Looking Inside—Having a Cracking Time

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Introduction

If you wish to look inside an arbitrary piece of material and you own a light microscope (LM) or scanning electron microscope (SEM), there are probably three ways that you may accomplish your aim: (a) embed the material in epoxy, then grind and polish it flat; (b) embed the material and use a microtome to obtain cross sections; or (c) CRACK IT. In this short article, it is the last option that will be discussed. The last method is the simplest and quickest of the textbook sample preparation methods for SEM [1].

Both light and scanning electron microscopes are interesting instruments; they are very clever at picking out faults in our preparation techniques. If, for example, you cut your material with a hope of viewing its internal structure, then the scanning electron microscope is most likely to demonstrate to you the effects of any cutting tool used to peer inside. The instrument will show the effect of cutting with a new scalpel, an old scalpel, or even a pair of scissors. A simple way to visualize the internal structure of a material is to crack it, so that only the forces that made it crack have an effect. The medium that often makes this route possible is liquid nitrogen; this is the stiffening agent that enables us to crack many materials.

Methods

To begin, let us look at the simplest possible cracking method—just break it! I have looked at a wide variety of materials that we have been able to crack, as is, with our bare hands! Polymers, plastics, and medical pills are typical examples of materials that may be cracked without any form of preparative equipment (Figure 1).

If we need to use freezing methods, a small polystyrene cup placed on a non-conducting pad makes a good container for the liquid nitrogen. The operator should wear safety glasses and work from above the container. In all the cases mentioned in this article, the material should remain in the liquid nitrogen until the liquid stops boiling—an indication that it is down to liquid-nitrogen temperature. To lift material out of the container, use tweezers and place the material on a pad of soft tissue to absorb the condensation that will arise.

If your material is a powder in suspension in some medium, consider the simplest possible technique, the paint technique. If your material is in a medium that will dry out, "painting" the suspension onto a piece of aluminum foil could be your answer. Once the medium has solidified on the foil, simply place the foil in liquid nitrogen. The differing rates of contraction between the foil and the medium will cause the medium to crack and break away from the foil. Catch the pieces and you have ready-made cross sections that, with some physical support and possibly sputter coating, may be viewed in the light microscope or the scanning electron microscope (Figures 2 and 3).

The next simplest technique requires the sample material to have some rigidity but be quite thin—less than about 3 mm (1/8 inch) thick. In this case, cut the material down to about

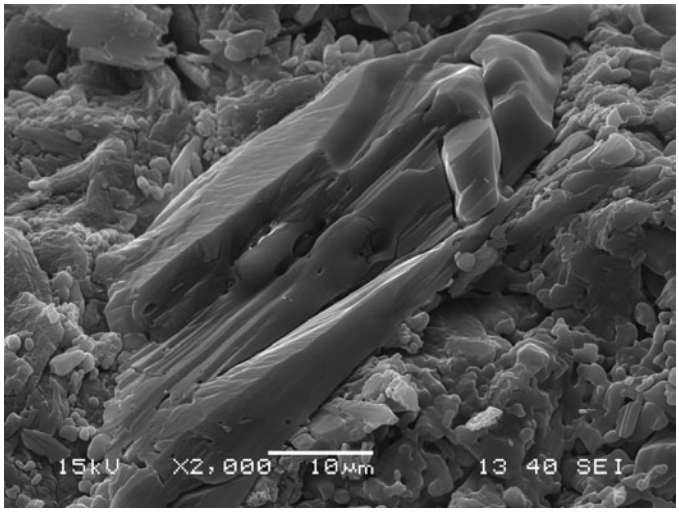

Figure 1: Crystals within a sinus medication pill. A simple fracture made at room temperature of a pill. The fracture was gold coated and imaged in the secondary electron mode at 15 kV.

