FOCUSED ION BEAM TECHNOLOGY: MORE THAN A SHARP KNIFE

I confess that high-tech laboratory methods have long been a fascination of mine. I am always eager to see what the newest technology has to offer for recovering even more information out of one's samples; an obvious trend over recent decades has been the characterization of geomaterials at ever smaller scales. Selecting from the spectrum of available analytical tools, it is now possible to get detailed information about the physical and structural properties or the chemical and isotopic compositions of a sample at the submicron scale. Some methods are even able to provide detailed information down to the atomic scale, which is truly amazing.

Focused ion beam (FIB) technology has been available for investigating geomaterials for the better part of a decade. Most people familiar with this method know it as a means of milling precisely positioned sample foils: basically it is a very sharp knife used to prepare samples for the transmission electron microscope (TEM). After a location of interest on the surface of a polished sample has been selected—for example, a grain boundary that one wants to investigate—a FIB instrument can produce a thin foil penetrating down into the top tens of microns of the sample and with a positioning accuracy on the order of tens of nanometers. The sample foil, as little as 50 nm thick, is milled out of the sample using a very finely focused gallium ion beam (Fig. 1). FIB sample preparation has superseded older approaches relying on, for example, clouds of argon ions for eroding holes through a sample and thereby producing a bevelled edge at a random location.

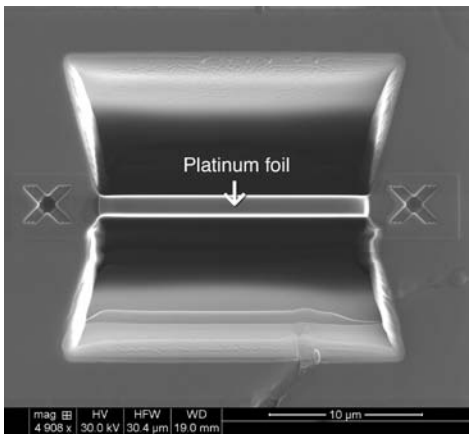


Figure 1 Image of an ~800 nm thick foil milled from a sample. The foil includes a thin layer of platinum metal, which was deposited above the film in order to protect the underlying material. Such "single-beam" applications have been available to the mineralogist for nearly a decade.

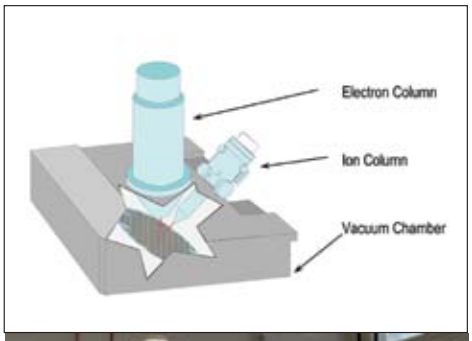


Figure 2 Photo of the Potsdam FEI Quanta dual-beam tool, which in many ways has the appearance of a scanning electron microscope. Above: cartoon showing the general layout of the sample and the two probe beams. Courtesy of FEI company

A few weeks ago I took a walk across the hallway here in Potsdam to visit the dual-beam FIB laboratory, which features a FEI Quanta tool (Fig. 2). I had a very interesting chat with my colleague Luiz Morales, who runs the facility, and I learned much about recent advances in FIB technology. In contrast to previous-generation FIB instruments, "dual-beam technology" combines a finely focused ion beam (primarily used for eroding material from the sample surface) and an electron beam (used for extremely fine-scale imaging of the sample). Simply said, this configuration represents nothing less than the integration of FIB technology with a high-resolution scanning electron microscope (SEM). This tool offers not only the capability of sample milling at the nanometer scale but also the ability to characterize major element chemistry, crystal orientation, and the texture of a material as the foil is being prepared.

Here I would like to highlight two projects that particularly impressed me about this technology. The first was a study of the 3-D structure of a quartz–quartz grain boundary from Kruhl et al. (submitted paper). After initially milling a 10 µm wide, cube-like structure into the sample surface (Fig. 3A), an image of the 3-D geometry of the grain boundary it contained was built up by alternating between sample imaging using secondary electrons (SEM mode) and eroding back the surface of the cube in 100 nm steps (FIB mode). By repeating this cycle many times, it was possible to build up a very detailed map of the grain boundary over the entire 10 × 10 × 10 µm volume. The entire procedure took 10 hours, after which the exact structure of the grain boundary between the two quartz crystals could be visualized at ≤100 nm resolution in all directions. Using appropriate software, a detailed model of the grain boundary was generated, which can be visualized in a 3-D virtual space. The most obvious conclusion from this study is that this particular grain boundary was most certainly not a flat plane at the nanometer spatial scale (Fig. 3B).

