

Shale plan view sample preparation for scanning electron microscopy

Careful mechanical preparation of shale samples for ion milling increases the speed of the milling process and improves the quality of the resulting images.

Materials

- Hot plate
- Crystalbond™ adhesive
- Low-speed saw with diamond wafering blade or diamond wire saw
- Aluminum SEM stub
- Grinder/polisher
- Abrasive disks (600, 800, and 1200 grit)
- Diamond lapping films (3 and 1 μm)
- Heat gun
- Fischione Model 1060 SEM Mill

Cutting the sample

Break the shale sample in to a manageable size, for example $\leq 20 \text{ mm} \times 20 \text{ mm}$ (Figure 1). Note that the SEM Mill accepts a maximum sample size of 20 mm (length) \times 20 mm (width) \times 15 mm (height).

You will be mounting the sample onto an aluminum SEM stub for ion milling. The sample should sit as flat and as parallel to the aluminum SEM stub as possible. If the sample fits this description, you can proceed directly to grinding/polishing. If not, you will have to cut the shale using a saw.

Affix the sample to a low-speed saw with a diamond wafering blade or to a diamond wire saw (Figure 2). Follow the saw manufacturer's lubrication recommendations. Cut the shale sample parallel; the resulting sample should still be of sufficient height to tolerate the removal of enough material to create a polished surface.

Grinding/polishing

You can use any automated grinder/polisher; the grinder/polisher shown in this application note is a parallel polishing fixture. Follow the manufacturer's lubrication recommendations.

Use Crystalbond and a hot plate to mount the sample on the parallel polishing fixture (Figure 3).



Figure 1. Select a shale sample that is approximately 20 mm \times 20 mm.

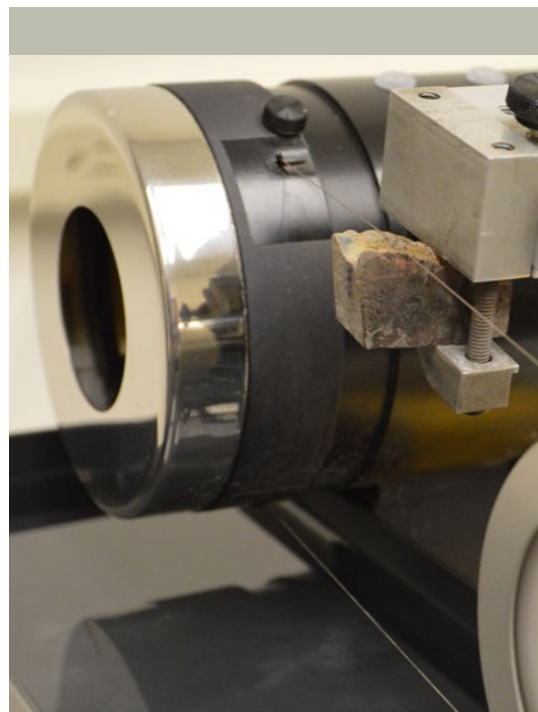


Figure 2. Cut a flat, parallel surface on the shale face in preparation for grinding.

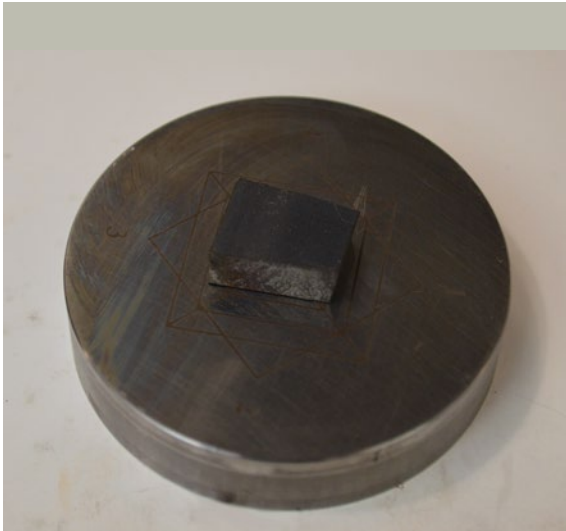


Figure 3. Sample mounted on the parallel polishing fixture.

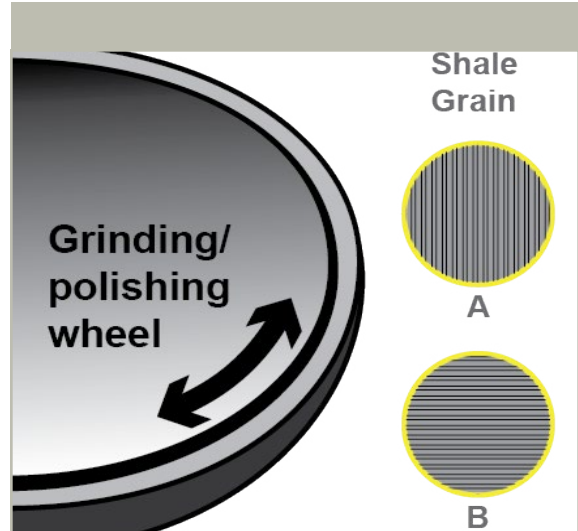


Figure 5. Note the direction of the shale grain. Grind with the grain (A), not against the grain (B).

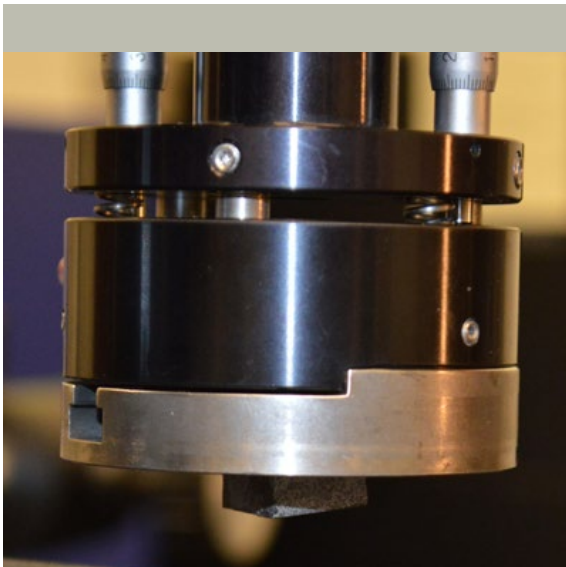


Figure 4. Mount the sample/fixture assembly onto the grinder/polisher arm.

Mount the fixture onto the automatic grinder/polisher (Figure 4). Using a 600 (rough) grit abrasive paper, grind the surface of the shale

until all evidence of initial processing is removed. Continue grinding with increasingly finer grit paper until the surface is smooth, level, and free of scratches or gouges.

While grinding/polishing, note the direction of the shale grain (Figure 5) as it emerges. Because the layer structure of shale fractures easily, grind with the grain. Similarly, apply vertical pressure to the sample/fixture assembly with caution.

Use 3 and 1 μm diamond lapping films for final polishing. Dry the sample with a heat gun.

Ion milling

With the polished side of the sample facing up, use Crystalbond adhesive to mount the shale on an aluminum SEM stub. Insert the SEM stub and sample into the Model 1060 SEM Mill.

Suggested ion milling parameters

Fischione Instruments recommends the following milling parameters: operate both ion sources at 5 kV, 45% focus, and 5° beam angle for one hour with continuous rotation.



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