

# Electron Backscatter Diffraction



Essential  
Knowledge  
Briefings

Second Edition, 2015



Front cover image courtesy of EDAX. EBSD map from a 75% rolled copper sample. This color-coding indicates the crystal direction parallel to the surface normal direction.

## CONTENTS

- 4** INTRODUCTION
- 6** HISTORY AND BACKGROUND
- 15** IN PRACTICE
- 24** PROBLEMS AND SOLUTIONS
- 28** WHAT'S NEXT?

## INTRODUCTION

Lots of materials - both natural and synthetic - are crystalline, with a regular, ordered structure. There is now a powerful technique for studying these materials, which can reveal the intricacies of their crystalline microstructure and so allow scientists to explore how this microstructure affects their properties and functions.

Known as electron backscatter diffraction (EBSD), this technique has so far mainly been used to study the microstructure of classic crystalline materials such as metals and ceramics, including the latest alloys and superconducting materials. It has also been adopted by industries that either produce metallic materials, such as the steel industry, or rely on the structural integrity of metallic materials and ceramics, such as the nuclear power industry.

Increasingly, though, scientists are beginning to apply EBSD to other examples of crystalline materials, including geological samples, biological shells, fossils, meteorites and even mineral granules produced by earthworms. In some cases, EBSD is one of several techniques used to study these materials, but in other cases it has proved to be the only technique up for the job.

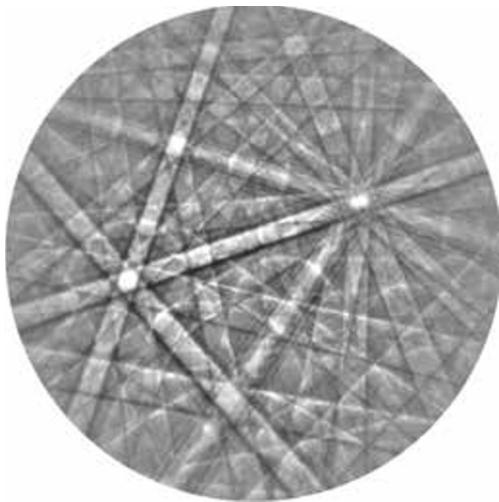
EBSD determines crystal structure by recording the patterns produced on a phosphor screen when a beam of electrons is diffracted by the surface of a crystalline material, with the patterns reflecting the underlying crystal structure. By scanning the electron beam across the surface of the material, modern EBSD systems can quickly produce a wealth of information about the material's crystal microstructure and display this information in a variety of useful ways. What is more, combining EBSD with a related analytical technique such as energy-dispersive spectroscopy can help to expand its analytical capabilities still further.

This Essential Knowledge Briefing provides a general introduction to EBSD. It explains how the technique works, including how the crystal structure is determined from the patterns produced by the diffracted electrons, outlines the kind of information that EBSD can produce, and details how that information can be displayed and utilized. The briefing also outlines various practical issues related to the technique, describes potential problems that may arise and how to solve them, and provides examples of how EBSD is being used by scientists in their research. Finally, it reveals how the technique is poised to develop and advance in instrumentation, integration and applications over the next few years.

## HISTORY AND BACKGROUND

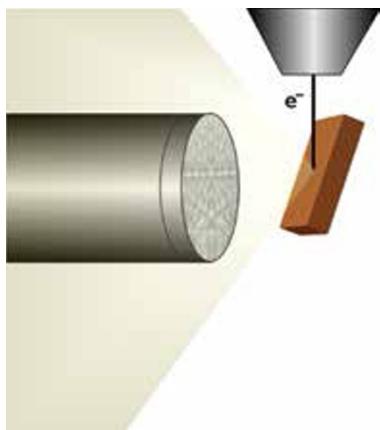
In EBSD, a scanning electron microscope (SEM) fires a tightly-focused beam of electrons at a crystalline material. On encountering the material, some of the electrons in the beam are diffracted away by the material's crystalline structure and hit a phosphor screen placed close by, generating patterns of light that are detected and recorded by a digital camera.

