

Introduction to Automated Particle Analysis by Focused Electron Beam

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INTRODUCTION

This little document is intended to provide a brief entry-level introduction to the concepts and technology employed in the automated particle analysis systems manufactured by ASPEX Corp. My objective is to convey a big-picture sense of what our instruments do, and roughly how they do it. Along the way, I'll introduce some of the buzz-words that are used in our area of technology. My ultimate objective is quite limited – I'm not going to try to explore all of the interesting variations and applications of electron beam imaging technology and we're going to pretty much ignore the fascinating physics involved in this instrumentation – instead we'll be trying to home in on the most basic things one needs to know in order to understand how/why our specific products work.

That being said, however, one can hardly talk about automated electron beam technology without discussing the Scanning Electron Microscope (or SEM as it's usually known). As you can gather from its name, the SEM is a microscope – that is it makes little things appear big so that we can study them. The way it does this, however, isn't at all like the way a familiar light microscope or magnifying glass works.

SEM FUNCTIONAL OVERVIEW

Figure 1 illustrates the way a SEM creates an image. The most important thing in this figure is the beam, which is a focused stream of electrons. The function of the column is to generate the beam and focus it down so it is a really tiny spot at the place where it hits the specimen. (How tiny is the spot? You could easily fit a million of them into the period at the end of this sentence.) The column can also laterally *deflect* the beam so that the location where the *beam spot* strikes the specimen can be precisely controlled. The electrons in that focused beam are traveling very fast, so complicated things happen when they hit the specimen. One thing that happens is that some of those energetic electrons just bounce off the atoms of the specimen: we call those Back-Scattered Electrons (BSE). Another thing that happens is that some of the incident electrons get absorbed in the specimen, at the same time knocking other electrons loose from the atoms of the specimen, and we call those newly liberated ones Secondary Electrons (SE). Also, the absorbed electrons give some of their energy to the atoms they strike and we say that those atoms are "excited" by the beam. One way that an excited atom can get rid of this extra energy is by emitting a photon – if it's a low-energy photon, we see it as visible light, but the most important photons are the high energy ones we call X-rays. In

other words, the impact of the beam gives rise to a whole bunch of different kinds of emissions and it turns out that each carries different information about the local properties of the specimen. For each kind of emission we can use a specialized detector that "sees" those emissions and produces a proportional electrical signal. Each of those different kinds of signal depends on some property of the specimen so as we move that focused beam spot across the specimen, the signal gets "brighter" or "darker" depending on how that property varies across the specimen. We call this variation in signal brightness *contrast* and we make use of it to generate a picture. We do this by moving the electron beam rapidly over the surface of the specimen, and as we do this we simultaneously change the brightness of the corresponding pixel of the image screen to correspond to the strength of the signal. So where the signal from the specimen is strong, we see bright pixels on the screen, where it is weak, we see dark pixels. The overall pattern of varying pixel brightness forms a picture of the specimen.

Now it's important to note that we've drawn Figure 1 with really distorted scales. Specifically, the electron beam is about the diameter of a human hair at its widest point (where it comes out of the column) and converges down to a much smaller spot where it hits the specimen. Also, the angle of deflection we're depicting in this figure is much larger than is usually the case. Instead, we typically sweep that really tiny beam of focused electrons over a comparably tiny region of the specimen – say a square $1/100^{\text{th}}$ of an inch on each side. When we then display the resulting pattern on our display screen, the dimensions of the screen might be 10 x 10 inches – in other words, we are producing a picture that is magnified 1000 times (10 inches divided by 0.01 inch). By choosing how big an area we want to scan, we can thus vary the magnification of our picture – by scanning a bigger area, we reduce the magnification, by scanning a smaller area we increase it. Of course, at the same time we're doing that, we also need to make sure that the size of the focused beam spot is appropriate. It's kind of like painting: you can't paint a clear miniature picture with a big brush, nor can you efficiently paint a large picture with a really tiny brush. Just as you want to match the size of the brush to the job, we want to make the diameter of the electron beam spot corresponds to the area represented by a single pixel on the display screen.

