

# Electron Microscopy: An Integral Tool in Mechanical Testing

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**Improved performance, ease of use, and affordable price has made SEM a well-accepted tool in mechanical testing and failure analysis.**

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For decades, microscopy has been one of the failure analysts' most powerful tools in determining the causes of failure in a wide variety of materials. As manmade materials have evolved from the macroscale to the micro, nano and even picoscale, the need for higher resolution microscopy techniques have been required. Scanning electron microscopes (SEMs) are one of the most powerful, versatile tools for scientists due to their large depth of field (compared to light microscopes), high spatial resolution (high magnification) and analytical capabilities (energy dispersive x-ray, or EDS and wavelength dispersive spectroscopy, or WDS) for chemical analysis. Another part of their flexibility is the ability to add a variety of electrical, mechanical, and chemical test equipment to them making the microscope a self contained "micro laboratory."

Transmission electron microscopes extend magnification into the micron range allowing atomic resolution imaging and the ability to obtain elemental analysis and crystallographic information at the same time. In many cases, determining the cause of a failure is as simple as looking at a single image. However, as discussed below, a wide variety of techniques and microscope accessories may be required to determine the cause. Figures 1a and 1b show a failed integrated circuit due to ESD (electrostatic discharge). Figure 1c shows a large depth of field, higher magnification of brittle fracture mechanics.

## Specimen Size Considerations

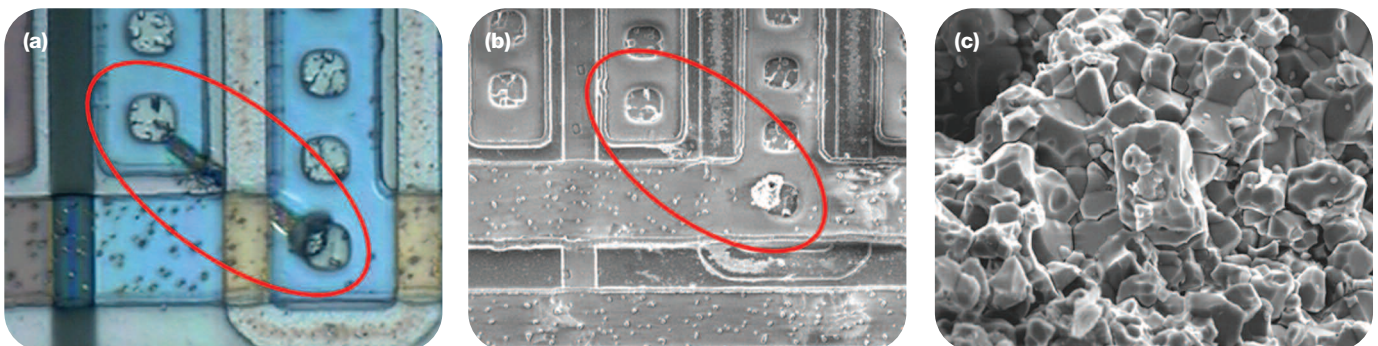
When performing failure analysis or forensic evaluation of a specimen using an SEM, it is important to consider whether the SEM analy-

sis can be performed in a nondestructive fashion, thus allowing for other testing to be performed on the specimen later on. The solution in SEM would be to perform the analysis in a large-chamber, low-vacuum SEM, so the sample can be imaged without cutting it to size and without applying a sputtered coating for conductivity, steps traditionally required for SEM imaging. The ability to image the entire specimen without the need to cut it down in size to fit in the microscope chamber also eliminates any potential for losing important components or adding artifacts during the sample preparation process. Both the size of the specimen chamber and the stage travel are important. The specimen chamber of a JEOL JSM-6610LV SEM allows observation of samples up to 300 mm in diameter by 90 mm tall (~12 by 3.5 in.). An entire turbine blade is easily accommodated inside this SEM (Fig. 2).

The SEM specimen chamber size, as well as the overall position and number of available accessory ports, also dictate what additional detectors, substages, manipulators, and other components can be fit onto the SEM. Accommodating multiple detectors and analyzers makes the SEM a multipurpose instrument that can perform a variety of analyses on the same specimen in a nondestructive manner.

## Tensile Testing and Fatigue Analysis

Tensile/compression testing and fatigue analysis play an important role in understanding the mechanical properties of materials. Newer SEMs allow much higher resolution at lower accelerating voltages. The advantage to this imaging regime is that at lower voltage the image is more sensitive to surface topography



**Fig. 1** — Optical (a) and SEM (b) image of delayed device following an ESD event; (c) intergranular brittle fracture (phosphor bronze).