These patterns, known as Kikuchi patterns, take the form of an array of intersecting bands that reflect the crystalline structure of the material, with the bands corresponding to planes in the crystalline material. The intensity and width of the bands are directly related to the type and spacing of atoms in the crystal planes, while the angles between the bands are directly related to the angles between the planes. A great deal of information about the crystalline structure of the material can thus be determined from these patterns.



**Fig 1. Kikuchi pattern from silicon**

For this to happen, however, the individual components of the EBSD system have to be oriented correctly. The crystalline material is placed on a stage at the intersection between the SEM beam and the face of the phosphor screen, which are at an angle of  $90^\circ$  to each other; the beam usually comes from above the material while the phosphor screen and digital camera are placed in line with the material. The crystalline material itself is placed at an angle of around  $70^\circ$  from horizontal, because this helps to ensure the maximum amount of electrons are diffracted towards the phosphor screen.



**Fig 2. Illustration of the geometry inside the SEM showing the electron beam and phosphor screen with a sample at a  $70^\circ$  angle to the horizontal**

The origins of EBSD date back to the 1920s, when Japanese physicist Seishi Kikuchi first spotted the diffraction patterns that now bear his name while firing an electron beam at the mineral calcite, with the diffracted electrons forming the patterns on a

photographic plate. Although various research groups investigated these patterns over the following 50 years, it wasn't until the development of the SEM in the 1970s that scientists thought about using the patterns to probe the structure of crystalline materials.

Even then, EBSD didn't really take off as an analytical technique until the late 1980s. This was when D. J. Dingley at Bristol University, UK, combined SEM with a phosphor screen and a video camera to bring together all the components of the modern EBSD system. A few years later, Stuart Wright and his colleagues at TSL Inc (which was later bought by EDAX) came up with the first software tools for automatically relating the geometric arrangement of the Kukuchi bands to the crystalline structure, a process known as indexing. The earliest commercial EBSD systems incorporating all these features subsequently became available in the early 1990s.

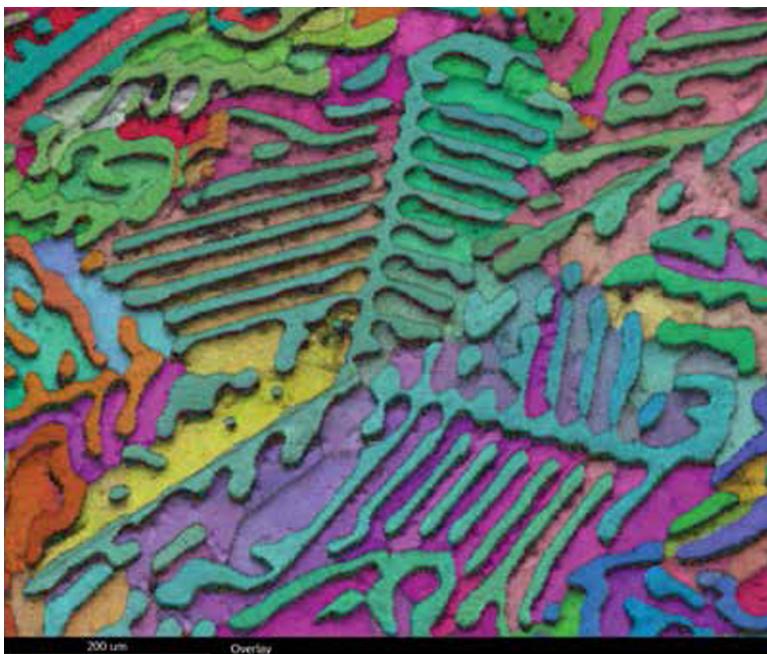
Prior to the development of these automated software tools, the Kikuchi bands had to be identified manually, by an operator locating and drawing lines on the image. This process was both tedious and prone to error as the images of the Kikuchi patterns were not always very clear. Not only did this lack of clarity make the bands tricky to spot by sight, but it also created enormous difficulties for automated image analysis techniques.

The problem was finally resolved by using a technique known as the Hough transform to convert the bands in the image into points in a mathematical construct known as Hough space. Points are generally much easier than bands to locate automatically, and so combining the Hough transform with image analysis techniques allowed the development of software tools for automatically identifying Kikuchi bands. The final indexing step involves

simply determining the angles between the bands to reveal the crystal structure of the original material.

