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A preparation method of TEM specimens using combined techniques of Focused Iron Beam milling and conventional Ar-milling

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Focused Ion Beam (FIB) milling has recently evolved into one of the strongest means of preparing TEM specimens. The technique offers new capabilities beyond those exhibited by conventional Ar ion milling, especially including: site-specificity at the sub-micrometer scale and homogeneous thinning of the samples having multi-layers of different phases or two-phase interface. However, several difficulties still remain for applications of High Resolution TEM (HRTEM) and Electron Energy Loss Spectroscopy (EELS): (1) surface contamination and damages by the Ga beam; (2) sample thinning to less than 100 nm-thickness. In order to overcome them, we herein report an application of combined techniques of FIB milling and conventional Ar-milling for the preparation of TEM specimens from high pressure samples. The specimen examined in this study is one of molten iron coexisting with ferropericlase, synthesized from laser heated diamond anvil cell experiments. The recovered sample was mechanically polished and milled to leave a foil of about 1 μm thickness through the region of interest, using a FIB milling system equipped a micromanipulator. The removal of the foil from the host sample and gluing it onto the Cu grid are performed in situ at vacuum with a Tungsten needle driven by the micromanipulator. After the FIB milling, a conventional Ar-milling was used at 3 kV to reduce the thickness to 40 nm for EELS qualitative analysis. Transmission electron microscopy of the foil was

performed with Philips CM20 FEG operating at 200 kV. The relative thickness of the foil was estimated from the ratio of zero-loss and low-loss intensities in EELS spectra acquired with Gatan 666 PEELS, assuming that the inelastic mean free path (λ) in iron is 115 nm. The foil prepared by the FIB technique was transparent to the electron beam. The thickness (t) was fairly uniform over the whole area containing metallic iron and ferropericlase. However, the thickness of about 130 nm (t/λ = 1.13) is too thick for EELS. In the process of Ar re-thinning, the thickness of the edge decreased to 40 nm (t/λ = 0.35), which is thin enough to get a good signal-noise-ratio in the core-loss region of $O-K$ edge and *Fe-L* edge spectra. The combination of focused ion beam milling with conventional Ar-milling is important for HRTEM applications and EELS qualitative analysis of oxygen in molten iron, for which very thin and clean TEM foils are needed.