Focused Ion Beam (FIB) and Microtome Combined Ultrathin Slice Preparation for Transmission Electron Microscopy (TEM) Observation

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Introduction: With the advances of microbeam analytical techniques, integrated analyses have been performed on a single micron-sized sample to understand its formation and evolution history. Especially, crystallographic information combined with morphological, chemical, isotopic measurements are crucially beneficial to constrain the geological or cosmochemical processes of the sample. Therefore, it is important to develop technology to prepare the transmission electron microscopy (TEM) slices (≤100 nm) from a specific micron-sized grain on a polished section or deposited on surface of a sample mount. Focused ion beam (FIB) is good at precisely lifting out. Microtome is applicable to prepare several ultrathin slices. Here, we combide the FIB and microtome techniques. Take an identified micron-sized presolar graphite for example, it is surrounded by amorphous carbonaceous residue with solar isotopes [1].

Samples: The carbon-rich residue of Qingzhen (EH3) meteorite has been separated by acid etching [2], which was dispersed on a high pure gold foil mount. Presolar graphite grains were identified based on anomalous C-isotopic compositions by NanoSIMS measurements [1]. The spherical presolar graphite selected has cauliflower-onion morphology, glassy-type with D/G = 1.13 in Raman spectra, and relatively high in ^{12}C ($^{12}C/^{13}C = 99 \pm 2$) and ^{29}Si ($\delta^{29}Si/^{30}Si = 172 \pm 36‰$).

Experiments and Results: The TEM slices preparation process included two main steps. (1) Sample transfer with FIB. A carbon fiber was picked up and shaped via FIB, and an epoxy resin mount was trimmed with a blackface and Au-coated. The selected graphite spherule was attached to the top of the carbon fiber and moved to the top of the resin base together. The carbon fiber was cut free finally. (2) Sections slicing with the microtome. The sample was embedded in the resin drop, with the aid of a micromanipulator under an optical microscope. After curing, the sample was sliced with the microtome, and ultrathin sections were located on the holey-C-coated Cu TEM grids for TEM observations.

Under TEM, the graphite slice was found on one end of the carbon fiber, divided by the deposited Pt. The spherule has poor crystallization with turbostratic layering of contorted lattice planes and short-range layer continuity, which is consistent with its Raman signature. But no other submicron-sized inclusions were seen with diffraction contrast imaging and EDS measurements.

Advantages: First, several ultrathin sections can be obtained from a micron-sized grain. Second, reducing the risk of grain loss caused by static charge with the help of FIB. Third, carbon fiber served as a fiducial marker would be benefit for grain relocating.

Implications: Such FIB-microtome technique can be used to prepare TEM sections of micron-sized grains. Furthermore, the samples returned by future mission, either Lunar or small bodies, contain abundant micron-sized [3]. This technique can be a critical experimental method for theses retuned samples. In addition, it is also can be applied to tiny high pressure mineral, porous specimens, and other rare and unique micron-sized grains of terrestrial rocks and meteorites.

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