The last thing we need to point out in Figure 1 is the stage. In one sense, the stage is nothing more than the platform we lay the specimen on. In practice, however, it's a lot more important than that. First of all, let's note that the object we've

depicted as a specimen is supposed to represent a piece of filter paper. When we're looking at particles with our electron beam, it's quite common to deposit them on a filter paper or something similar. We need to realize that the particles we are looking at are so small that they probably aren't visible to the unaided eye (and some of them will still look like specks when viewed under a light microscope). At the same time, the filter paper we have collected them on might be an inch or two in diameter, so as one person put it, "it's like looking for BBs on a football field" (and that's actually a bit understated!) In order to cover the entire area in sufficient detail, we need to break it down into a series of smaller *fields*. We will detect all the particles in a field and then move the specimen to the next one and repeat the process. So the ability to move the stage in small and precise increments is really important to our process (we don't want gaps or overlaps between the fields we analyze).

Another way that Figure 1 is oversimplified is that it looks like we're examining our specimen out in the open. That's not the case. You need a pretty good vacuum in order to maintain a focused beam of electrons because the electrons in the beam would be scattered all over the place if they encountered air molecules. So an important aspect of the instrument is the *vacuum chamber*.

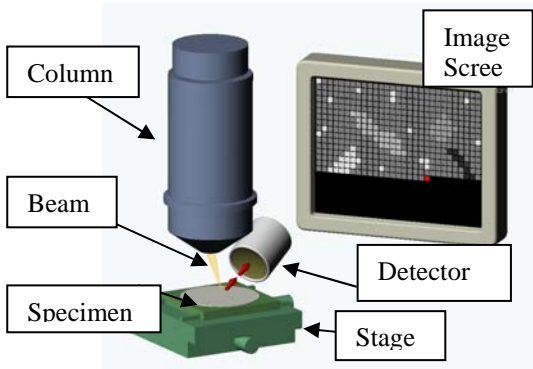


Figure 1: System Diagram

Figure 2 is a little more detailed than the first one. Once again, we have the focused beam being produced by the column hitting the specimen mounted on the stage. This time we have shown the specimen stage mounted inside of a vacuum chamber. Not shown in this figure, but obviously necessary, are the pumps and other equipment needed to evacuate the vacuum enclosure, but we don't need to go into those details here.

Another thing we've added to this figure is that we're showing the three principal kinds of detectors that are used for SEM: the Secondary Electron Detector (also called the SED), the Back-Scattered Electron Detector (also called the BSED), and the x-ray detector, also called the EDX spectrometer. We're not going to worry very much about the SED, since although it's the most common kind of detector used for making "pretty pictures," we don't use it for automatic particle analysis. On the other hand, the BSE detector (BSED) is really important to us, so let's see why. You might have a hard time seeing the BSED in the above figure, because it's a pretty small circular device tucked right under the pointed end (*polepiece*) of the column. Figure 3 shows an exaggerated picture of what it actually looks like and does.

