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A SYSTEM APPROACH TO AUTOMATED SCANNING ELECTRON MICROSCOPY

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by Timothy J. Drake and Frederick H. Schamber

A System Approach to Automated Scanning Electron Microscopy

Much of the innovative activity in scanning electron microscopes (SEMs) has been devoted to ultrahigh-magnification instruments for the use of researchers characterizing materials and processes at nanometer scales, but there is also a growing interest in robust industrial tools operating at more modest magnifications. Industrial applications often require throughput rates that would be impractical for traditional manually operated instruments; in an ever-evolving workplace, companies are also increasingly relying on engineers and technicians to collect the needed data in close proximity to the process being monitored, rather than the services of professional microscopists working from a central laboratory. The need for large quantities of quality data as quickly and consistently as possible is driving new expectations for reliability and automation—an emphasis that is encouraging an integrated solution-driven approach to SEM technology. This new approach represents a different way of thinking about SEM performance, and has fostered a "system" approach to SEM design.

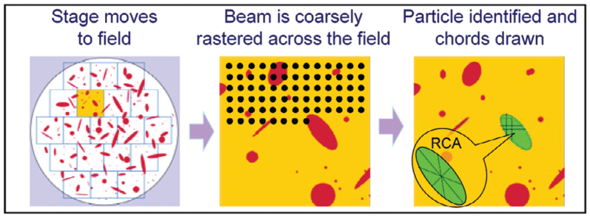

Figure 1 Dynamic beam control for scanning samples. Minimal time is spent on "empty" pixels by dynamically analyzing the specimen.

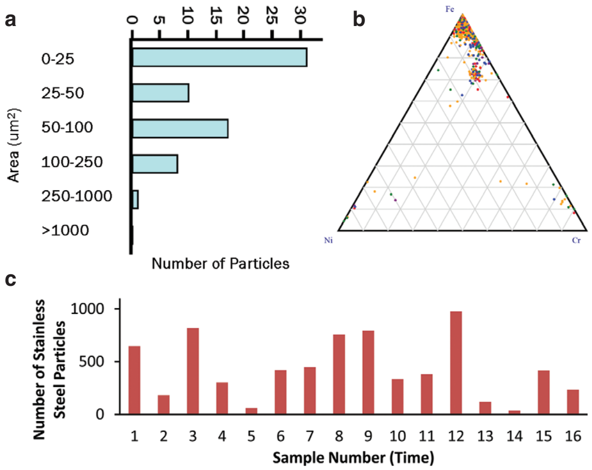

Figure 2 Automated reporting output for wear debris analysis. Size distribution histogram can be vital to understanding the type of wear mechanisms that can occur in a system (a). A ternary phase diagram of steel particles illustrates the difference between different alloys of steel (b). Finally, the automated trending capabilities provide investigators with the ability to easily monitor changes in the process through control charts (c).

Technologies and techniques

A system approach to automated microscopy implies that all aspects of the instrument are optimized for the efficient performance of the specific task(s) to be addressed. Whereas instruments designed for research applications are typically characterized by technical performance specifications, such as attainable resolution, a solution-driven tool is most meaningfully characterized in terms of its ability to assess key specimen metrics with the necessary speed and precision. In order to optimize this, all components of the system (including both hardware and software) must function together as an integrated whole.

Use of dynamic beam control to optimize data collection

A key technology for efficient automation is dynamic beam control. The basic idea is to spend as little time as possible collecting pixels that have no relevancy to the problem. To accomplish this, a "smart" beam control algorithm is employed (*Figure 1*) wherein the beam is rapidly stepped across the specimen using a relatively coarse raster that will pick up the smallest features (e.g., particles) of interest. When an interesting feature is detected, a sizing algorithm is initiated with a much higher resolution and an X-ray spectrum may be collected. Thus, features of interest can be characterized to arbitrarily high precision while minimizing the time spent in collecting uninteresting pixels. This is in contrast to the more conventional frame-based type of computerized analysis, where a complete frame of pixels is first measured at the finest resolution required, and is then analyzed by image-processing software. Since a SEM is a sequential-pixel device, and the vast majority of pixels in a typical image frame are not of analytical interest, dynamic beam control results in dramatic improvements in measurement speed.