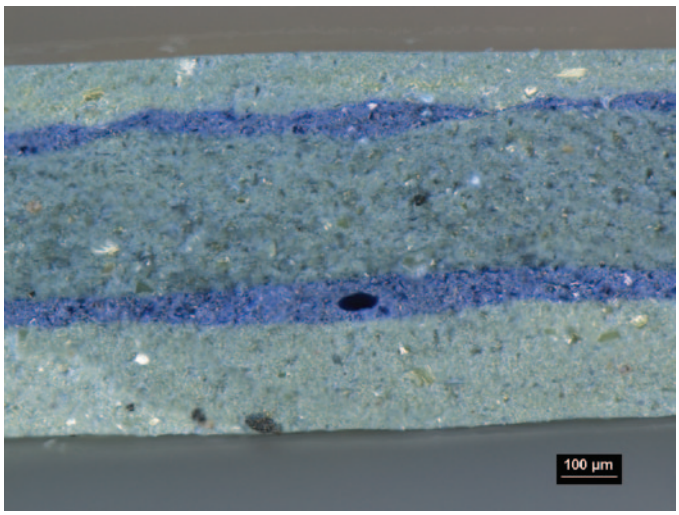

Figure 2: Light microscopy image of a 5-layer paint film.

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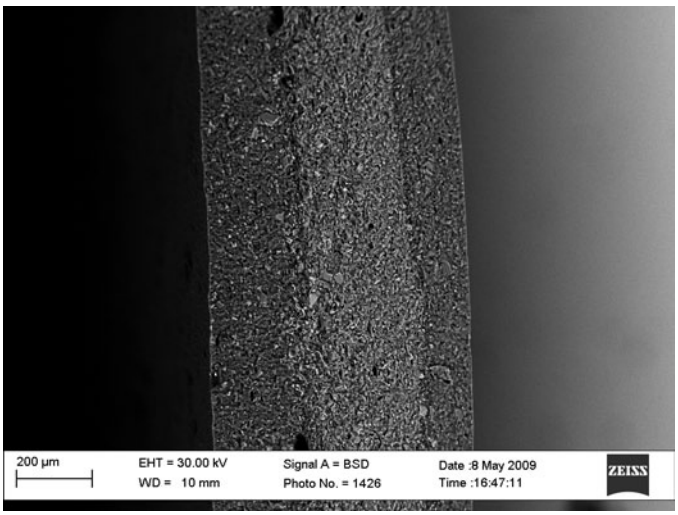

Figure 3: A 30-kV backscattered electron image of the 5-layer paint film shown in Figure 2.

2.5 cm (1 inch) high by 6 mm (1/4 inch) wide and place the material in liquid nitrogen. This time lift the material out of the liquid using a pair of light pliers, and, with a second pair, take hold of the opposite end and flex the material once to crack it. If the material will not break immediately, you need a modification to the technique: "necking." Necking means cutting down the width of the material at its center as shown in Figure 4. Note: even 3 mm of viewable area has been known to keep an operator occupied on a scanning electron microscope all day! Once cracked, you now have two pieces of material to observe by light microscopy or scanning electron microscopy.

Things now become a little more complicated. If you are trying to look at fibers that may not become brittle by freezing, we have to help them crack by offering a brittle support. There are two techniques that may be suitable. In the first method, feed the fiber or stranded material into a drinking straw about 3 mm in diameter and cut off a piece 30 mm to 40 mm long. Block one end of the straw with wax or modeling clay and syringe water into the straw to fill it completely. Block off the second end with wax or clay. Lower the straw into the liquid nitrogen until it is cooled, then remove it with pliers. Use a second pair of pliers, as in the last technique, to crack the straw. Note which are the cracked ends and once the material under investigation is dry, mount the cracked ends for light or scanning electron microscope observation.

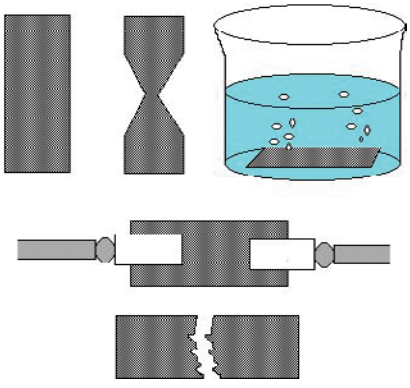

Figure 4: Diagram showing typical shapes of samples and the method of cracking with pliers. Note the example of necking.