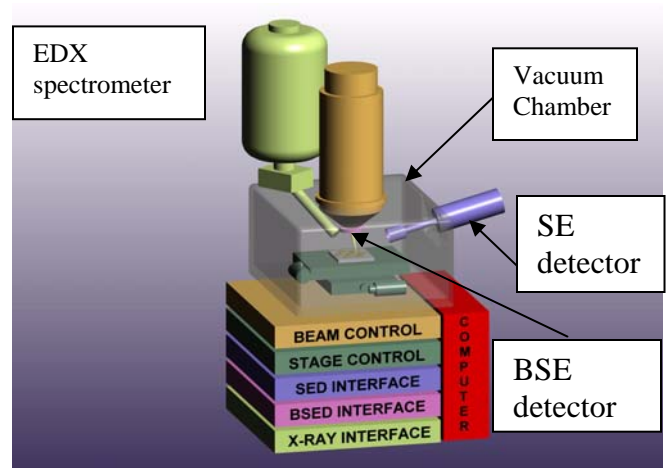


Figure 2: More Detailed System Diagram

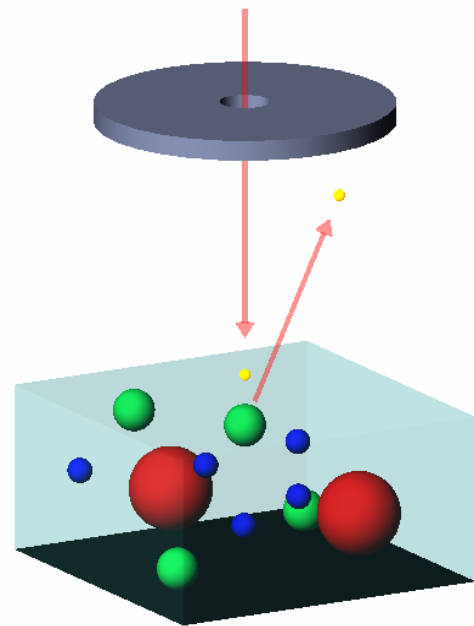


Figure 3: BSED Diagram

BSED IMAGING

That round washer-like device represents the BSED – it's about 1 inch in diameter. It has a hole in the middle where the electron beam passes through on the way to the specimen. The figure shows one of the incident electrons hitting an atom of the specimen and bouncing backwards so that it will hit the detector. I trust that it is obvious that the scale of this picture is all out of whack – electrons and atoms aren't remotely that big in comparison to the detector! But this picture still helps us to appreciate why the backscatter detector is so useful. As the figure suggests, any solid material is mostly empty space – the atoms are actually spaced quite far apart and so the incident electron may penetrate quite a ways before it hits anything. As you might also expect, the probability that the incoming

electron gets bounced back to the detector is going to depend on the size of the atoms in the specimen – big atoms are easy to hit and will reflect a lot of electrons, whereas small atoms aren't going to get hit so often. That's exactly what happens and that's what Figure 4 shows.

The vertical scale of the graph is the *back-scatter coefficient*, which is just another way of saying that it's the probability that an incident electron is back-scattered. The horizontal scale is atomic number, which is proportional to the size/weight of the atom. Thus, materials with low atomic number (at the top of the periodic table) produce relatively few backscattered electrons and "heavy" elements (at the bottom of the table) produce a lot more. Consequently, materials that are mostly made up of carbon, hydrogen, nitrogen, and oxygen (*organics*) produce a weak backscatter signal and look dark in an image. Those things we call *minerals* are somewhat heavier and appear a bit brighter, alloy metals (steel and brass) are still heavier and appear brighter still, and so forth. This is a very useful effect since it allows us to easily discriminate classes of materials just by looking at the brightness of the backscattered electron signal.

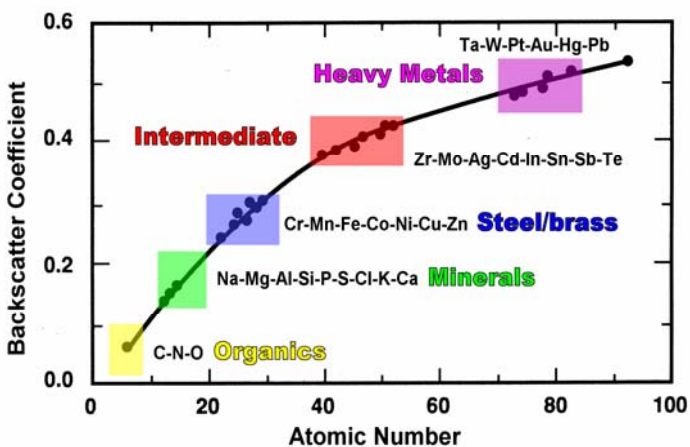


Figure 4: Backscatter Coefficient Vs. Atomic Number

To illustrate how useful the Backscatter Detector is, look at the pair of pictures in Figure 5. The picture on the left was taken with an ordinary light microscope at about 50X magnification. It's a picture of metal particles removed from an automotive mechanism and mounted on a carbon-based adhesive. It's really pretty hard to discern how many particles are there and what their shapes are. Now look at the image on the right. This is exactly the same sample imaged via the BSED signal. Here the carbon-based adhesive looks essentially black (because it is low atomic number) and the metal particles stand out clearly. Which image do you find easier to interpret? And if the BSED image is a lot easier for you, imagine how much easier the problem is for computer software, which is still a very long ways from matching the sophistication of the human visual system!

