Low Voltage FESEM of Geological Materials

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Low voltage field emission SEM (*i.e.*, operated at several hundred volts to 5 kV), offering advantages in surface imaging due to reduced beam penetration,[1] was found to be particularly useful in the investigation of uncoated, fine, geological materials down to the nano-scale. Here are four examples to highlight projects being conducted in our FESEM facility.

Kaolinite is one of the most important industrial minerals. Information about its surface properties and cation exchange capacity are important in both ore processing and applications of the clay mineral.[2] Low voltage (LV) secondary electron (SE) imaging (Fig. 1a) reveals detailed (001) surface features of kaolinite, which are not obtainable with high voltage SEM (Fig. 1b) and TEM. Kaolinite is highly sensitive to beam damage and can show obvious degradation after just a few seconds under the TEM beam. However, microtomed TEM samples may be easily examined under LV SEM to show their thickness perpendicular to (001) and cross section features. Such information allows easy access to direct surface area determination of clay minerals.

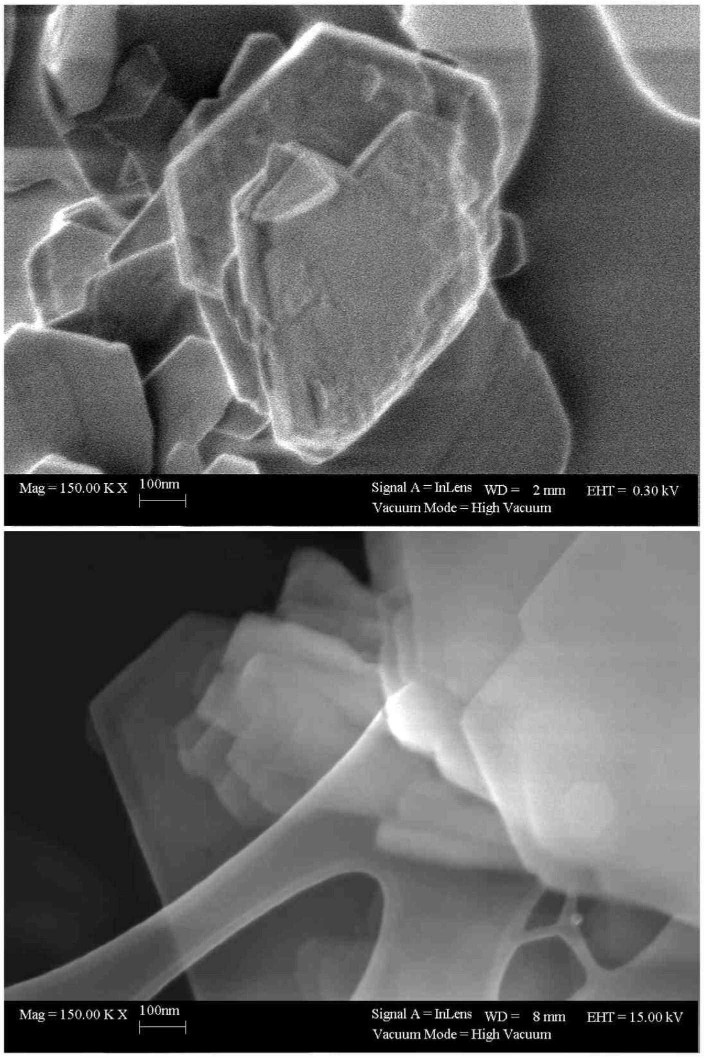

FIG. 1. (a-top) Low voltage SE image (0.3 kV) revealing (001) detailed surface features of kaolinite plates. (b) 15 kV voltage SE image showing 'transparent' kaolinite plates.

Rose quartz occurring commonly in pegmatites has been mined as gem or carving stone since ancient time. Only recently have studies revealed that massive rose quartz owes its color to fibrous nanoinclusions of a borosilicate related to the mineral dumortierite whose pink color is due to Fe-Ti intervalance charge transfer.[3,4] Low voltage SE imaging of extracted and dispersed fibers on holey carbon film shows clear crystal surface features (Fig. 2), which contributes to a complete characterization of the fibers from rose quartz, along with HRTEM and AEM results. The regular surface, as shown in the low voltage SE images, indicates that the irregular or wavy surface layers in HRTEM images are likely caused by the beam damage.

