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X-Ray Analysis of Materials: Avoiding the Pits and Other Practical Hints

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EDS analysis of samples in a SEM or TEM are usually portrayed as a very simple process; just image your specimen, turn on the x-ray analyzer and all will be revealed about the chemistry of your sample. Unfortunately, that approach can be very misleading at best, and disastrous at worst. The analyst should get used to preparing himself for EDS x-ray analysis just like he or she does for any other process. A set of preparation steps should be followed until they become an unconscious method to be used before accepting the information coming from the EDS x-ray spectrum. After proper basic preparation, interpretation of the results and the selection of operating parameters is still very important due to the detailed nature of the specimen.

Preparation: The most basic need is for the EDS system to be calibrated. This can be a difficult or simple task depending on the age of the system. Next, the basic geometry of the beam, sample and EDS detector needs to be considered. Before starting to analyze a specimen, the operator has probably put a good deal of effort into obtaining an optimum image. But the optimum parameters for imaging and x-ray analysis are rarely the same. The working distance for optimal imaging is usually much shorter than the optimum for EDS, so the sample must be moved. The operator should know the optimum distance for the EDS detector and what must be done to achieve it. Too low a count rate and shadowing of features will occur with working distances that are too far from the optimum. Also, to achieve higher count rates the beam should be well aligned. A bit of beam misalignment may not greatly affect an image but prevent optimum count rates from being obtained. In addition to working distance, the takeoff angle to the detector must be considered. At least 30 degrees of takeoff angle would be good practice, so the sample should be tilted to achieve that angle. Older microscopes may involve a large amount of tilt.

The beam voltage is very important to achieve proper excitation of the elements in the sample. In SEM, generally the overvoltage should be >2 but <10 to 20. In TEM, if the sample is truly thin, then the overvoltage just needs to be >2. If the sample is completely unknown to begin with, the elements must be found before making the final choice of voltage. So the process becomes one of trial and repeat. Too often operators become lazy and use one voltage to do all their work and do not optimize according to the situation. They may use 30 Kv and have too much absorption of light elements or too poor a spatial resolution.

The operator should also keep in mind the shape and size of the region from which x-rays are being generated. Since it is usually much larger than the features that can be imaged by electrons, that should be taken into account in the interpretation of the data.

Topology and Homogeneity: The topology of a sample can have the greatest influence on the quality of the data from the x-ray spectrum. The operator should learn to recognize from the spectrum itself when the data is being compromised by excessive absorption. The operator should be aware of the direction of the detector with regard to the image on the screen so that problems can be avoided. The worst case is trying to analyze a feature deep in a hole or pit in the specimen, where perhaps almost all or all of the x-rays generated are being absorbed. Careful orientation of the pit towards the detector may solve the problem and allow identification of the feature if it is not too deep in the specimen.

Homogeneity of a sample also has a large influence on the operating mode chosen. For example, if the problem at hand is to verify a type of metal alloy, which has inclusions or multiple phases, then operation at low magnification and a rastering mode is called for. This will attempt to "average out" the composition variations across the sample. This method is just a compromise, because the correct method to account for composition variations is too time consuming or difficult. When average atomic number changes are large from one area to the next, this method can lead to large compositional errors. The most common example of this is in the analysis of Pb-Sn solders. If a BSE detector is available to you, use it often to check for regions of differing composition. Let it guide you to finding areas of interest. Most materials samples presented for analysis in the SEM are only prepared to the extent that they can be placed under the beam. That is to say, the sample is to be examined as close to "as is" as possible. It may be coated to prevent charging. A light coating is usually all that is required. If a light coating is used, and the element of the coating is chosen so as to not overlap an element in the sample, no problems should occur. Check that the grounding is adequate to prevent a slow sample drift, which could affect a spot mode analysis or an element map.

In reporting data from an EDS system, the operator must have a good understanding of the limitations of the method. Minimum analyzable amounts, statistical precision and the accuracy of the technique should be understood. The people who bring you samples for analysis probably don't know these things and expect more than can be given. Learn the limitations so that they are not overstated or understated to your requester. ■

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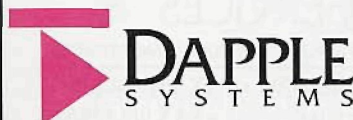

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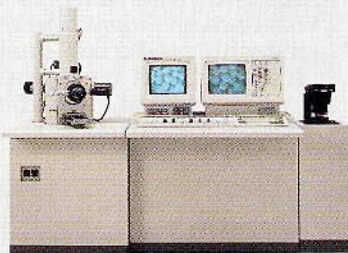

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