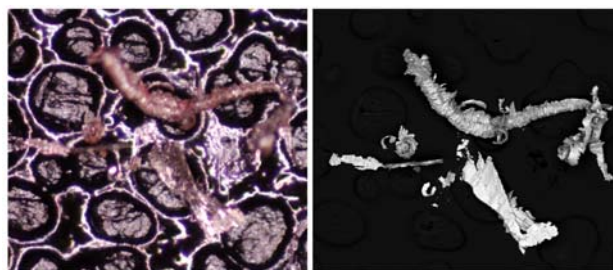


Figure 5: Optical Image and BSED Image

ADVANTAGES OF SPECTROMETERS

But though the BSED signal is an important tool, the x-ray signal may be even more powerful. The reason for this is because the energy (or wavelength) of the x-ray emitted by an excited atom is unique to the element that emits it. What this means is that if we measure the energies of the emitted x-rays, we can tell exactly which elements are present in the specimen, and in what proportion. We call the x-ray detector a *spectrometer*, because it allows us to view the *spectrum* of x-ray energies that are emitted. We're not going to spend a lot of time discussing this fascinating piece of technology, but Figure 6 shows what an actual x-ray spectrum looks like.

Each of the peaks in this spectrum represents a particular emission, and labels have been applied to indicate the elements they arise from. This particular spectrum contains silver and iron emissions (the specimen was flecks of silver-coated steel). Now, if you're not used to looking at spectra like this, the pattern of peaks may seem mysterious, but x-ray emissions actually follow precise and relatively simple rules that make it fairly easy to identify what elements we are looking at. And these rules are really robust – it doesn't matter what we do to the specimen, whether we heat it, form chemical compounds, apply pressure, roughen the surface, or whatever – the characteristic spectral lines for iron (for example) will always be found at the energies shown above.

So let's stop and summarize for a moment. What we've described so far is a technology that allows us to look at really tiny fragments of material and not only easily discriminate important classes of materials (i.e., minerals and metals) from the organic substrate on which they are presented, but once we have located these particles, we can tell precisely what their elemental composition is. And this works for a wide range of particle sizes – from micron scale on the small end to millimeter scale on the large end. Oh yes, and we can also take very detailed pictures of these tiny objects, so we know just about everything there is to know about them. And one last thing to note: we don't have to work very hard to prepare these samples for analysis. In fact, often it's nothing more than collecting the particles on some sticky tape and putting them into the SEM. Are you impressed? You should be! The power and versatility of the SEM/EDX combination is such that any materials laboratory worthy of the designation probably owns one (or several) and uses it as one of its principal tools.