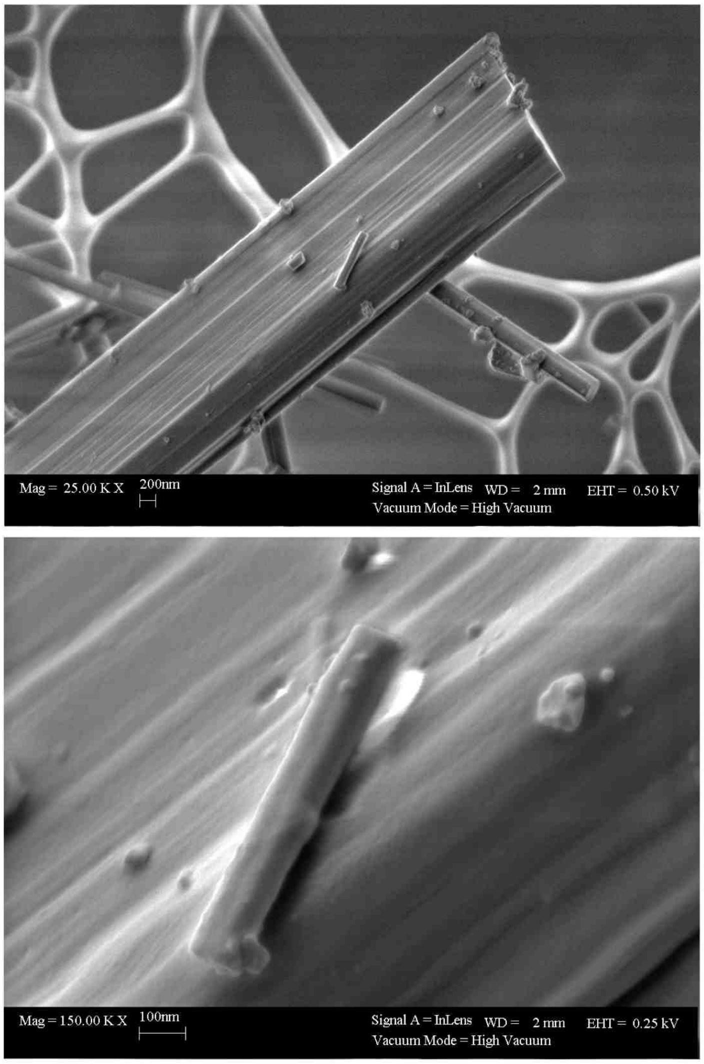

FIG. 2. Low voltage SE images revealing crystal surface features of fibrous inclusions from rose quartz. (a) 0.5 kV accelerating voltage, (b) 0.25 kV accelerating voltage.

Rainbow hematite displays a spectrum of beautiful colors (Fig. 3a). A thin film of an aluminum phosphate was found to coat this hematite. These thin films, with an index of refraction which differs from hematite, are believed to cause the color.[5] Our LV FESEM imaging (Fig. 3b & 3c) shows that the thin film consists of nano-crystals arranged in three directions (120° apart). The rod-shaped crystals have a diameter from 15 to 35 nm. We now need to determine how

