analyses. Lamellar enrichments could be resolved for Fe₉₅Cu₅ samples mechanically alloyed for 2h, while a nearly homogeneous solution of Cu in Fe was found after 20 h of mechanical alloying. A completely amorphous structure without any chemical inhomogeneity was detected for a Ti₅₀Cu₂₅Ni₂₀Sn₅ sample mechanically alloyed for 20 h.