Data→information→knowledge

Rapid collection of data is important, but perhaps more important is turning those data into application-relevant information and knowledge. An automated instrument can easily produce a deluge of data that would be overwhelming without the aid of appropriate report-generating software that consolidates the measurements into readily interpreted metrics and trends. Ideally, such reporting software is further structured with knowledge of the particular kind of application so that the reports express the metrics most important to the industry or application being served. If we take a wear debris application as an example (*Figure 2*), material wear is fundamental to understanding how a system performs over time. Consequently, the knowledge gained from the data is not in the individual data points but in the distribution of particle sizes and shapes and the pattern of change over time.

Addressing the X-ray bottleneck

Due to steady improvements in refining the dynamic beam technique, it can now be demonstrated that a random mixed field of circular features can be identified

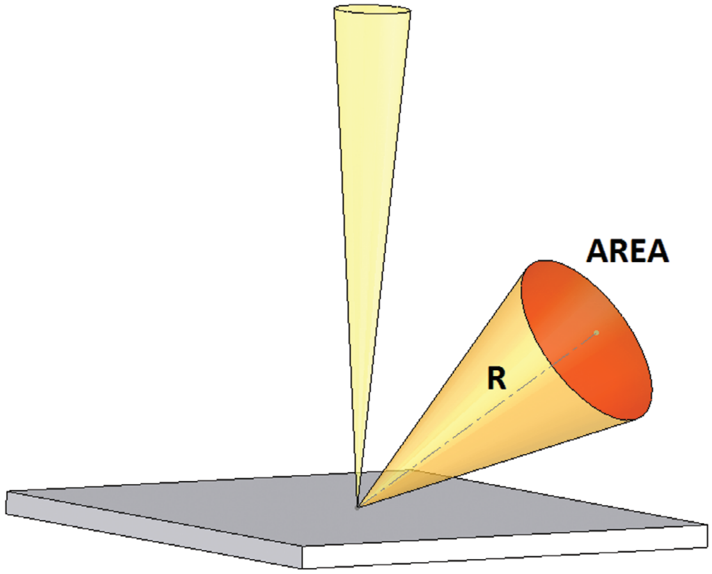

Figure 3 Maximizing omega to improve collection efficiencies of X-ray detectors. If the detector is represented by a circular surface area, located a distance R from the point where the beam impacts the specimen, then the solid angle (omega) is expressed as the area divided by the square of the distance (R). Ω = area/R^2.

and measured at a rate of 600/min with a size precision of better than 50 nm. Impressive though this may be, until recently the hard reality was that, when it was necessary to assess elemental composition, the collection of an X-ray spectrum required a few seconds per particle, which thus becomes the critical limitation for analysis throughput in most practical applications.

A technology breakthrough in the measurement of X-rays first surfaced approximately 10 years ago as the new technology of silicon drift detectors (SDDs) began to replace the venerable lithium-drifted silicon detector. Not only did the new SDD technology eliminate the requirement for liquid nitrogen cooling, but excellent energy resolution could be sustained at much higher counting rates. However, to translate this capability into higher throughput, it is necessary to deliver more X-rays to the detector. To some degree, this can be addressed by using higher beam currents, but since this also means larger beam diameter, spatial resolution is eventually compromised. Thus, increased emphasis is now directed toward increasing the collection efficiency of the detector itself. Two viable strategies are the use of larger detectors and multiple detectors, and though these are indeed effective approaches, they also have a substantial cost impact (as well as other practical limitations).

