
Introduction: Information on crystal chemistry and structure is fundamental to understanding the chemical and physical processes that occurred during the formation of the solar system. The combination of atomic-resolution imaging and compositional analysis make the transmission electron microscope (TEM) a powerful tool for gaining insight into such processes and addressing a wide range of geochemical, geophysical, and cosmochemical problems.

A sample must be transparent to electrons for TEM analysis, and such transparency is generally achieved by reducing the thickness of the specimen to \( \leq 100 \) \( \text{nm} \) in the direction of the electron beam. Here we show that focused-ion beam/scanning electron microscopy (FIB-SEM), combined with an in situ lift-out tool, offers unprecedented control over the site-specific creation and extraction of electron-transparent sections for TEM analysis. We demonstrate in a companion abstract (see Zega et al., 2006, this meeting) that such sections can be analyzed using multiple techniques, enabling several types of information to be obtained from the same region of interest (ROI).

Methods: Electron-transparent sections were created using an FEI Nova 600 FIB-SEM equipped with an Ascend Extreme Access lift-out tool. We analyzed the sections using a 200 keV JEOL 2200FS TEM equipped with an energy-dispersive spectrometer (EDS), in-column energy filter, and bright- and dark-field detectors.

Description of the technique: A 1-\( \mu \text{m} \) thick layer of Pt was deposited over an ROI in the matrix of the Murray CM chondrite to protect the material beneath from Ga\(^+\)-ion implantation during the milling process. The ion beam was then used to remove material above and below the Pt (Fig. 1a), creating a slice measuring 1.5-\( \mu \text{m} \) thick that is sufficiently robust for in situ extraction.

A microtweezer is used to extract the section and is fabricated from an end effector, EE, (Ascend Instruments, LLC) using the ion beam. The EE is composed of either Cu or Mo and consists of a base measuring 3-\( \text{mm} \) across to which an arrowhead like tip is attached by a hinge. The microtweezer is created out of the tip of the EE and consists of a 3-\( \mu \text{m} \) diameter hole centered between two prongs that are separated by a 1-\( \mu \text{m} \) slot (Fig. 1b).

Extraction is a multi-step process that involves capturing the section, detaching it from the substrate, and lifting it out of the hole in which it sits. The microtweezer is positioned above the section and rotated so that the plane of the slot between the prongs is parallel to the top and bottom surfaces of the section. It is then lowered so that the prongs spread around the section (Fig. 1b), and, once secure, the ion beam is used to mill away material from the base of the section, thus freeing it from the substrate. The EE is translated upward to lift the section out of the hole (Fig. 1c).

The as-lifted out sample measures 1.5-\( \mu \text{m} \) thick and must be thinned to \( \leq 100 \) \( \text{nm} \) to be transparent to electrons. To perform in situ thinning, the section is rotated into edge-on orientation with the Pt facing the ion beam to minimize implantation of Ga\(^+\) ions during milling. The ion beam is used to remove rectangular areas of material from above and below the Pt on the part of the section that is suspended beyond the prongs of the microtweezer. With careful monitoring of the sputtering process, thicknesses of \( \leq 100 \) \( \text{nm} \) can routinely be achieved.

Results: The layer of Pt that was deposited onto the matrix prior to milling appears as a bright band at the top of the section in the high-angle annular-dark-field (HAADF) image (Fig. 2a). The prong of the microtweezer appears as a dark region extending diagonally from the top-left corner of the image (Fig. 2a, white arrowhead), occluding the 1.5-\( \mu \text{m} \) thick region. A knife edge occurs where the 1.5-\( \mu \text{m} \) thick region meets the 100-nm thick electron transparent area and extends from the base to the top of the section at a 70\(^\circ\) angle (Fig. 2a, white arrow).

The thickness of the as-lifted out region causes it to be opaque to electrons and produces relatively uniform intensity. In comparison, the region thinned in situ is transparent to electrons and reveals areas with bright and dark contrast, which is indicative of material with high and low atomic number, respectively. Bright-field imaging at the bottom part of the section reveals a euohedral grain measuring 280-nm wide (Fig. 2b). EDS analysis shows that the grain is composed of Fe, Ni, and S. Measurements on its high-resolution TEM (HRTEM) image and selected-area electron-diffraction (SAED) pattern (Fig. 2c) indicate 0.36-, 0.58-, and 0.51-nm d-spacings. The compositional and crystallographic data are consistent with pentlandite \([\text{Fe},\text{Ni}]_9\text{S}_8\) as the identity of this grain.

Discussion: Ion milling has been the only site-specific method of creating large, electron-transparent regions in samples (reviewed by [1]). However, the necessity of supporting a region of interest with another material (typically a 3.0-\( \text{mm} \) O.D./variable I.D. Cu washer) and the large beam size of the ion mill (the Ar\(^+\)-ion beam can reach 4- to 5-
mm in diameter under typical operating conditions) make difficult the preparation of micrometer and nanoscale features of interest. The FIB overcame the site-specific limitation of ion milling [2-7], but the sections lifted out ex situ were supported on amorphous films (typically C or SiO). Supporting the section by such films precludes compositional analysis of elements in common with them and adds to the total thickness of material through which the electron beam must propagate once inside the TEM, which can lead to blurring of image features.

The in situ lift-out technique that we describe overcomes the problems of the ex situ lift-out. The section is well supported by the microtweezer with the region of interest cantilevered beyond the prongs in free space. Once inside the TEM, the electron beam can transmit solely through the ROI thus eliminating problems with compositional analysis and potential blurring of image features. The section from Murray shows that the spatial relationships of matrix minerals are maintained (Fig. 2a,b) and further demonstrates that broad, electron-transparent areas can be created from heterogeneous and friable materials. Although the ion beam can create an amorphous layer on the top and bottom surfaces of the section [2-7], milling at low currents ($\leq 0.5$ nA), especially during the final polishing cycles of the in situ thinning process, creates areas suitable for atomic-scale investigation as evidenced by the HRTEM image (Fig. 2c).

**Conclusion:** The FIB-SEM combined with an in situ lift-out tool provides unprecedented control over site specificity and enables the creation of uniformly thin electron-transparent sections for TEM analysis. This new capability will be important for studying a wide range of earth and planetary materials.

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**Figure 1** Secondary-electron images of a section from the matrix of the Murray CM chondrite. (a) The 1.5-µm thick section after coarse cutting (black arrowhead points to the protective layer of Pt). (b) Capture of the section using the microtweezer. (c) Extraction of the section from the substrate. Scale bars equal 5 µm.

**Figure 2** TEM images of the matrix section. (a) HAADF image showing a prong of the microtweezer (white arrowhead), and the knife edge (white arrow) separating the 1.5-µm thick region (left of arrow) from the 100-nm thick area (right of arrow). (b) Bright-field image of the area delineated by the white box in (a). (c) HRTEM image of the area delineated by the black box in (b). SAED pattern inset.