Characterizing Coarse Particles with Dual Energy Dispersive Spectroscopy (EDS) Detectors

Introduction
Performing EDS analysis on a specimen with coarse particles on the surface can be difficult because of shadowing effects as shown in the schematic in Figure 1. However, this effect can be mitigated in two different ways. One is to collect a set of maps from an area, rotate the sample 180° and then collect the same set of maps over the same area, rotate the results and merge them with the results from the original scan. A better approach, which we will explore in this technical note, is to use two well-balanced detectors with different azimuthal angles simultaneously and then merge the two sets of maps.

Dual Detectors
An example of two EDS detectors mounted on an SEM is shown in Figure 2. Using dual EDS detectors enables the shadowing effects to be mitigated. This approach is much more efficient as the area needs to be mapped only once. It also reduces any errors associated with registering the two sets of maps using a single detector. However, simply summing the two datasets together is not adequate to properly capture the missing data. A normalizing procedure is required. The best way to do this is to first select the maximum signal at each pixel and then divide this value by a factor based on ZAF correction.

Example
Figure 3 shows a secondary electron (SE) image from particles mounted on carbon tape, which is mounted on an aluminum stub. Many of the coarse particles in this field of view are roughly spherical and consist of Ti, Nb, and Al. In this case, the particles themselves are of interest, not the surface below the particles. The challenge is anticipating summing artifacts from the particles lying near each other.

Figure 1. (a) Schematic of an X-ray scattering at a single point; (b) shadowing exhibited in an element map.

Figure 2. Interior view from below of an SEM (JEOL 7000F) with dual silicon drift detectors.

Figure 3. Secondary electron image of coarse particles.
Figure 4 shows EDS maps from the two different detectors. The shadowing is quite obvious in these maps.

Figure 5 shows an EDS map constructed by simply summing the data from both detectors. The shadowing effects are reduced but are still present in the merged map. In this map, the question remains whether the darker hued areas are truly deficient of the mapped element or are shadowing artifacts. The maximum signal map with ZAF normalization eliminates this ambiguity.

Figure 6 shows the resulting maximum signal map after ZAF normalization. The shadowing effects are nearly eliminated. There are still areas in black which cannot be obtained using either detector. It should also be noted that the surface features in the carbon tape are eliminated as well, showing that the dual detector approach not only helps with the coarse particles but also reduces more subtle topology effects.

Conclusion
Dual detectors are very useful for resolving the shadowing problems associated with coarse particles. Similarly, dual detectors are helpful when solving similar problems on samples with rough surfaces, such as fracture or wear surfaces. In addition, having two detectors can result in more efficient collection of data on smooth surfaces. Essentially, the count rate is doubled by merging data from the two detectors.