In today's systems, the video camera has been replaced by a digital camera, and the whole system is computer-controlled, with both the SEM beam and the stage on which the material is held able to be moved automatically. Such systems are able to capture images with a resolution as low as 10nm in just a fraction of a second, with the tightly-focused electron beam forming a small, elliptical spot on the surface that defines the region from which electrons are diffracted, known as the interaction volume. To produce an image of the whole material, this spot is scanned over the surface by computer, generating Kikuchi patterns at tens of thousands of points. The distance between these points tends to vary from a few micrometers to just a few tens of nanometers, depending on the desired resolution and scan speed.

In this way, EBSD can build up a picture of the crystal structure across the entire surface of a material. This is especially useful for studying polycrystalline materials, in which the same basic crystal structure occurs at a variety of different orientations, with modern EBSD systems able to distinguish crystal orientations that differ by less than  $0.25^\circ$ . This process is known as orientation imaging microscopy (OIM). EBSD is also useful for studying materials that comprise various different crystal structures, known as phases, with EBSD able to identify structures from all seven main crystal systems. Because the crystal orientation can only be calculated for known phases, the phases present in a material are usually determined first, if they are not already known, before the various orientations are then calculated.



**Fig 3. Orientation map of a aluminum-germanium dual phase material**

Regions of a crystalline material with a specific phase and orientation are known as grains. By determining how the phase and crystal orientation changes over the surface of the sample, EBSD can produce a map of those grains, identifying their size, shape and the boundaries between them.

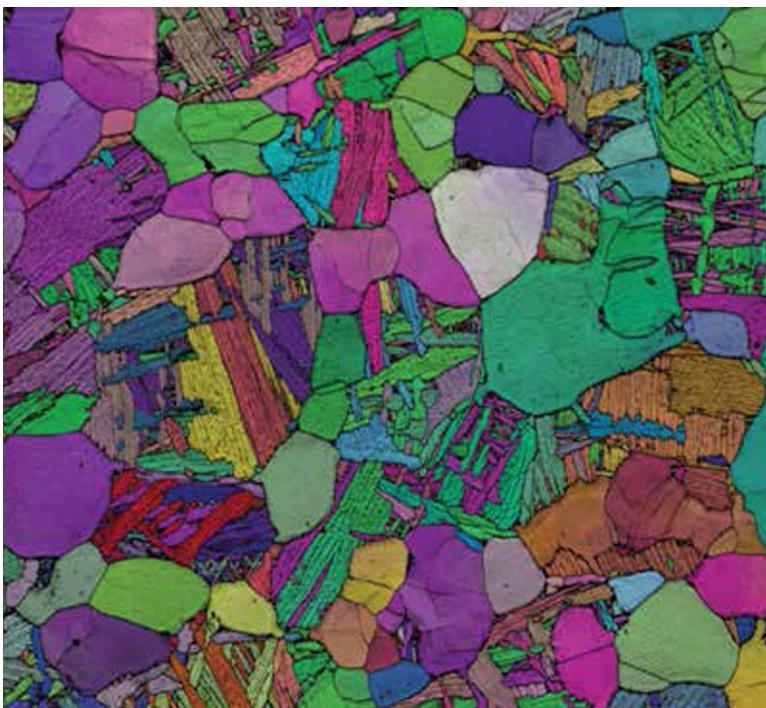
This ability to determine the structure of complex materials comprising lots of crystal grains at different orientations and phases is one of the main advantages EBSD has over other techniques for determining crystal structure, such as X-ray diffraction (XRD) and transmission electron microscopy (TEM). EBSD is faster than either XRD or TEM, while the system is generally less expensive; it also has a higher spatial resolution than XRD, although not

TEM. Furthermore, EBSD requires less sample preparation than TEM, able to analyse bulk materials rather than requiring them to be prepared as thin films.