# and Failure Analysis

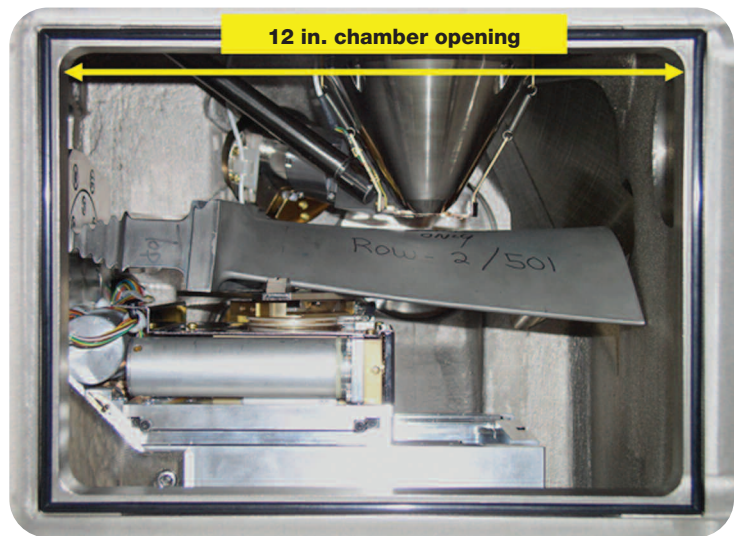
(less electron beam penetration). This is crucial when observing fatigue striations, which can be extremely fine structures on the surface of a fracture face. This is especially critical in lighter metals such as aluminum alloys. The difference in image quality between high and low kV imaging of fatigue in aluminum is shown in Fig. 3. Lower accelerating voltages also facilitate easier imaging if the surface is oxidized, as the oxide is nonconductive and will charge at higher voltages, making imaging more difficult.

In-situ computer-controlled dynamic testing experiments can also be performed in the SEM chamber, providing a better understanding of the cause of the deformation while examining the microstructure in the region of interest where failure is propagating. It can also be used to simulate hypothesized conditions of a failure. These tests can be performed in real time to record quantitative load/extension characteristics. Tensile experiments inside the SEM can eliminate uncertainties in interpreting traditional stress/strain data performed ex-situ and can track the deformation as it happens as opposed to a traditional post-failure examination.

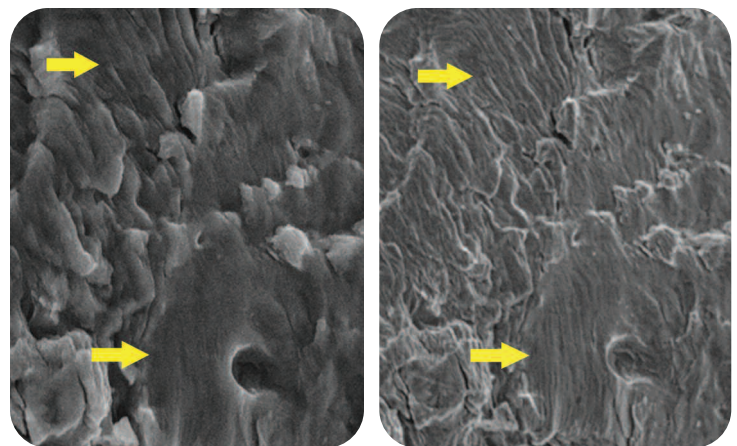
## Structural Analysis

Electron diffraction is the technique of choice when both microstructural (diffraction) and morphological (imaging) data are needed from a specific area of the specimen. In the case of SEM, the sample preparation technique is key for obtaining optimal results. While mechanical preparation procedures are most widely used for large area specimens (several mm to a few inches in diameter), ion-beam polishing produces the best specimens for smaller specimens and micro and nanograin structures and interfaces. Ion-beam polishing offers a strain-free, artifact-free (no smearing or particle embedding) specimen preparation technique. Figure 5 shows a comparison between a mechanically cleaved solar thin-film specimen vs. a sample prepared using a JEOL cross-section polisher (CP). The CP-prepared sample shows channeling (grain orientation) contrast and grain structure in various film layers, making this an ideal specimen for EBSD (electron backscattered diffraction) analysis. EBSD analysis can help determine whether the specimen exhibits a preferred orientation, the dominant grain boundary orientations, strain mapping, texture, grain size distribution, and a variety of other crystallographic characteristics, all of which can help understand the mechanical behavior of the material. It can also provide phase identification (austenite or ferrite, for example) and can be combined with EDS for combined crystallographic and chemical mapping.

