# Large area X-ray mapping in SEM/EDS

SEM/EDS (Scanning Electron Microscopy and Energy Dispersive Spectroscopy) mapping measures elemental distributions in a sample. Typically this technique produced an analysis of one field of view by scanning the electron beam in a grid pattern to produce a map. This document discusses using Thermo Scientific<sup>™</sup> Pathfinder<sup>™</sup> X-ray Microanalysis System to control SEM sample stage motors to acquire extremely large X-ray maps by acquiring multiple fields of view in a grid pattern and combining those maps into one large X-ray map. This expanded field of view is easier to comprehend, captures X-ray maps larger than the field of view provided by the SEM, and can capture features missed when analyzing just a limited field of view.

#### Key words

X-ray mapping, stage automation, Pathfinder X-ray Microanalysis Software, EDS maps

#### Introduction

Element distribution maps made by SEM/EDS have been acquired by analysts for over five decades. Initially the method was to write a white dot on a sheet of photographic film each time an X-ray for a given element was detected as the electron beam was scanned. This gave way to digital X-ray maps in the early 1980s.

Figure 1 shows a digital X-ray map from ca 1985. Note that this required about an hour to acquire relatively few X-ray counts.

Since then every aspect of SEM/EDS mapping has improved a great deal.

SEMs are much more capable of focusing many electrons into a small spot. In addition, many modern SEMs are equipped with motorized stages that can be controlled by the EDS system.

Computers, storage systems and displays are all much more capable. Processing just one spectrum used to require over five minutes. Today, many thousands of spectra are processed per second.



Figure 1: Digital X-ray map from ca 1985. This required about an hour for acquisition.

Most importantly, the EDS detector itself is far faster than before. The Si(Li) detector design has been superseded by the Silicon Drift Detector (SDD). Even the SDD has seen many generations of improvement. Today, the Thermo Scientific<sup>™</sup> UltraDry Silicon Drift Detector can acquire X-ray data at the rate of several hundred thousand counts per second while providing resolution equal to or better than the Si(Li) detector. Moreover, newer UltraDry models offer large solid angles that provide more X-ray counts at lower beam current. Even faster measurements can be achieved by using two SDDs at the same time thereby doubling the throughput.



#### Acquiring very large area maps

Combined with the newest Pathfinder software electronics, the entire SEM/EDS system can now produce high quality X-ray maps in minutes.

The constant theme running through the decades is that the analyst chooses a particular field of view to measure and report to those requesting data.

The vastly improved capabilities of modern equipment provide the analyst with the opportunity to expand the view presented to requestors and collect very large X-ray maps by collecting many individual maps and merging those into one large map. The resulting maps can be over tens of millimeters on one side.

The time required to produce a wide area map correlates with the size and level of detail of the map. The assertion here is that very large maps can be acquired in the reasonable time of one to three hours, a half day of instrument time. For a valuable sample which took perhaps some length of time to obtain and a day or more to mount and polish this is a reasonable amount of time considering the value of the data produced.

Because of the difficulties in collecting information quickly, analysts often collect maps only from a small field of view.

It takes imagination to see how this bit of information fits into the overall structure of the sample. A large field of view provides a scene which is much easier to interpret, especially for those not accustomed to viewing SEM/EDS data.

A larger field of view can show broad distributions of features in a sample, not just one or two examples as provided in the past. It can show relatively rare features that might easily be missed when looking at just one or two areas. This is important because the composition of a sample is often not apparent just by observing the electron micrograph. It also maps distributions that are larger than the field of view provided by the SEM, providing views that were otherwise not possible.

Large area mapping by SEM/EDS is much faster than analysis by some other techniques because most of the spatial information is acquired by moving the electron beam which is very fast. Relatively little slower stage motion is required.

Finally, because Pathfinder software mapping saves X-ray spectra at each point in the map this rich data set can be revisited in the future to find the composition at any point in the region analyzed.



Figure 2: Array of three rows and four columns of images representing 12 SI maps acquired in a grid pattern.

#### Technique

Thermo Scientific<sup>™</sup> Pathfinder<sup>™</sup> X-ray Microanalysis Software has a feature, Analysis Automation, that controls the SEM stage while acquiring Spectral Imaging (SI) X-ray maps. Analysis Automation moves the sample stage in a grid pattern or it can utilize a set of random locations. The set of locations can be saved for future use.

To collect a large map, the analyst sets up the system just as for collecting a single map. The SEM beam current and kV are set, the associated image size is set, the map array size is set and the pulse processor time constant is selected.

Next, the analyst sets a termination criterion telling the system how much data to collect in each map. Maps can be stopped after a fixed time or after acquiring a specific amount of data.

Finally, Analysis Automation is invoked to set the pattern of locations to analyze and the start button is pushed. The data are acquired without intervention and stored on the mass storage system.

Individual maps may be processed or the entire data set can be processed as one large map.

To create a tiling of the entire data set, the analyst selects elements, data type and related parameters just as when viewing a single map. Results may be calculated as atomic percent, weight percent or simply net counts. Next, batch processing is invoked and the name of the data set is selected. Pathfinder software then calculates a result. The many individual maps are treated as one large map. The result is a series of images in one data set. There is one image for the electron image and one for each element selected. Each image is as large as all of the individual maps tiled together.

