Cryogenic large volume 3D and TEM sample preparation with multiple ion species plasma FIB

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Direct quantitative investigation of the inner morphology and structure of soft materials is of critical importance to provide profound insights for properties evaluation. The scanning electron microscope (SEM) and focused ion beam (FIB), combined known as FIB-SEM or DualBeam, are conventionally recognized as a highly effective method to acquire 3D volume information. FIB-SEM together with integrated serial sectioning software has made the automated 3D data acquisition and analysis possible. However, slicing using Ga ion beam at room temperature has been found inducing severe damage to the beam sensitive soft materials, resulting in significant deterioration of the 3D data quality. Cutting edge cryogenic FIB-SEM technique provide a fully automated workflow which allows large volume, damage-free ion beam slicing and high spatial resolution SEM acquisition during serial sectioning under cryogenic conditions. With automated 3D reconstruction of the micrograph stacks, we can subsequently recover the comprehensive volume information of such beam sensitive materials. In addition, cryogenic multiple ion source PFIB has been confirmed to be capable of fabricating high quality large area TEM lamellae without damaging the beam sensitive bulk samples. Coupled with cryogenic in-situ nanomanipulator, the TEM samples can be easily lifted out under cryogenic conditions and subsequently transferred to TEM for further investigation.

In this paper we present a series of large volume 3D imaging results and TEM sample preparation examples of extremely beam sensitive soft samples. The samples were processed on Thermo Scientific Helios 5 Hydra Plasma FIB platform with multiple ion species (Xenon, Argon, Oxygen and Nitrogen) combined with integrated state-of-the-art rotatable cryo stage. The slicing and imaging acquisition was achieved using the latest generation Automated Slice and View software and the subsequent data processing was conducted using Avizo 3D analysis and visualization software. The unique technical experiment set up and comprehensive application experience will be discussed in this presentation. In addition, inert gas transfer workflow of the beam sensitive materials from the plasma FIB to TEM will be discussed.

Keywords:

Cryogenic, beam sensitive, soft materials