EBSD can thus be used to investigate a wide range of properties and processes in various different crystalline materials, including metals, ceramics and geologic minerals. This means everything from corrosion in metal structures to mineral crystallization in rock formation to the effect of grain size on high-temperature superconductors. The high speed and resolution of EBSD also allow it to study dynamic processes in materials, such as the effect of elastic strain on crystal structure. Elastic strain can distort the crystal structure and this distortion shows up as slight changes in the Kikuchi patterns.

The only real limitation is that EBSD is restricted to studying the surface of these materials, as the electrons only penetrate a few nanometers before being diffracted, but even this limitation can be overcome by combining EBSD with a focused ion beam. In this set-up, the focused ion beam repeatedly slices off the top layer of the material to reveal the layer underneath, which can then be analyzed by EBSD. These separate EBSD analyses of successive layers through the material can be combined by computer to produce a three-dimensional image of the crystalline structure of the whole material. This is known as three-dimensional EBSD.

EBSD can also be combined with various other analytical techniques to enhance its abilities still further. Perhaps the most commonly used combination joins EBSD with energy-dispersive spectroscopy (EDS), which determines a material's chemical composition by detecting the characteristic X-rays produced by each element in the material when bombarded with high energy



**Fig 4. EBSD orientation map from a titanium alloy exhibiting two distinct microstructures**

electrons. As both techniques involve scanning an electron beam over the surface of a material, they are fairly easy to join together, allowing information about the crystal structure and chemical composition of the material to be obtained at the same time.

As well as simply providing more information about the material being studied, this combination also helps to improve the accuracy of both techniques, for the information on crystal structure can help to determine the chemical composition and vice versa. Different phases in a material may have the same chemical composition but different crystal structures, known as polymorphs; these polymorphs can't be distinguished by EDS, but they can by EBSD.

However, determining the various phases making up a material from the Kikuchi patterns can be fraught with difficulty. It's not a problem if you already know what mixture of phases make up your material, as different known phases can be distinguished fairly easily from their Kikuchi patterns, but it's much more difficult to identify unknown phases in a material from their Kikuchi patterns.

In that case, the phases need to be identified by comparing the generated Kikuchi patterns to those in a database. This process is much easier if you have an idea about the chemical composition of the material, because that helps to narrow down the list of possible phases. If the phases are very different from each other, then you don't need EBSD at all, because the phases can be identified purely from their chemical composition. You only really need EBSD if the phases have similar chemical compositions and so need to be distinguished by their crystalline structure.

Even if the phases in a material can be determined purely from their chemical composition, there is often still benefit from combining the two techniques, as EBSD can provide information that EDS can't, such as the crystal orientation of the various grains making up the material. Nevertheless, the two techniques are not always directly comparable, with the available spacial resolution being the main difference between them; whereas the resolution of EBSD can be as low as 10nm, with EDS it's only 1μm at best when studying bulk materials.

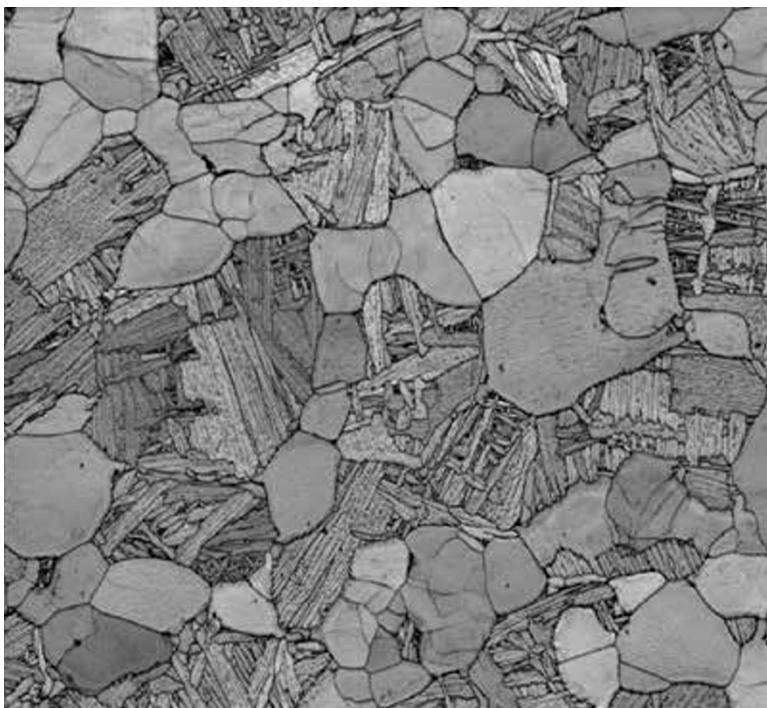


Fig 5. EBSD image quality map from a titanium alloy exhibiting two distinct micro-structures