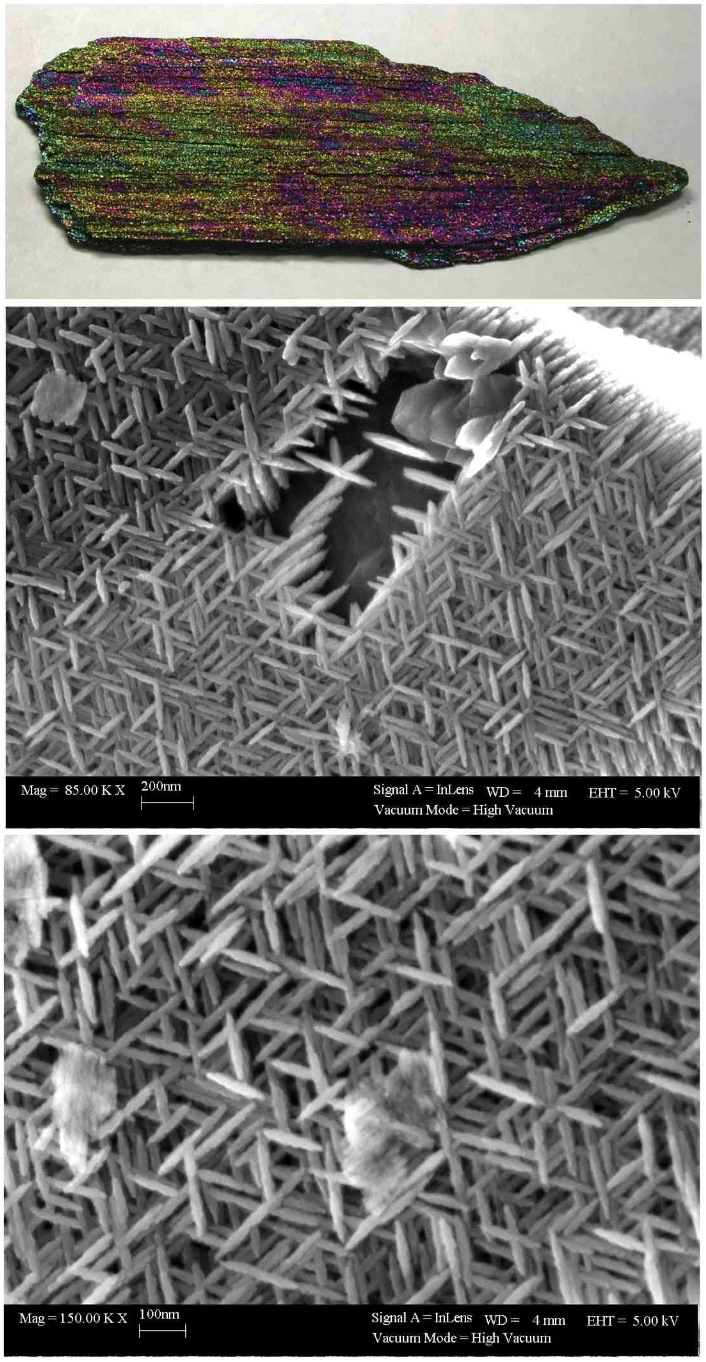

FIG. 3. (a) Rainbow hematite (11.5 cm wide). (b) & (c) Low voltage SE images showing nano-crystals on the surface of rainbow hematite.

these nano-crystals might contribute to the cause of the colors. The LV FESEM contributes an exciting new dimension to this problem that simply could not be observed under conventional SEM.

Non-crystalline opals in hydrothermally altered ignimbrite from Lake Tecopa, California, and an Australian gem quality opal, are being studied with LV FESEM. Although it is well known that opal consists of silica spheres, low voltage SE imaging of the specimens coated with carbon film (<5 nm in thickness) presents more surface information on these spheres. Silica spheres are devoid of surface features when formed in a void where they sit loosely (Fig. 4a). When minerals like feldspar are completely replaced by silica spheres, the spheres are closely-packed (Fig. 4b). Contact features on the close-packed silica spheres (Fig. 4c) indicate that the spheres are weakly cemented. In the gem-quality opal, the spheres are tightly packed and strongly cemented (Fig. 4d) where the fracture surface breaks through the spheres. With the help of LV SEM we are trying to understand how natural opal formed and how it becomes packed. ■

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References

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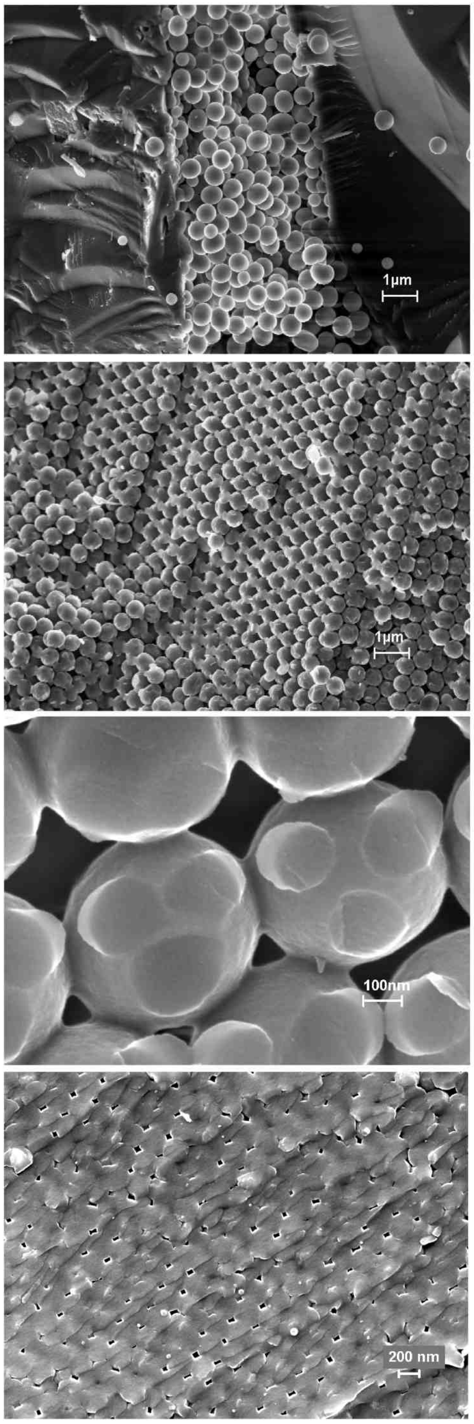

FIG. 4. Low voltage SE images of opal showing (a) silica spheres formed in a void, (b) & (c) closely-packed spheres, and (d) tightly-packed silica spheres of a gem quality opal.