HIGH-SPEED EBSD

Quantitative information on the crystallographic orientation of the constituent grains in a polycrystalline microstructure is best characterized via electron backscatter diffraction.

Stuart Wright and Matt Nowell**
EDAX-TSL
Draper, Utah

An electron backscatter diffraction (EBSD) pattern is formed when a focused electron beam is positioned on a grain within a highly inclined (typically 70 degrees) sample in a scanning electron microscope. As the incident electron beam interacts with the crystal lattice within a small volume, electrons are coherently diffracted out of the sample. If a phosphor screen is placed near the sample, then the pattern formed by the diffracted electrons can be imaged with a digital CCD camera (Fig. 1).

The pattern is composed of a geometrical arrangement of bands that represent planes in the crystal. The imaged pattern can be transmitted to the computer and the pattern automatically indexed to show the corresponding orientation of the crystal lattice. Fully automated orientation measurements with angular resolutions better than half a degree can be achieved with modern EBSD systems. These systems control the position of the electron beam over the sample surface so that orientations can be measured over a regular grid. The grid data then becomes the basis for generating an orientation-based color image (Fig. 2). This technique is sometimes called orientation mapping or orientation imaging microscopy.

Automated EBSD has proven to be a valuable tool for characterizing crystalline materials. This is evidenced by the large number of papers published where the technique has been applied (over 5000 since inception of the automated technique in 1992).

This article discusses the operation of high-speed EBSD instruments, including angular resolution, indexing, and operating conditions, then shows how the technology can be applied for high-spatial-resolution scans, texture analysis, and grain size measurements.

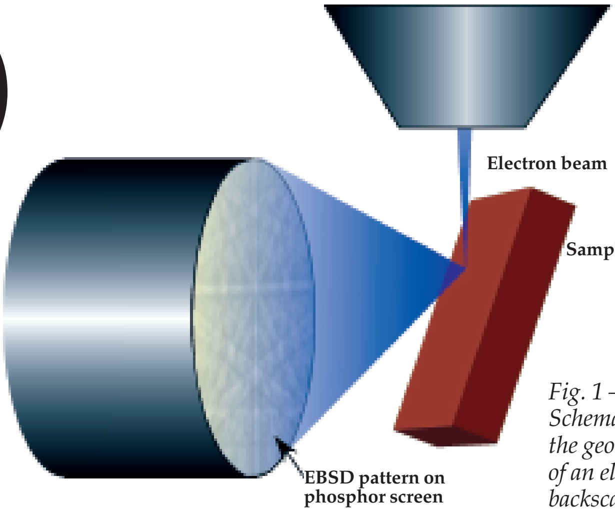

Fig. 1 — Schematic of the geometry of an electron backscatter diffraction system shows the electron beam striking the sample at an angle. The diffracted beam activates the phosphors on the screen, generating an image.

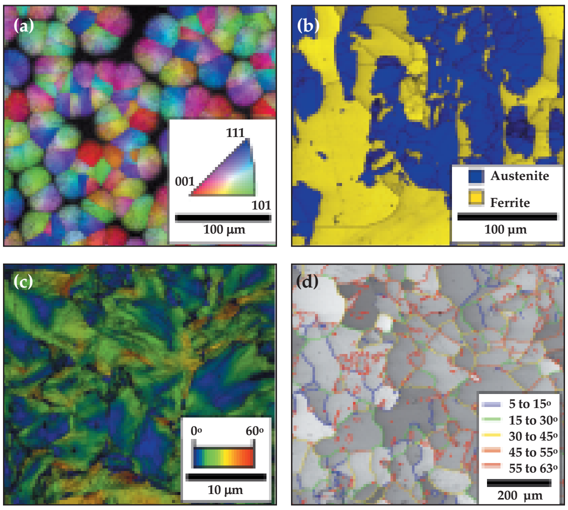

Fig 2 — Maps reconstructed from EBSD data: (a) a map colored according to the crystal direction parallel to the normal direction of a Co-Sn-Te thin film sample; (b) phase map of a duplex steel; (c) a map showing the orientation variation within individual grains in a deformed steel rod; and (d) a map with grain boundaries colored according to misorientation in titanium. All color maps are overlaid on a gray scale map based on the EBSD pattern quality; lighter shades represent high contrast patterns and darker shades correspond to weak patterns. The EBSD data in (c) and (d) has been post-processed; less than 1.5% of the data points were modified.