## IN PRACTICE

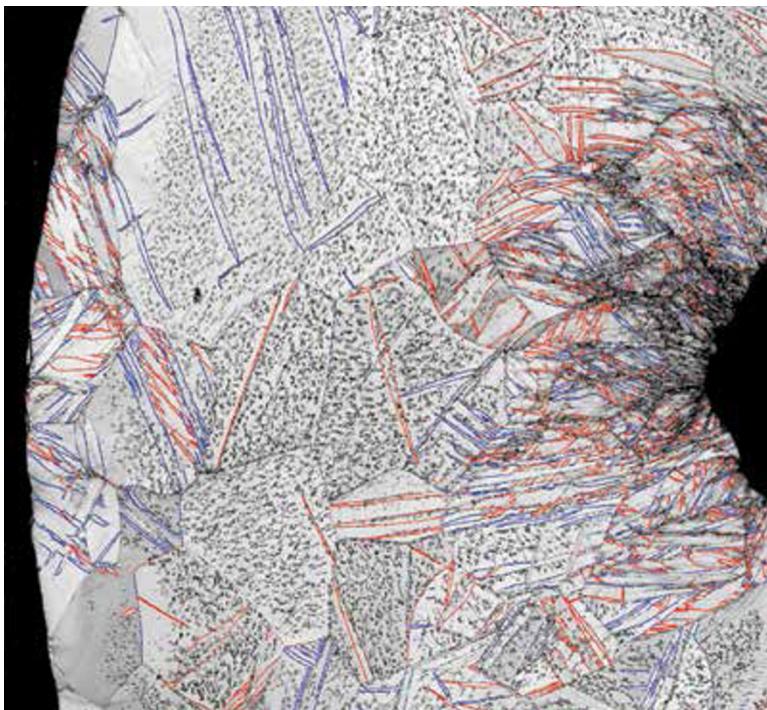
In today's EBSD systems, almost everything is automated. The electron beam from the SEM is scanned automatically over the surface of the material and the Kikuchi patterns projected onto the phosphor screen are automatically recorded and collected by the digital camera and then analyzed by a computer.

The system can even be calibrated automatically. Such calibration needs to be performed after altering the geometry of the system, such as the distance between the material and the SEM, as happens when analyzing different size materials. Today's systems automatically calibrate themselves when first turned on, by collecting Kikuchi patterns at various different distances between the material, SEM and screen, and then using this information to adjust for any subsequent alterations to the geometry.

Today's EBSD systems can also automatically determine a variety of different information about a material's crystalline structure from the Kikuchi patterns and then present this information in various different ways. So they can produce images mapping how the different phases making up a material are distributed over its surface, with each phase presented as a different color in the subsequent image. They can also show how the crystal orientation changes over the surface of a crystalline material by again displaying different orientations at different colors, with the degree of color change corresponding to the degree of change from some fixed orientation.

With this information on phase and orientation, EBSD systems can also map the many different grains making up a crystalline material. This is done by comparing the orientation of two neighboring points on the surface of the material and assigning

them to different grains if the difference in their orientation exceeds a certain value, say  $5^\circ$ . This produces an image of the different grains over the surface of the material, highlighting their different sizes and the boundaries between them. Alternatively, the orientation information can be used to produce plots of the changing texture over the surface of the material.



**Fig 6. EBSD image quality map from deformed titanium with twin boundaries displayed in color**

As well as producing all this information about crystalline structures, modern EBSD systems can also provide an assessment

of the accuracy of that information. This is necessary because the indexing process is not infallible: a consequence of variable image clarity and the fact that more than one possible orientation can often match the same pattern. One assessment method developed by EDAX involves determining a parameter known as a confidence index, indicating the degree of confidence that the orientation calculation is correct.

