

SEM Questions

1. SEM is an electron microscopy technique used to image and map surfaces up to 50,000 times magnification. SEM fires beams of electrons at the surface of materials. The direction of the outgoing electrons can be detected to indicate characteristics -- including texture and composition -- of the specimen's surface. SEM can be used in combination with EDS to determine the elemental composition of the material being imaged. EDS links the x-rays induced by the high energy electrons with characteristic x-rays diagnostic of the presence of each element to yield composition information. Because the EDS is linked with the SEM, it is possible to map certain areas of the sample in question to create an elemental distribution.
2. SEM-EDS samples must fit on the sample mount, which is about 1.5 inches in diameter. Both organic and inorganic samples can be prepared for SEM analysis, but neither can have volatile chemicals in or on them, as they will vaporize immediately in the vacuum chamber. Furthermore, non-conductive materials (most organics, plastics, ceramics, and lithics, for instance) must be coated with a conductive copper or carbon layer to allow electrons to flow around and through the sample. SEM can image cross sections, flakes, and entire objects, however it is preferable that the samples are polished and/or flat.
3. EDS detection limits depend on the machine in question. In general, EDS can measure elements at .1 weight percent or more. Also of note is that since EDS is a sub-category of XRF, it cannot measure light elements (eg atomic numbers below ~10).
4. Samples must be flat and small enough (1.5 inch diameter) to fit on the sample holder. Additionally, metal samples must be etched and polished in order to observe grain boundaries.
5. SEM-EDS is non-invasive. It is only destructive if the sample is cut (to fit on the sample mount or to expose a cross section), polished, or etched.
6. All types of materials can be analyzed by SEM and EDS. The only limitation is that light elements won't be measured by EDS.
7. SE (secondary electrons) can give images and textures of surfaces. Backscattered electrons give topographic relief and indicate homogeneity by enhancing contrast.

XRF/XRD Questions

1. XRF uses characteristic x-rays scattered from atoms previously excited by primary x-rays to determine the composition of a bulk sample. It generally provides a readout of elements present, but provides no structural information about the sample or the arrangement of the atoms. XRD is used primarily to determine the identity of the crystalline phases of a material. Working backwards from recognized and unique spectra, it is possible to infer composition, but this is indirect information which can be much more easily provided by XRF. For the most part, XRD is only used to learn the identities of the crystalline phases of

materials, not composition. (XRD may be helpful for identifying crystals which contain light elements with atomic number $< \sim 10$, as XRF cannot detect those.)

2. XRD cannot identify any amorphous materials. That is, materials must be crystalline to yield resolved peaks on an XRD spectrum, otherwise peaks will appear broad and uninformative. Plastic, glass, fibers, and dyes are generally amorphous, and thus cannot be identified by XRD. Metals (especially in oxidized states), soils, ceramics (but not glass glazes), and lithics are usually crystalline and can be studied by XRD.
3. The upper limit of XRD and stationary XRF sample sizes is about two inches in diameter, which is the size of sample mount plate. On the other hand, handheld XRFs can be manipulated more easily and can thus measure larger samples. Samples should be larger than about a centimeter in diameter, as the XRF and XRD beams are not particularly fine and measure bulk composition.

Also a good reference/comparison table:

<http://www.materialinterface.com/methods.html>