High-speed EBSD

The acquisition speed of today's automated EBSD systems is more than a thousand times faster than the original automated systems. This is due primarily to improvements in digital camera technology as well as the increased processing power of modern computers. However, various "shortcuts" must be taken to achieve these speeds. For example, higher speeds are possible with lower-resolution patterns, i.e. 80x80 pixels. This is done through binning of the CCD camera, which improves the effective light sensitivity of the camera, leading to shorter exposure times. It also improves the throughput because fewer pixels are transmitted through the processing

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pipeline. On the other hand, fewer pixels can potentially lead to more ambiguities in the indexing and a loss of angular resolution.

Angular resolution

The effect of pattern pixel resolution on angular accuracy can be measured in several different ways. One approach is to run scans on a single crystal of known orientation at different pixel resolutions. The spread in orientation for all of the measurements in the scan is then calculated. Figure 3 shows results for the spread measured on a silicon single crystal at different binning sizes.

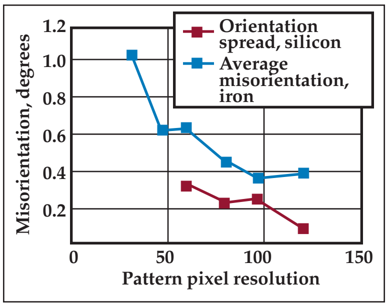

Fig. 3 — This graph shows the angular resolution as a function of binning size (smaller bins correspond to higher speeds). The angular resolution is measured using two different approaches: a single crystal based approach in silicon and a direct comparison approach in a steel polycrystal.

Another approach is to simulate the binning by running a scan over a polycrystal and then saving the patterns to disk. The pattern can then be binned using software, and the scan re-run using the binned patterns. The orientation measured at different amounts of binning can then be compared one-to-one. This approach prevents any effects due to carbon contamination or drift, rather than re-running the scan over the same area at different binning sizes. Such an approach has been taken on a ferritic steel sample (Fig. 3). Results show only a slight loss in the angular resolution of the measurements.

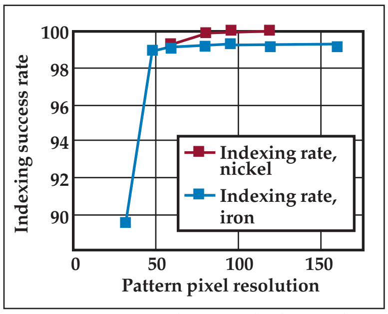

Fig. 4 — This graph shows the indexing success rate as a function of binning size using different approaches: Running scans over different areas at different binning sizes on a nickel super alloy sample; and collecting patterns at high resolution on a steel sample and then re-running the scan by binning the patterns in software.

Indexing success rates

Indexing success rates can be calculated by a variety of methods. For example, a simplistic approach would be to define success rate as the number of patterns that can be indexed divided by the total number of points in the scan. However, this makes no concession to the fact that some patterns may be indexed incorrectly.

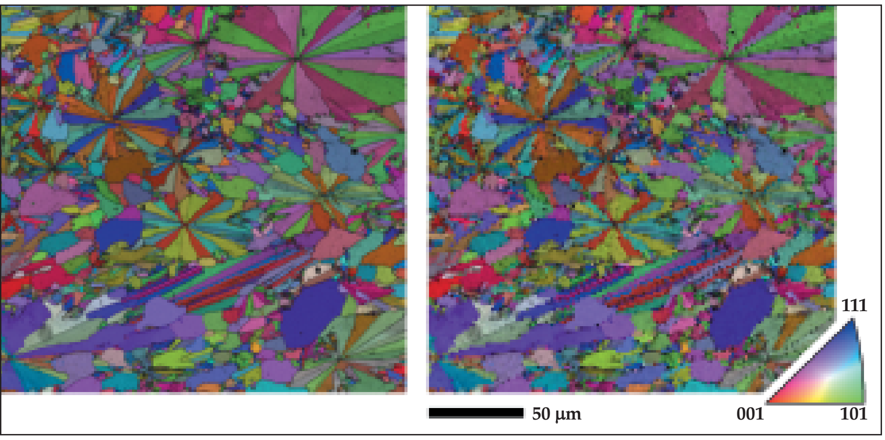

Fig. 5 — These two scans are from a polycrystalline silicon sample. The scan on the left is at high resolution, and the one on the right has a step size four times larger.