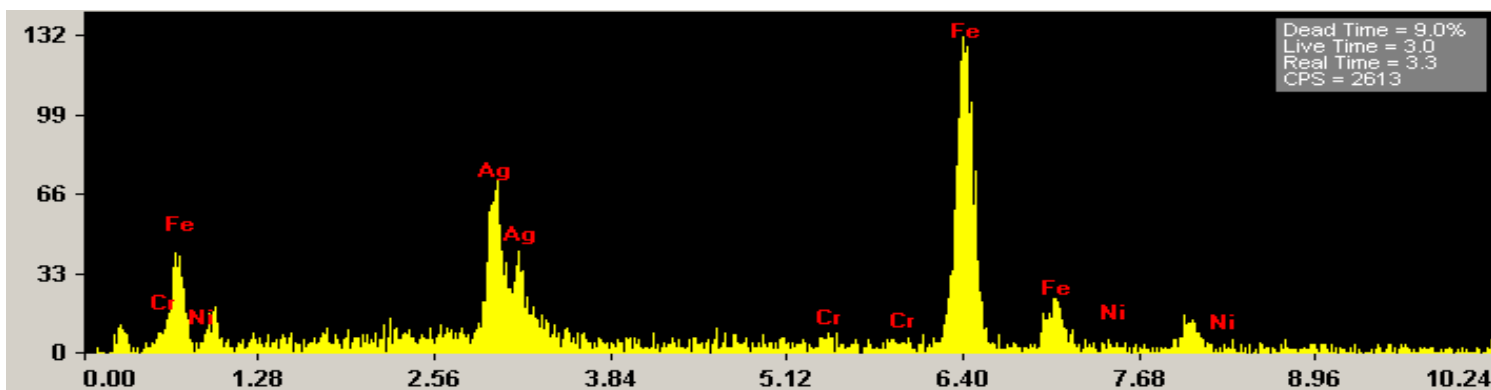


Figure 6: X-ray Emission Spectrum

SEM/EDX isn't a "perfect" tool, of course, and so we'll briefly itemize some of the limitations. Probably the biggest limitation is that pesky vacuum requirement – the simple fact is that there is no such thing as a material that is transparent to the passage of electrons. Not only does this mean that we have to get rid of the air in our specimen chamber, it also means we can't look at things inside transparent containers like we do with a light microscope. Further, since most liquids and many solids evaporate at low pressures, there are lots of materials that we can't look at in a completely natural state (like biological tissue, for example, which dries out and shrivels up when we put it in the vacuum). Then there is the fact that electrons are charged particles, and there can be a problem with charge build-up when we try to view insulating materials. Finally, we need to recognize that although x-rays are emitted by all but the three lowest elements in the periodic table, x-ray analysis isn't a very good tool for quantifying organic compounds (which are mostly hydrogen, oxygen, and carbon). But, in the grand scheme of things, those are relatively modest limitations to an extremely powerful technology, and by using specialized instruments and techniques many of these limitations can in fact be worked around.

ASPEX AND AUTOMATED ANALYSIS

What we've described so far is a relatively conventional SEM/EDX system. In fact, we could be describing pretty much any one of the tens of thousands of SEM/EDX installations that have been in use around the world for the past 30+ years.

With a skilled operator sitting at the controls, any one of those tens of thousands of units could do a credible job of examining selected particles of interest. So if that's the case, what's so special about the ASPEX system as a particle analyzer? The answer lies in that word "selected." The vast majority of those tens of thousands of conventional SEM/EDX systems are useful only for manual operation by a human operator. They are powerful machines that augment the human sensory system, but are completely non-functional unless human hands are present to manipulate their controls and human brains to interpret their results. It's a powerful combination when the task requires insight and imagination, but if it's a routine repetitive task, it's very inefficient.

It's now time to mention the most important parts of the system diagram in Figure 2: the supporting blocks of interfaces and controls and the way they are all interfaced to a controlling

computer. That's the key to the power of the ASPEX systems for automated particle analysis. Rather than requiring a human operator to make decisions and operate controls, these automated systems are pre-programmed for their task, and then accomplish it unattended. The net result is that they can analyze collections of particles orders of magnitude faster than any human can – and do it round the clock without a break, and make fewer errors as well! We'll now spend the rest of our time seeing specifically how this is done.

One way we could automate the process is what is called *frame-based* analysis. In this mode of implementation, a conventional SEM is used very much like a camera. Figure 7 shows how frame-based particle analysis is done with a camera.

The frame-based analysis begins when the stage is positioned so that a field of view is presented to the camera (left figure). The camera then "snaps" a picture of that field and transfers it to the computer (middle). Software algorithms in the computer then process this frame by locating the individual features and, by tracing around them, measuring them (right). As a final step, the computer may then direct the microscope optics to place the beam at the coordinates of a particle so that an x-ray spectrum can be measured. Once all the particles in the field have been analyzed, the stage is stepped to the next field, and the process is repeated. If we're doing this with a SEM and we are being smart, we've used our BSE detector to collect our images so that we've gotten a nice clear discrimination between our particles and the substrate they're lying on. Because it is all automated, it can perform a lot faster than any human operator. This all sounds pretty good, but it can be done a lot better.

