Focused ion beam milling: A method of site-specific sample extraction for microanalysis of Earth and planetary materials

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Abstract

Argon ion milling is the conventional means by which mineral sections are thinned to electron transparency for transmission electron microscope (TEM) analysis, but this technique exhibits significant shortcomings. In particular, selective thinning and imaging of submicrometer inclusions during sample milling are highly problematic. We have achieved successful results using the focused ion beam (FIB) lift-out technique, which utilizes a 30 kV Ga+ ion beam to extract electron transparent specimens with nanometer scale precision. Using this procedure, we have prepared a number of Earth materials representing a range of structures and compositions for TEM analysis. We believe that FIB milling will create major new opportunities in the field of Earth and planetary materials microanalysis, particularly with respect to ultraprecious mineral and rock samples.

INTRODUCTION

The introduction of ion milling in the 1950s for the preparation of mineral specimens for use with the TEM revolutionized the study of earth materials (Castaing and Labourie 1953; Paulus and Reverchon 1961; Barber 1970). Previous approaches to TEM examination of metals and biological tissues yielded unsatisfactory results when applied to the brittle oxides that comprise the bulk of the Earth and many meteorites. However, a new frontier opened in the microstructural analysis of Earth materials once it was shown that most minerals could be thinned to electron transparency with acceptably low levels of radiation damage by ion bombardment at ~6 keV. In conjunction with complementary techniques (such as dimpling and tripod polishing), Ar ion milling has evolved into the standard means of preparing TEM samples from minerals (Barber 1999).

Recently, Giannuzzi et al. (1999, and refs. therein) have shown that FIB milling may be used to prepare TEM specimens for metals, ceramics, composites, semiconductors, and biological materials. Here, we have extended the viability of this technique to a variety of mineral samples. FIB milling offers a host of capabilities beyond those exhibited by traditional Ar ion milling, including: (1) sample extraction from extremely small volumes of unpolished material; (2) site specificity at the submicrometer scale; (3) sample imaging by either secondary ions or electrons during the milling procedure; and (4) rapid processing of superhard materials. Our initial results indicate that FIB milling produces excellent TEM sections for a range of mineral structures and compositions, even friable clays.

Therefore, FIB sample preparation heralds a dramatic advance in the microstructural analysis of mineral surfaces and particularly of ultraprecious materials, such as extraterrestrial specimens returned to Earth and historically significant gemstones.

ALTERNATIVES TO ION THINNING

Prior to the introduction of Ar ion milling, the major methods for preparing samples included electropolishing, ultramicrotomy, and crushed grain suspension. The last of these is the simplest and remains appropriate for certain applications. A rock or mineral sample is ground in an agate mortar in an ethanol solution, and a drop of the suspension then is deposited on a holey carbon film. Upon evaporation of the ethanol, the grid is ready for TEM inspection. The drawbacks to the technique are threefold: (1) all textural information is lost when the sample is crushed; (2) the typically small amount of thin edge limits the applicability of high-resolution TEM; and (3) some materials are less susceptible to cleavage than others. Diamond, for example, will grind an expensive corundum mortar rather than vice versa.

Ultramicrotomy involves the impregnation of a sample in a wax or resin block, which then is shaved into thin slices by a diamond knife. Although this technique is standard for biological TEM, it remains a specialized approach in the mineral sciences, suitable for soft clays, cosmic dusts, and bacterial-mineral interfaces (Eberhart and Triki 1972; Noguchi, 1998; Barker and Banfield, 1998; Paquette et al. 1999). The brittle quality of most ceramics leads to a “chattering” of the diamond knife as it sweeps across the specimen, which responds by fragmenting into a series of laminar sections. When the styles and concentrations of structural defects are an integral part of a...