Integrating the X-ray detector for optimal performance

The most cost-effective means of improving detection efficiency is to place the X-ray sensor as close as possible to the point where the X-rays originate from the specimen (*Figure* 3). In practice, however, such optimization has traditionally been circumscribed by the conventional construction of the X-ray detector, in which the active sensor element is located at the end of a long tube that it is inserted through a port in the microscope chamber. Though this modular tube mount configuration has served the industry well for decades, it has also restricted how closely and flexibly the sensor element can be positioned relative to the specimen. This limitation has recently been addressed by the introduction of the OmegaMax™ technology (patents pending, **ASPEX**, Delmont, PA) in which the SDD sensor is built directly into the structure of the microscope. This has permitted the effective solid angle (omega) of the detector to be increased by several multiples over what had been achieved with conventional tube-mount detectors. In fact, the improvement in detection efficiency is so dramatic that smaller-area sensors can be employed that still deliver substantially enhanced detection efficiency, but with the additional benefit of improved energy resolution, and all without an increase in cost to the user. This dramatic advance was made possible by considering the X-ray detector not as a separate entity, but as an integrated component of a system whose objective is to provide practical industrial solutions.

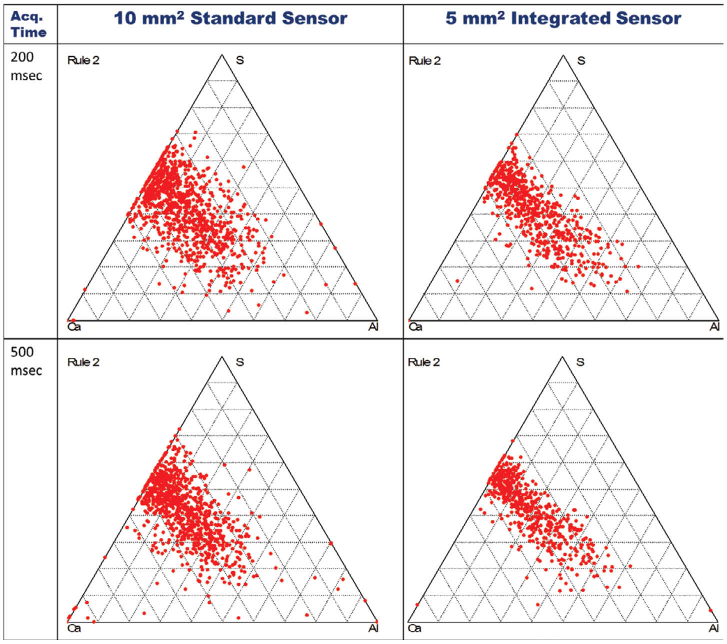

Figure 4 Comparative study of the difference between an integrated and traditional energy dispersive X-ray spectrometer. Ternary phase diagrams are used to evaluate the effective noise in the analysis whereby the scatter of the distribution is proportional to the effective noise. The results indicate that by integrating the detector directly into the SEM, one can achieve greatly improved statistical results.

Case in point: steel industry

Of course, because it is a solution-driven approach, the real proof is how well the technology works to solve actual problems. In the steel industry, for example, a major application is to characterize large populations of inclusions (small impurities) and relate their chemical makeup to process changes. Since unfavorable inclusion types can result in millions of dollars in loss of production in some cases, controlling their occurrence has a direct positive effect on profitability.

The challenge for SEM analysis is a fairly difficult one because the inclusions are small, light-element bodies in a steel matrix. Consequently, getting enough X-ray counts for good compositional identification is a key part of the challenge. In *Figure 4a*, a 10-mm^2 SDD of conventional design was used to produce ternary diagrams. As the acquisition time is increased from two-tenths of a second to one-half of a second, the clustering of the distribution becomes tighter, reflecting an improvement in measurement statistics. In Figure 4*b*, the same measurements were performed with the 5-mm^2 integrated OmegaMax detector (all other parameters held constant). It is easy to see that the clustering at 0.2-sec acquisition time is at least as good as that of the conventional detector at 1 sec. This factor of five represents a major benefit in attainable throughput and/or precision.

Conclusion

SEM evolution over the past two decades has produced robust automated tools that support industrial applications. Innovations in this field are driven by a solution-driven approach in which the SEM is designed as an integrated system with application-specific reporting capabilities. For the steel industry, this integrated systems approach has proven to reduce downtime; increase productivity; improve quality; and, ultimately, have a positive impact on profitability. Other industries, including automotive, aeronautics, and pharmaceutical, can expect to reap similar substantial benefits from this "different kind of microscopy" as it continues to evolve.

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