AN 379
Automated Particle Analysis by SEM/EDS

May 7, 2007 (Version 3.0)

Discussion

The automated analysis of small particles by SEM/EDS is quick and efficient. Particles can be characterized by size, shape, and chemical composition to reveal the presence and source of contamination that can adversely affect the operation of devices that depend on clean components. It is also used to evaluate cleaning steps for process engineering and vendor evaluation.

Statistical particle analysis using a scanning electron microscope and energy dispersive x-ray spectroscopy (SEM/EDS) can often be a slow and tedious process when performed in a manual mode. Each particle first needs to be located in the secondary or backscattered image, the electron beam is focussed onto the particle, and finally, EDS analysis of the particle can be performed. For the analysis of even 100 particles, this type of analysis can easily tie up an SEM for a full day.

EAG has an automated SEM/EDS system for inorganic particle analysis. The system is designed for analysis of particles having diameters 0.3 µm or larger and provides information regarding particle size, shape, and chemical classification of each particle as well as the particle location. Typical analysis runs range from 1 - 3 hours per sample, in which time hundreds of particles can be identified and classified. Figure 1 shows a typical analysis set-up for a single sample where a representative area near the center of the sample will be analyzed for particles 0.5 µm to 20 µm in size. This area is divided into a number of frames that will be sequentially scanned for particles. A semi-quantitative EDS spectrum is acquired for each particle. The number of frames is dependant upon the total set-up area selected and the minimum particle size chosen for the analysis. For this example each frame is equal to 0.079 mm^2 and there are 397 total frames making the total analysis an area of 31.36 mm^2. Automated particle analysis can be set-up to examine one sample or several samples as shown with the multiple sample set-up shown in Figure 2. Since multiple samples can be loaded at the same time, operator involvement is greatly minimized.

Particle detection and recognition is based upon backscattered electron images taken of each frame in the analysis area. Figure 3 shows a superimposed thermal colored backscattered

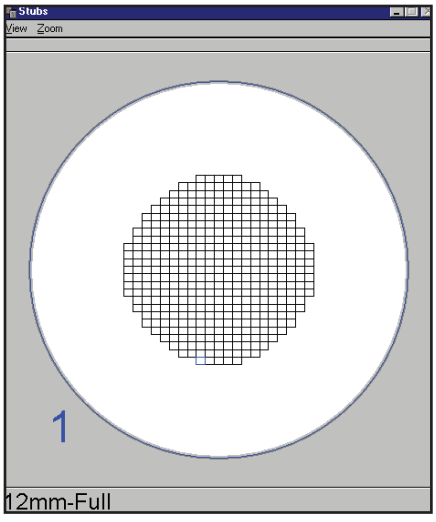

Figure 1. Single Sample Layout

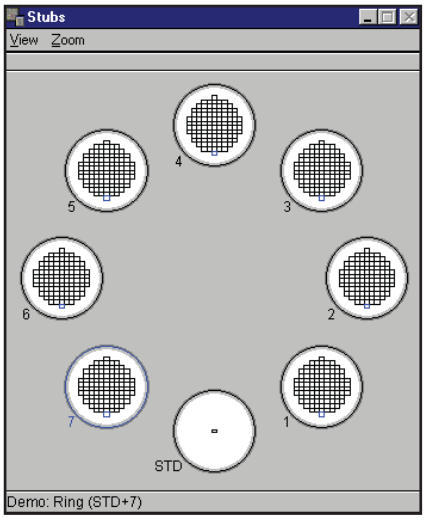

Figure 2. Multiple Sample Layout

electron and secondary electron image from a magnified area of a single frame revealing many particles against a field of 0.2 µm filter pores. Once the frame has been scanned, EDS data is collected from each particle that falls within the selected size range. When the analysis of the frame has been completed, the stage automatically moves to the next frame and the process is repeated until the analysis has been completed or terminated by the operator. An example of a summary result is shown in Figure 4.

Samples for analysis can be obtained in many ways. The preferred method is to have components of interest sonicated in some solvent to agitate the particles from the component and put them into solution. The particles in solution are then re-deposited onto a membrane filter by vacuum filtration. This method has the advantage of reducing the number of agglomerated particles and results in more even particle distributions on the filter. Another method is to use double-sided adhesive tabs to lift debris directly from the surface of a component or area. This simplifies sample collection but particles are frequently agglomerated or layered which complicates accurate chemical classifications.

Automated particle analysis is an ideal method for determining chemical contamination that can often lead to the source of the contamination and can also be used in process engineering to evaluate vendor supplied components or cleaning methods.

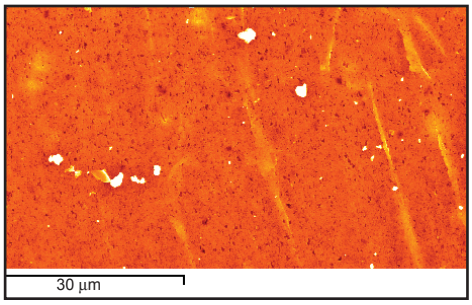

Figure 3. Backscattered Electron Image

Particle Analysis Summary

Area Done (%) 21%
Area Analyzed 3.82 mm²

Type	Number
Al Metal Oxide	3
AlTiO	1
AlArO	1
AlAu	1
AlFeMgO	1
AlO	49
AlTiO	12
AlTiO	31
Au	3
CrFeO	3
CrNiFe	12
Cu	1
FeO	1
SiO	1
Ti	50
TiO	18
ZrO	1
Total	**189**

Particles/mm²	49.48
Particles/Frame	3.15

Figure 4. Summary Report

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