An EBSD system added to an SEM is a valuable new tool in the materials characterization arsenal.

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Electron backscattered diffraction (EBSD) is the technique by which an SEM can be used to evaluate the microstructure of a sample based on crystallographic analysis. It is a quantitative technique that reveals grain size, grain boundary character, grain orientation, texture, and phase identity. The technique enables analysis of centimeter-sized samples with millimeter-sized grains, as well as thin films with nanograins. The nominal angular resolution is ~0.5 degree, and spatial resolution is related to the resolution of the SEM, but 10 nm grains can be distinguished in modern field emission SEM’s. Sample size depends on the SEM sample-handling capability and other geometrical considerations, as well as the time available.

EBSD as it exists today was developed during the 1980’s at Bristol University by David Dingley, and in the early 1990’s by Stuart Wright and colleagues at Yale University. Concurrently and independently, Niels Schmidt in Denmark developed a general solution to the computer-aided indexing problem that was the first to accurately index all seven-crystal systems.

This article describes the operation of EBSD and then shows how it can characterize the microstructure in an aluminum weld and map an area of multiple phases.

Operation of EBSD
EBSD operates by arranging a flat, highly polished sample or an as-deposited thin film at a shallow angle (usually 20 degrees) to the incident electron beam. The SEM stage tilts the plane of the sample to this shallow angle (Fig. 1). With moderate to high electron beam acceleration voltages (10 to 30 kV) and incident beam currents of ~1 nano-ampere, the electron beam is diffracted by the crystal lattice of the sample at the incident beam point on the sample surface (Fig. 2). With the beam stationary, an electron backscatter diffraction pattern (EBSP) emanates spherically from this point in all directions.

If an EBSP detector (Fig. 3) is placed close to the incident beam point, it intersects a portion of this diffraction pattern. The detector is in fact a digital camera similar to that found in a consumer digital camera but contained within a vacuum compatible environment.
and retractable body. The camera’s CCD chip is illuminated by a phosphor screen that intersects the spherical diffraction pattern. The phosphor converts the diffracted electrons into light suitable for the CCD camera to record.

With a stationary beam on a point on the sample, the EBSP (Fig. 4) is analyzed and in some cases stored. The EBSP is uniquely defined by the lattice parameters of the particular crystal under the beam, and by its orientation in space.

By selecting the expected crystal phases from a phase database, all possible identities and orientations of the crystal under the beam are matched to the EBSP until the best fit is found. The pattern is then considered indexed, and the best-fit orientation and phase is reported as the orientation and phase of the point on the sample.

As the speed of pattern analysis has increased, it has become practical to scan the beam over multiple points on the sample to create an orientation map (OM). This is now the most common method for a microstructural investigation with EBSD. A map is defined by its location and size on the sample surface, and by the sampling step size between points. In this way, the resolution of the map may be adjusted to reveal the grain structure and grain boundary character, depending on the electron beam resolution under the sampling conditions, time available, and size of the sample area required. Speed has risen over the years, from manual indexing in the 1980’s to 100 indexed patterns/second today and has approximated an exponential increase.

**Interfacing to the SEM**

The EBSP detector attaches to a free port on the SEM chamber. Ideally, the port should be orthogonal to the stage tilt axis, so that the sample may be easily tilted toward the detector at ~70 degrees, although other orientations are possible. Typically, the port should allow the detector to have a nominal working distance of about 20 mm, since a highly tilted sample necessitates moderate working distances. Special detectors are available for less-favorable port positions.

Most SEM’s are equipped with EDX spectrometers for chemical analysis by characteristic X-ray production under the incident electron beam. Today, EDX systems take control of the beam location on the sample via the external scan interface on most SEM’s. EBSD requires the same interface to the SEM and thus, for most retrofits to existing...
systems, a simple electronic method to share this external interface is required. An intelligent switch box is placed between the EDX and the SEM, and this arbitrates between the EDX and EBSD systems' access to the SEM (Fig. 5).

In addition to beam control, integrated stage motion is required for large sample area coverage. SEM motorized stages are often accessible via an RS232 serial computer interface or Ethernet connection, which can be addressed by the operating software.

**Microstructural characterization**

Figures 6 and 7 show maps typical of the EBSD technique. Here, a friction stir weld in an aluminum alloy (AA2024) is examined in cross-section. Figure 6 shows an Orientation Map in which each grain is color-coded by its orientation. When analyzed quantitatively, a strong brass texture is found.

With the orientation of each pixel in the map known, grain boundaries can be defined by the critical misorientation angle between neighboring pixels. This differs from a light microscope equipped with a traditional image analysis system, in which grain boundaries are identified visually by a brightness difference alone.

As shown in Fig. 6 and 7, the grain boundaries are defined by a critical misorientation angle of ~7.5 degrees. On the left side of the map, the fully recrystallized “nugget” region is shown with small, relatively equiaxed grains having a mean grain size of 4.9 µm. On the right side, the thermally affected zone shows larger, elongated grains of mean grain size 13.5 µm. These grains also have a high number of low-angle boundaries, which are illustrated in the deformation map (Fig. 7).

Figure 8 shows how the residual strain state can be deduced from EBSD data. Here, rolling and heat treatment of an Fe-Al binary alloy resulted in a microstructure that is partly deformed (red) and partly recrystallized (blue). Again, texture information (Fig. 9) can be extracted from the two populations.

**Phase discrimination & identification**

EBSD can be set up to map a sample containing multiple phases and discriminate between each. For example, EBSD can discriminate between α (hexagonal) and β (BCC) titanium phases, based purely on the structural differences (Fig. 10).

In cases where a phase is unknown and an EDX is available, it can be identified using a phase database such as those available from National Institute of Standards and Technology (NIST), or the International Centre for Diffraction Data (ICDD). In this scenario, chemical information from the EDX is used to produce a subset of candidate phases from the phase database. EBSP’s are then indexed relative to all the candidate phases and the unknown phase is identified as that which best fits the experimental data.

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