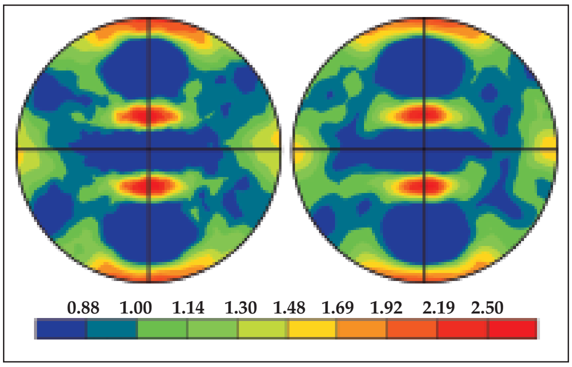

Fig. 6 — A comparison of textures measured using conventional X-Ray diffraction (left) and EBSD (right) in the form of (110) pole figures on cold-rolled steel sheet.

A more rigorous approach is to consider only those measurements with confidence index (CI) values greater than 0.1 to be successfully indexed. (A value of 0 indicates a potentially ambiguous solution.) The CI data is processed to some level: if a measurement has a low CI value but has the same orientation as its neighbors, then its CI value is upgraded to that of its neighbors. Figure 4 shows indexing success rates for different scans over different areas with different binning sizes for a nickel superalloy sample, and then based on software binning of patterns from a single scan on the same ferritic steel sample described previously. The binning has relatively little impact on the resulting indexing success rates until extreme levels of binning are reached.

Operating conditions

Advances in EBSD detector technology facilitate high speed data collection without a corresponding significant increase in electron beam probe current. This is important because typically as beam current is increased, the probe size and corresponding spatial resolution decrease. By operating at lower beam currents, spatial resolution performance is improved. For this work, probe currents ranged from 3 nA to 5 nA at an acceleration voltage of 20 kV.

Sample preparation is also a significant factor in EBSD performance. Often a mechanical polishing routine with specific emphasis on the final polishing stages can produce good EBSD results.

Range of applications

EBSD scans that formerly required hours can now be done in a matter of minutes. Thus, high speed EBSD enables a wider range of applications. Consider the following examples:

- **High spatial-resolution scans:** Although it is possible to scan the same area in less time with a high-speed EBSD system, many operators choose to cover the same area in the same amount of time with a smaller step size between scan points to generate much more detailed spatial information. For example, grain boundaries can be characterized more accurately, local orientation variations due to residual plastic strain can be studied in more detail, and small second phase particles can be characterized as well. Figure 5 shows the advantages of a higher resolution scan on a polysilicon sample. In addition to the additional detail collected, the faster acquisition speeds minimize potential problems from localized charging or drift.

• **Texture analysis**: Preferred orientation or texture can be characterized. The high speed systems enable many grains to be sampled quickly. Studies have shown that in materials with moderate texture, the orientations of approximately 10,000 grains need to be measured to reach statistical reliability equivalent to that of conventional X-ray diffraction (XRD). This can easily be done in just a few minutes via automated EBSD. Figure 6 compares textures measured on rolled steel with XRD and EBSD, showing a high level of agreement between the two methods. The subtle differences are due more to differences in the sampling depth of XRD vs. EBSD (EBSD is a very surface sensitive technique) and the numerical processing of the data than in the sampling statistics. The EBSD data was collected in less than 15 minutes. (A comparable XRD measurement typically requires about an hour.)