PARTICLE DETECTION

Here's how ASPEX systems perform the same task. Once again, the stage is positioned so that a field of view is centered under the optics. Instead of just snapping a picture of the field, however, ASPEX systems may subdivide this larger "stage field" down into smaller "mag fields" that can be individually defined by deflecting the beam. (Note how the larger stage field is divided down into 16 individual mag fields in Figure 8.) This is done so that we can work with lots of smaller manageable-sized fields rather than a few big ones. And since we're moving between fields electronically, this is a lot faster and more accurate than mechanically moving the stage.

Rather than capturing a high-resolution image of the field, however, the ASPEX system instead moves the beam across this field in an array of fairly coarse steps. At each point, the brightness of the BSED signal is noted. If the signal is bright enough to indicate that a particle is present at this position, then the software initiates a particle-sizing sequence as will be illustrated shortly.

Note the coarse grid of sampling points in Figure 9. The spacing of the points is chosen to be no larger than the smallest particle of interest, thus at least one “hit” is assured for every particle of minimum size. However, particles smaller than the grid spacing will frequently be missed. In other words, the effect is like using a “seive” where particles too small to be of interest are allowed to “fall through.” That saves a lot of time.

PARTICLE DATA ACQUISITION

When a particle is detected, a sizing sequence is initiated. There are several algorithms that can be used for this purpose, but for simple particle shapes, the “rotating chord” algorithm is both accurate and exceptionally fast. Figure 10 shows the steps of the method.

The first step is to locate the center of the particle. This is done by a bisected chord method as follows: (Left): The centering sequence begins when one of the sampling points hits a particle (signal above detection threshold). The beam is then moved horizontally in small steps until the signal again falls below threshold. This defines the first horizontal chord. (Middle): The beam is then moved to the center of this horizontal chord and then stepped first upwards until the upper extent of the particle is located, and then downwards to the bottom edge. When this is accomplished, this vertical chord is bisected and a new horizontal chord is established. (Right): By repeating the process of drawing a vertical chord, bisecting it horizontally, then bisecting the horizontal chord vertically, the procedure converges quickly to a point where the vertical and horizontal chords cross at their respective centers – this is the geometric center of the particle.

Once the particle center is found, the last step is the “rotating chord” process illustrated in Figure 11. A

series of chords are drawn through the particle center at equal angular spacing as shown. These chords allow us to provide some very useful measures of the particle size and shape. For example, the longest and shortest chords are a measure of the aspect ratio, or we can average the chord lengths for an average diameter. By connecting the tips of the chords, the perimeter and area of the particle are also determined.

Finally, the beam is placed again in the center of the particle, and its x-ray spectrum acquired.

Though this may sound like a great deal of activity, it all happens very fast. In fact, for the vast majority of cases, this procedure locates and sizes particles several times faster than the frame-capture method (which is way faster than a human could do it). The major reason for this improvement in speed is because it only spends time collecting detailed data where particles are known to be present, rather than wasting time capturing and transferring vast numbers of “empty” pixels. Since there is almost always much more empty space on the

specimen than space occupied by particles, the result is a big speed advantage.

This method is also intrinsically more accurate since the chords can be drawn with arbitrarily fine spacing, whereas frame-based methods are restricted to the pixel spacing of the

captured frame image. The advantage of the ASPEX method of dynamic sizing is particularly apparent when both large and small particles are present in the same field. For a frame-based method to deal with this situation either the pixels must be made rather coarse (at the expense of precision in measuring small particles) or a huge array of small pixels must be captured, at the expense of speed. By contrast, the ASPEX method of dynamic sizing has no difficulty dealing with both large and small particles in the same field – it adjusts to the precision needed.

There’s a great deal more that could be said about this methodology, but this isn’t the place for such detail. However, it probably should be mentioned that the rotating chord method will obviously fail for certain kinds of complex particle shapes as illustrated by the two shown in Figure 12. ASPEX has also developed a “complex feature” dynamic sizing algorithm that accurately handles shapes of arbitrary complexity, albeit at some cost in speed.

PARTICLE CLASSIFICATION

Once each particle is fully characterized (size, shape, and elemental composition) user-defined “rules” are then used to assign the particles to meaningful classes. For example, aerosol particles might be classified by their size, aspect ratio, and composition to correspond to various inhalation risk categories. Again, this is automatically done by the software.