Now for the most complex technique I use for cracked material investigations. In this case, the sample is placed between two SEM stubs that have been glued together, face to face, with a weak-bonding adhesive. Drill through the stubs using a 3-mm (1/8-inch) to 6-mm (1/4-inch) drill. Feed your material through the hole and then flood the hole with a water-based carbon suspension, such as Aqua Dag® (Figure 5). When the unit is dry, place it into liquid nitrogen. Lift the unit out of the liquid when it has had time to freeze and set it on a soft surface. (A couple of layers of tissue works well.) Take a single-edged razor blade or scalpel and force it into the joint between the stubs. We need the blade to crack open the unit, not to cut through, so take care! Once cracked and dry, you again have two specimens for examination by light microscopy or scanning electron microscopy.

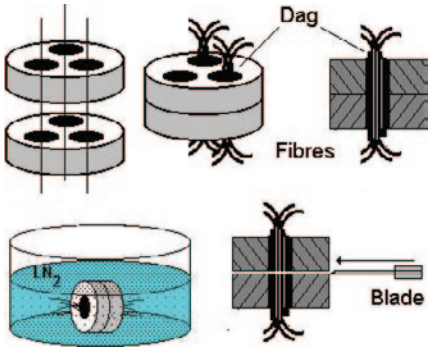

Figure 5: Diagram showing the technique that places the material in Aqua Dag between two stubs prior to fracture. The fibrous material has been forced to fracture by offering the support of the metal stub and the impregnation of an adhesive.

The most complex materials I have investigated by the latter technique were freezer bags, which were breaking in use. I spiraled each bag through the stubs, a bad one and a good one, soaked them in the carbon solution, and cracked them as described. What proved interesting was that the bags that failed were made up of only four layers; the good bags were made up of five layers, the fifth layer being aluminum. Whilst this work was company confidential, I have presented examples of freezer bag fractures in Figures 6, 7, and 8.

For those of you who have a cryosystem, similar techniques to those mentioned above may be used under total

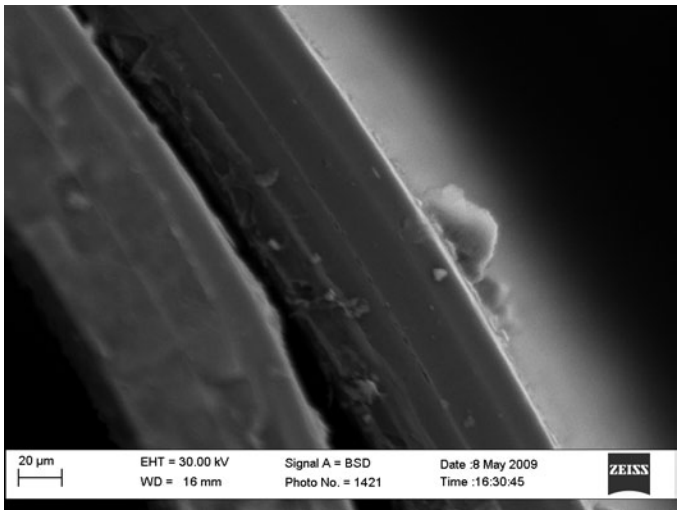

Figure 6: SEM micrograph of a four-layer freezer bag. A backscattered image of a fractured four-layer freezer bag taken at 30 kV.

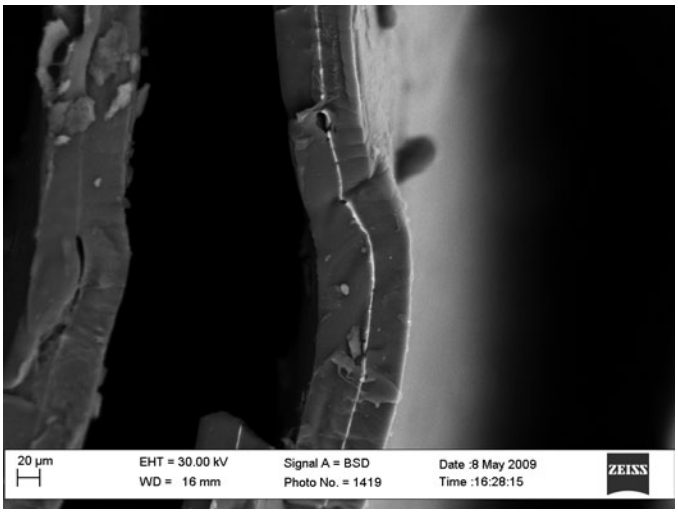

Figure 7: SEM micrograph of a three-layer freezer bag. The 30-kV backscattered electron image shows a middle layer of metal.