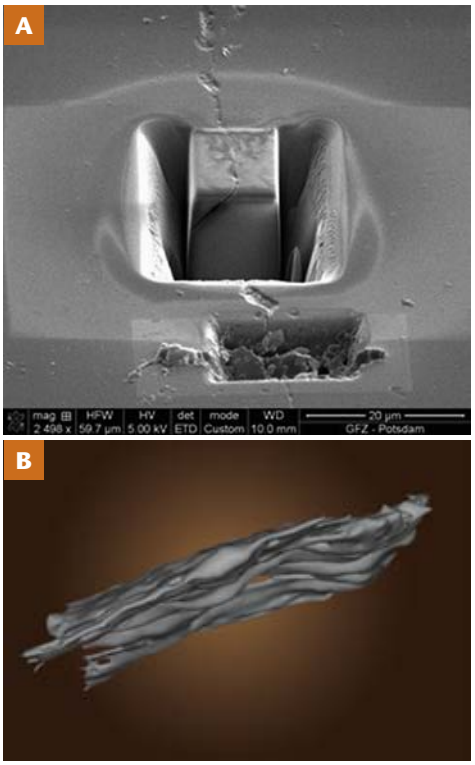


Figure 3 (**A**) 10 µm cube milled into the sample surface as it appeared prior to 3-D imaging. The trace of the grain boundary across the top and front faces of the cube is clearly visible. (**B**) Two-dimensional projection of the 3-D geometry of a grain boundary between two quartz crystals. The smallest visible topographic features are of the order of a few tens of nanometers in size.

Cont'd on page 90

Cont'd from page 89

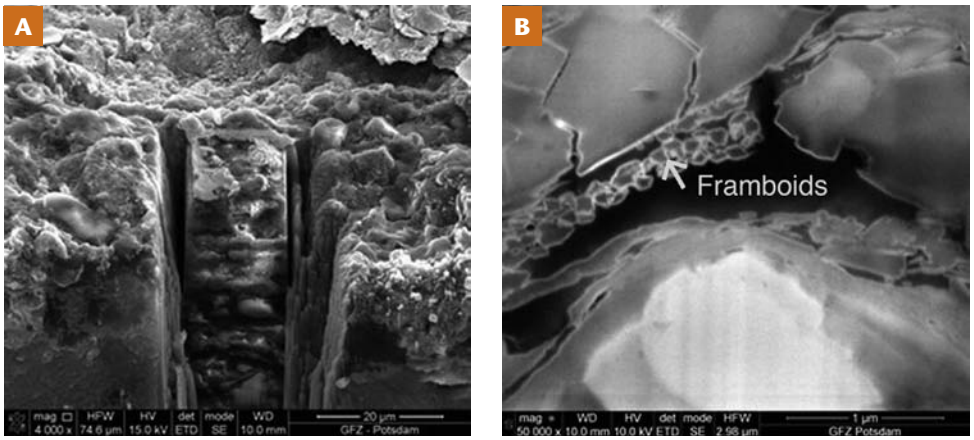


FIGURE 4 (**A**) Secondary electron image of a shale sample with two parallel trenches milled into its surface. The intervening material has already been largely removed by the 3-D imaging process. (**B**) High-magnification image from one of the imaged planes showing 50 nm sized framboidal iron sulfide which has partially infilled a micrometer-sized void in the shale; the major element composition of the framboids was determined by the EDX analyzer.

The second investigation involved the 3-D imaging of microstructures within a sample from the Lower Jurassic Posidonia Shale. Two parallel trenches were milled into the sample surface, followed by the milling of a third, broader trench connecting the two (FIG. 4A). By alternating between milling (FIB) mode and imaging (SEM) mode, both the structure and the mineralogy of this complex material could be described at an impressive spatial resolution. Again using software for manipulating the data set within a 3-D virtual space, one can visualize the complex network of voids and secondary mineralization present within the highly heterogeneous, fine-grained matrix (FIG. 4B). Dual-beam FIB is obviously a very useful tool for a broad range of research themes: rheology, diagenesis, low-temperature mineralization, hydrology, and others.

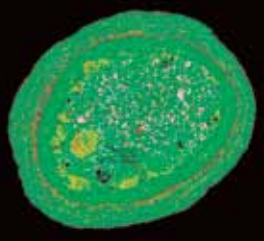
Anyone interested in this technology is encouraged to have a look at some of the films resulting from these studies—I think you will be impressed. Visit Youtube.com and then search for "FIB + Potsdam + Quanta." Focused ion beam technology has clearly moved from being a high-tech knife for cutting thin foils; it has become a powerful research tool in its own right.

Michael Wiedenbeck
(Michael.Wiedenbeck@gfz-potsdam.de)
Potsdam, Germany

REFERENCE

Kruhl J, Wirth R, Morales L (2012) Quartz grain boundaries as fluid pathways in metamorphic rocks. Earth and Planetary Science Letters (submitted)

Mapping the world ... one micron at a time



ultrafast contextural quantitative
SEM-based Petrographic Analyzers

QEMSCAN® image of typical bauxite ore from Asia. Bauxite is the principle commercial source of aluminium. The distinctive sub-rounded particles in the image are known as pisoliths, which form at the Earth's surface as a result of breakdown of a previous rock, typically basalt. No two pisoliths are the same – although they generally have a nucleus and core, which is then surrounded by several concentric layers.

Extraction of the aluminium from the ore involves complex chemical digestion techniques, and the efficiency of this process is significantly reduced if impurities other than aluminium hydroxide (green) are present in the ore, such as quartz (pink), clays (brown) and iron oxides (orange).

QEMSCAN® MLA
www.fei-natural-resources.com

FEI™ Explore. Discover. Resolve.