Once all of the analysis and characterization is performed for one mag field, it jumps to the next mag field and does the same thing until all of the mag fields have been analyzed. At that point, the stage is moved to the next stage field and the whole process repeated until the predefined area of the sample has been covered.

How fast does this all go? It depends a lot on the specimen and what we are trying to accomplish. In favorable cases, if we don’t need to collect x-ray spectra, we can process particles at rates of something like 500/minute. If we need to collect x-ray spectra for each particle, that can add a few seconds per particle. By the standards of some technologies used for particle analysis (such as flow counters, where the particles are in a fluid passing rapidly in front of a sensor) this is a relatively slow analysis. But given the quantity and quality of information obtained (not just the number of particles, but accurate assessment of dimensions and composition) electron beam particle analysis is a marvelous technology that gives answers that others can’t.

DATA REPORTING

At the end of the complete specimen analysis, the results of the analysis are available as a large table containing the spatial coordinates, and size, shape, and composition parameters for every particle analyzed. In a typical run, this might range from a few hundred to many thousand particles. Accompanying each particle is a little “thumbprint” image so we can see exactly what it looked like. And if we need to, we can

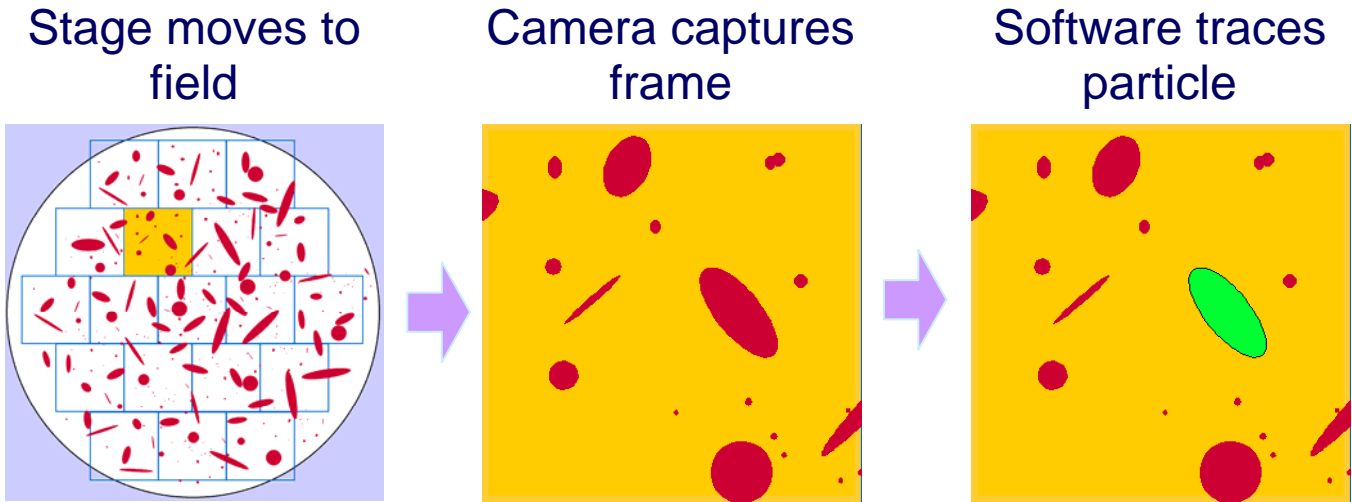


Figure 7: Frame-based Particle Analysis With A Camera

