Application of Cryo-FIB/SEM Technology in Materials Research

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One long-standing challenge for electron microscopists is specimen preparation for microstructural investigation of environment-sensitive materials (e.g., oxidation and hydration/dehydration in air) and examination of wet specimens’ in-situ. Cryogenic electron microscopy (Cryo-EM) has been considered as a solution to overcome the issue of microstructure damage due to high energy electron beam irradiation. The application of cryo-FIB/SEM techniques in materials and life sciences research has thus gained much momentum over the past decade. However, little documentation exists regarding artifact induced during sample preparation at cryo-temperature. This study focuses on preservation of the true sample microstructure by use of cryogenic sample preparation and cryo-FIB milling.

Cryogenic sample preparation is crucial in preserving the microstructures of sensitive or biological samples. The common problem of ice forming on sample surface during cryogenic sample transfer can be prevented by careful operation, such as temperature control and freeze etching time. Sample preparation for cryo-FIB/SEM was performed using Leica Microsystem’s EM VCM, EM VCT 500 and EM ACE 600. SEM observation and focused ion milling were carried out in Tescan’s FIB/SEM (GAIA). The details of cryo-FIB/SEM sample preparation will be described.

Results from cryo-SEM investigation of the morphology and internal structures of the samples will be presented. Materials degradation due to ion beam irradiation at cryogenic temperature will also be discussed.