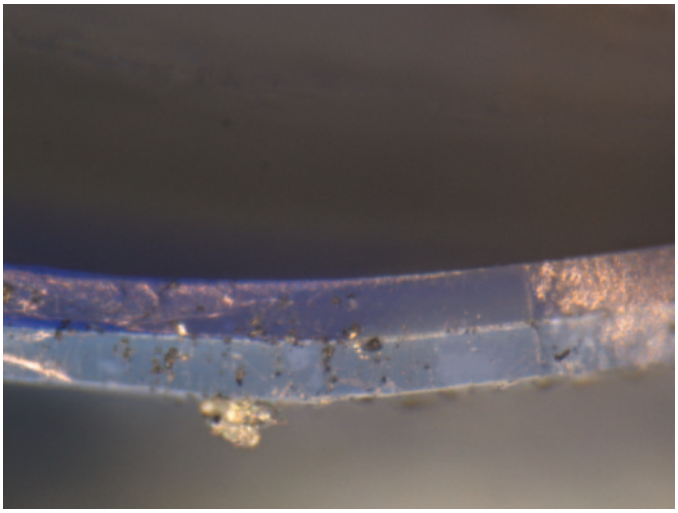

Figure 8: Light micrograph of the three-layer freezer bag. Note that the metal layer is less apparent than in Figure 7.

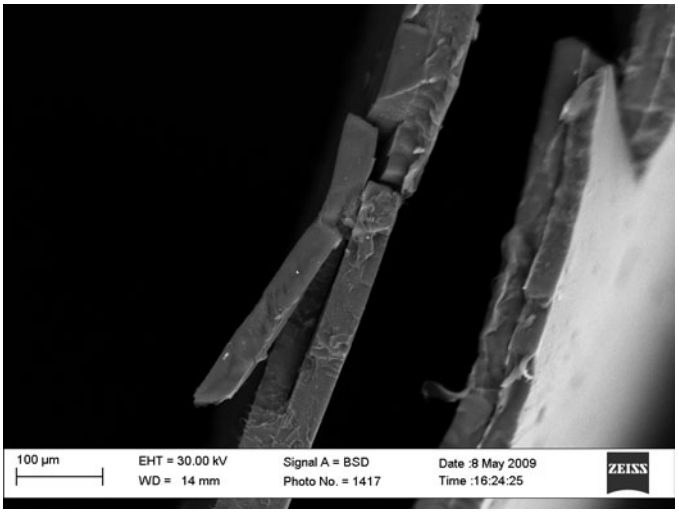

Figure 9: A broken fracture of a freezer bag. A 30-kV backscattered electron image demonstrating how components may fail to advantage when being fractured, the failure itself demarking the different materials.

cryoconditions. I have used the straw technique, mounted so the straw protrudes about 6 mm above the cryoholder, for liquid suspensions and fiber samples (mounted in water or other fluid media) [2].

A couple of final points. Firstly, be aware that when cracking a material, the fracture produced is similar to a metal fracturing, and some surface details may resemble this type of failure. However, the microstructure usually is not disturbed by the fracture, and a change in fracture plane level, through a mix of layers, often exaggerates the layers to advantage (Figure 9). Secondly, if the microstructure of the material is likely to be better displayed though the density of its phases, backscattered electron imaging in the SEM may be useful. In the latter case, higher accelerating voltages may help improve image contrast by increasing the collected signal.

Conclusion

Sometimes the simplest methods are the most effective. Cracking the sample is quick, easy, and revealing.

References

[1] P Echlin, *Handbook of Sample Preparation for Scanning Electron Microscopy and X-ray Microanalysis*, Springer, New York, 2009.

[2] AW Robards et al., *Low Temperature Methods in Biological Electron Microscopy*, Practical Methods in Electron Microscopy Series, Elsevier, 1985.

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