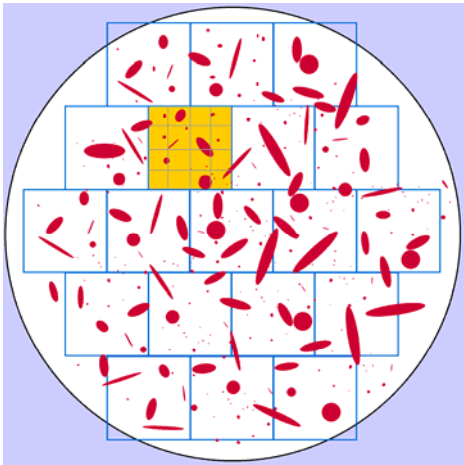


Figure 8: Mag Fields in a Stage Field

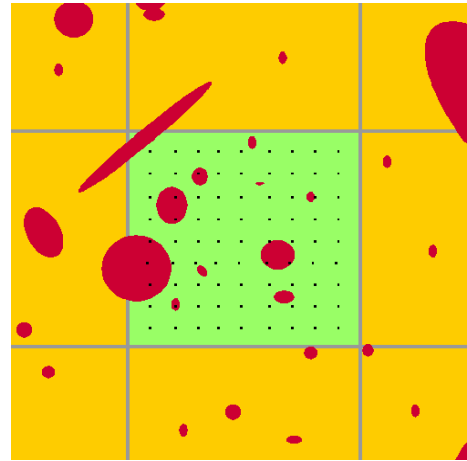


Figure 9: Sampling Point Grid

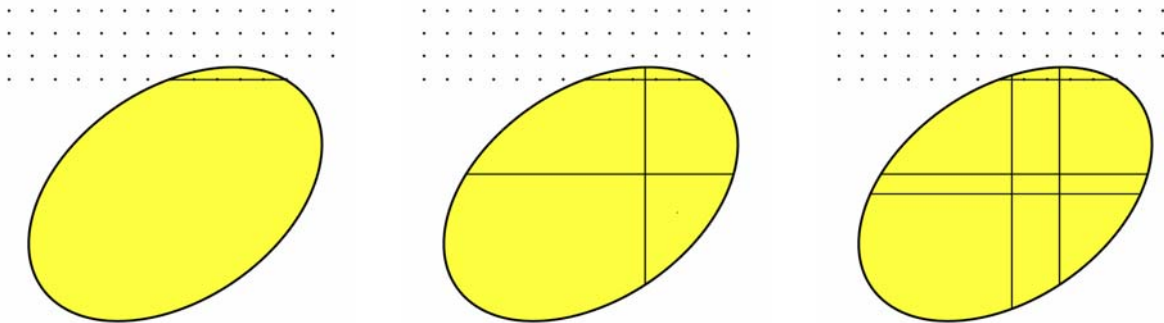


Figure 10: Locating the Center of a Particle

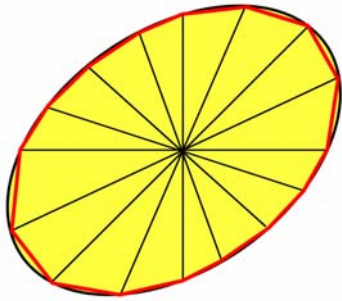


Figure 11: Rotating Chords

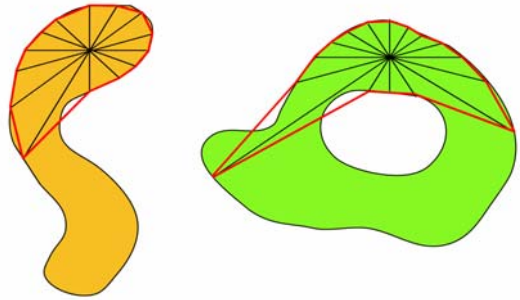


Figure 12: Failure of Rotating Chord Method

tell the system to relocate any particle on the specimen – then a human operator can analyze it in even greater detail. That's really important in Gun Shot Residue detection where a forensic scientist must testify to having personally viewed the GSR particles.

The last step is to generate the final report. Software tools are provided that allow customized reports to be readily configured to suit the needs of individual customers. Some customers want a lot of detail in their reports; other applications might only want a "Go/NoGo" indication.

There is actually one further step possible – the results of all the analyses may be exported to a *database*. This allows the user to efficiently monitor long-term trends for purposes of improving the product or process.

CONCLUSION

So that's the "big picture." It should be apparent that ASPEX systems utilize relatively conventional mechanical and electronic system components functionally similar to those found in conventional SEM/EDX instruments. The highly refined software provided with our systems provides a great deal of our unique value, but the ultimate value comes from the way all components of the system are designed to work together in a highly efficient manner for automated applications.