The element maps can be overlaid on the image. The brightness and contrast and color of each map can be adjusted.

#### **Example results**

Figure 2 shows an analysis of a ceramic brake pad sample. There is a grid of four rows and three columns of images representing twelve SI (Spectral Imaging) maps. Each map is a 512 x 384 array of spectra. These were acquired at 15kV. Each map required about five minutes to acquire.

Figure 3 shows the resulting image after merging the individual images.

Figure 4 shows the net counts maps for all of the maps calculated from this data set. Net counts maps have the X-ray continuum background removed and separate overlapped peaks.

There is so much data that a large monitor is useful to view it. Alternatively, printing the data as a poster can be very helpful. It allows several engineers to view and compare multiple maps while discussing the results.



 Figure 3: Image formed by merging together of the separate images shown in Figure 2.





Figure 5 shows some selected elemental maps of the same sample overlaid on the SEM micrograph. This illustrates several features of the sample.

It is immediately clear that materials containing Ba are very common. The Cu threads and Cl rich material (possibly a PVC) though large, are relatively rare. It would have been easy to select a field of view that omitted these materials. The Sb rich particles (actually SbS) are small and not evenly distributed. Imagine collecting the same twelve maps but printing them in a booklet form. The end requestor would have to flip through these one after another and would not have the benefit of the overall view. People are much better at extracting detail from a large, complex view than from a sequence of images ordered in time.

In addition to calculating element maps Pathfinder software has two more tools, COMPASS and Phase, which extract multi element phases from SI maps. Figure 6 shows two phases from the brake pad sample, (Ti, K oxide and SbS) overlaid on the micrograph. This shows at a glance the typical shapes, sizes and distribution of these materials.

Figure 7 shows two phases, a Ca ceramic (Mg, Al, Ca silicate) phase and a C and Cl (possibly PVC) phase. Again, at a glance it is easy to see the typical size, shape and distribution of these materials. Even though the C and Cl phase is rather large it would be easy to ignore it. In fact, because it is so large many analysts would tend to ignore it intentionally as an artifact and focus on the smaller, more common features. This is one advantage of acquiring data from a large field of view. It reveals large areas of the sample, not just those chosen by the analyst. What might be dismissed as an artifact may, in fact, be critical information.



Figure 7: Phase overlays of Ca rich ceramic (Mg, Al, Ca Silicate) phase shown in purple, and a CCI (possibly PVC) phase shown in green.



Figure 8: Silicon solar cell. The thin traces are composed of Ag paste.



 Figure 9: Segment of the Te map overlaid on the micrograph showing one Te inclusion.

Figure 8 shows a photograph of a silicon solar cell. Large area mapping was used to analyze the composition of one of the metallic traces over a long distance. These are nominally composed of Ag paste. In fact, analyzing many typical fields of view would show only Ag. Only when measuring over more than a centimeter of the trace did small areas of Te inclusions appear.

This measurement was similar to collecting a line scan. Two hundred fifty seven maps were acquired in one row. Each individual map was just 64 x 54 spectra. The pixel resolution is 2.1 microns. Each image is 512 x 442 with a resolution of about 0.27 microns. The total size of this map is about 3.5 cm by 115 microns. Each map with image took less than a minute to acquire. The entire acquisition time was about three hours.

Figure 9 shows a segment of the Te map overlaid on a micrograph. This is net counts data with the background signal removed.

There were long stretches of this sample showing no evidence of Te. If asked to characterize this sample an analyst might have inspected many areas and not having seen the Te simply reported the trace as pure Ag.

It was noted that Te appeared mostly where the trace appeared damaged.

Figure 10 is a spectrum obtained from one inclusion confirming the presence of Te.

For an engineer familiar with this product this kind of information is very helpful as it shows the kinds of defects present, their size and their frequency. This is difficult to obtain from viewing selected areas.

This data set has an aspect ratio of about 305 to 1. Enlarging the image to appear 10 cm high makes the image appear over 30 m wide. However, if there are any flaws in this material due to corrosion or a manufacturing flaw, then this detailed view is of great value in finding them.

This example illustrates the fantastic magnitude of data acquisition possible with the Thermo Scientific<sup>™</sup> UltraDry EDS Detector, and Pathfinder software with Analysis Automation.



Figure 10: Spectrum from one inclusion confirming the presence of Te.

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#### Summary

Analysts now have one more powerful tool in their arsenal to examine samples.

Large area maps whether in rectangular grids, a linear array or as random locations, allow the analyst to collect vast amounts of data from samples with little more effort than needed to collect a single X-ray map.

Improvements in SEM technology, the high throughput of the UltraDry Silicon Drift Detector, the speed of the Pathfinder software electronics and the improved capabilities of modern computers all combine to make this technique accessible to analysts. Because of the improved technology these large data sets can be acquired in a reasonable length of time.

For some samples, producing a map of a wide field of view makes the result more understandable to those viewing the data, and makes it more inclusive by covering more of the sample. The result is also more reliable because changes in composition are not always indicated in the SEM micrograph so covering more area in a map increases the chance of a more comprehensive analysis. All of these benefits improve the quality of the work provided by the analyst.

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