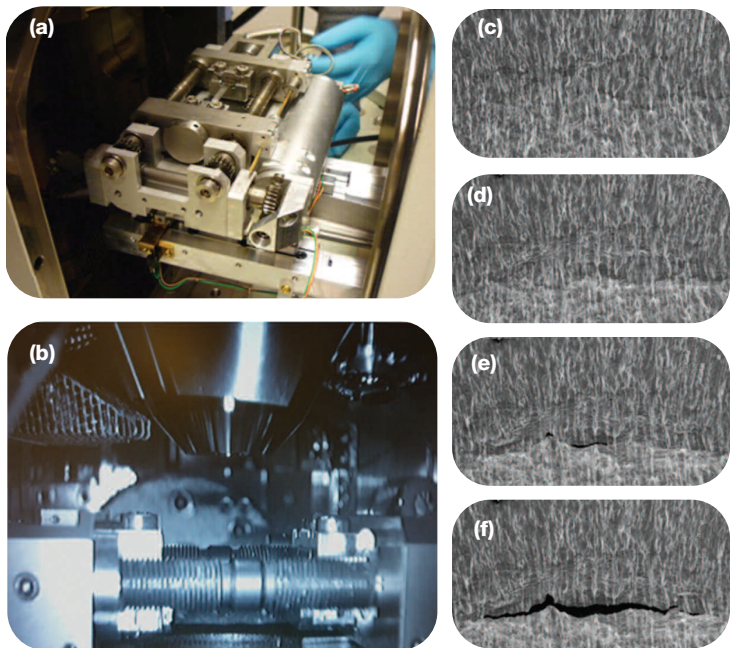
Using a combination of bright-field and dark-field imaging in a transmission electron microscope (TEM) can



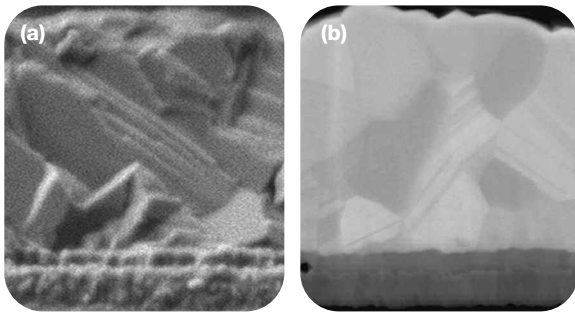
**Fig. 2** — Section of a turbine blade inside a JEOL JSM-6610LV model SEM specimen chamber.



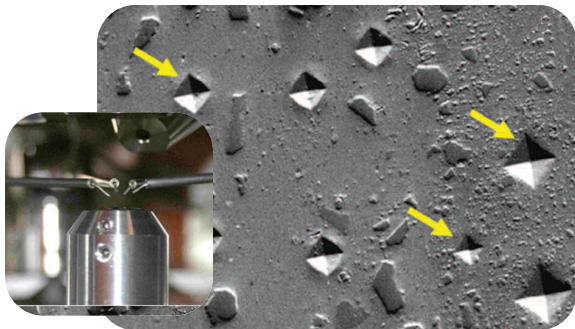
**Fig. 3** — Comparison of high kV (left) vs. low kV (right) imaging of fatigue striations in aluminum under low load, high cycle conditions.



**Fig. 4** — Custom modified Kammrath & Weiss tensile stage fitted onto JEOL JSM-7001F high-resolution field emission SEM (a and b) and series of images from a ferrite steel sample during the tensile test showing crack propagation (c-f). Courtesy of Mr. Pesci, ENSAM, Metz.



**Fig. 5** — Solar thin-film cross section; (a) cleaved and (b) polished using JEOL CP.



**Fig. 6** — Nano-manipulators inside JIB-4500 SEM/FIB chamber at Boston College (inset) and example of in situ hardness testing indents (arrows) imaged using SEM.

further elucidate the microstructure. TEM images can show dislocation density, stacking faults, precipitate structure and location, and structure of individual grain boundaries.

### Nano-manipulation and Hardness Testing

Hardness testing by microindentation can be performed either in-situ or ex-situ, and the results can be examined using SEM. Nano-manipulators can also be used to perform mechanical testing of nanostructures in the SEM. Figure 6a shows an example of four nano-manipulators installed on a JIB-4500 FIB/SEM at Boston College. Figure 6b shows an example of microhardness indentations imaged in SEM.

### Conclusions

Scanning electron microscopy (SEM) is an indispensable tool in the area of failure-analysis. New developments in technology in both microscopy and spectroscopy have pushed the limits of what can be analyzed beyond boundaries that existed just a few years ago. There has also been a dramatic improvement in automation, eliminating the need for a dedicated microscopist. Increasing performance, improving ease of use, and keeping the cost at an affordable level has made SEM a widely available and well-accepted tool in this field and across a wide variety of other disciplines. □

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