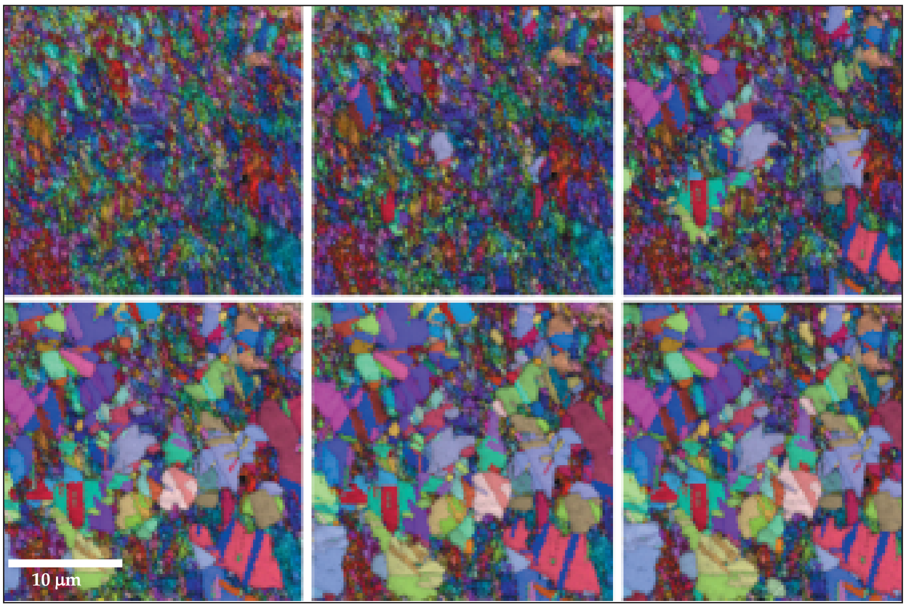

Fig. 7 — Selected individual scans are shown from an in-situ heating experiment on a copper specimen deformed through equal channel angular extrusion.

• **Grain size**: By combining the high spatial resolution of EBSD with larger area analysis, it is possible to directly measure grain size in polycrystalline materials with a high degree of statistical reliability. Grain boundaries are readily found, which enables accurate measurements of grain size in a fully automated fashion. Grains as small as 10 nm can be distinguished. However, it should be noted that such small grains are on the small side of a distribution with a larger average grain size. It is more accurate to say that orientation maps can be derived from materials with average grain sizes of 50 nm on a modern field-emission SEM.

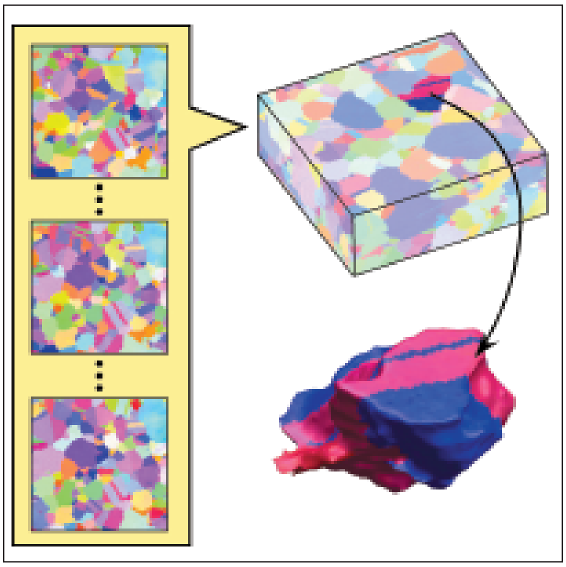

Fig. 8 — This schematic shows processing of individual serial sections into a 3D data cube and a grain reconstructed from the EBSD data.

• **In-situ EBSD**: High-speed EBSD also makes in-situ studies of dynamic processes much more tractable, as it is possible to continuously scan over an area during a heating and/or straining process. The high speed enables collecting more scans during an experiment, collecting data at higher spatial resolution, or collecting data over a larger area. The in-situ experiments make it possible to directly correlate the evolution of microstructural features such as grain size or local orientation gradients with crystallographic orientation.

Figure 7 shows 6 out of 67 frames collected from an in-situ recrystallization and grain growth experiment for copper. It should be noted that as EBSD is a surface technique, information of the behavior in the bulk must be inferred from observations at the surface.

Nonetheless, such measurements have proven useful for investigating phenomena such as grain boundary migration and twinning effects during recrystallization and grain growth, variant selection during phase transformation, and intergranular cracking during creep.

Three-dimensional EBSD

With the advent of new SEMs integrated with a focused ion beams (FIB), in-situ serial sectioning is possible. If this capability is combined with an EBSD system, then a three dimensional (3D) characterization of the orientation aspects of microstructure is possible. The high speed EBSD enables either high spatial resolution and/or more slices to be measured during such an experiment. Combining the orientation information with 3D spatial information provides a very complete characterization of microstructure. Figure 8 shows orientation maps from 3 out of 55 sections through a nickel sample and the resulting data cube, along with a twinned grain reconstructed from the data. ⬢

For more information: Stuart Wright, EDAX-TSL, 92 East 12300 South, Draper, UT 84020-9540; tel: 801/ 495-2750; stuart.wright@ametek.com; www.edax.com.