The confidence index takes advantage of the way in which the crystal orientation at a specific point is calculated. An estimate of the crystal orientation can be calculated from just three Kikuchi bands, known as a triplet, but many more bands (from seven to around 20) will be available in the image produced from a single scan point. So modern EBSD systems estimate the orientation of a scan point numerous times using different triplets (if an image has seven bands, then this will produce 35 different triplets) and then determine the most frequently estimated orientation as the correct one.

The confidence index is calculated from the ratio between the tallies of the most frequently estimated orientation and the second most frequently estimated orientation. Thus, if all the estimates agree on the same orientation, then this orientation will have a high confidence index, but if the most frequent and second most frequent estimates have similar tallies, then the orientation will have a much lower confidence index. The confidence index can vary from zero to one, but is usually between 0.1 and 0.9, with values below 0.1 highlighting suspect orientations.

The only thing that a user working with these EBSD systems needs to do is to prepare the material, which involves simply smoothing the surface using techniques such as mechanical

polishing or ion beam etching, place it on the stage and then set the analysis parameters. These parameters will include things such as the accelerating voltage, beam current, size of the SEM spot, size of the steps between each scan point, and the scan duration at each point, all of which influence the resolution of the subsequent image. Both the resolution and clarity of the image also depend on the material being studied, in particular on the atomic number of the material, with the resolution and quality of the Kikuchi patterns both increasing at higher atomic numbers.

The user can also specify whether and how the visual data captured by the digital camera should be binned, which involves combining several pixels in the camera display into a single pixel with an average value. While this obviously reduces the resolution, the advantages of binning are that it reduces background noise and also improves the effective light sensitivity of the digital camera, allowing higher scan speeds.

## CASE STUDY 1

*Despite the fact that mankind has been utilizing metals for thousands of years, only with the rise of analytical techniques such as EBSD are we now able to explore how the microstructure of metallic materials affects their physical properties. In particular, scientists such as Anthony Rollett, professor of materials science and engineering at Carnegie Mellon University, Pittsburgh, USA, are using EBSD to explore the effect of grain growth, size and distribution on these physical properties.*

*With its high resolution and speed, EBSD is an ideal analytical technique for these studies, as it allows Rollett to map the distribution of grains and crystal orientations over the surface of a metallic material in fine detail and then see how that distribution changes in response to deformation or heat. For example, Rollett has used EBSD to investigate how the microstructure of a nickel-based alloy responds to heat treatment and how the microstructure of multi-layered copper responds to deformation by large strains.<sup>1,2</sup>*

*‘EBSD is the only feasible technique for mapping orientation which provides info on gradients within grains and on grain boundary character,’ he says. ‘It allows a wider range of questions to be investigated in microstructure property relationships in materials.’*

*Sometimes this work can throw up some unexpected findings. For example, purely by chance, Rollett recently managed to capture an image of a quadrijunction using EBSD. Quadrijunctions form during grain growth, when two opposing grains grow to meet across an existing boundary between two grains. This causes the existing boundary to*

*disappear and a new boundary to form, but for a short amount of time all four grains meet at a single boundary known as a quadrijunction.*

*Because quadrijunctions only exist fleetingly, no one had actually seen one before. But with the fast imaging speeds available with EBSD, Rollett was recently able to observe one while investigating the effect of heat treatment on the microstructure of aluminium.<sup>3</sup>*

1. *Metallurgical and Materials Transactions A*, 2013, **44**, 2778-2798 (DOI: 10.1007/s11661-013-1749-0).
2. *Metallurgical and Materials Transactions A*, 2013, **44**, 3866-3881 (DOI: 10.1007/s11661-013-1749-0).
3. *Scripta Materialia*, 2013, **69**, 37-40 (DOI: 10.1016/j.scriptamat.2013.03.014).

## CASE STUDY 2

*Metallic materials may currently be one of the most common subjects for EBSD analysis, but Martin Lee, a professor in the School of Geographical and Earth Sciences at Glasgow University, UK, is showing how EBSD can be applied to some very different materials. These include Martian meteorites, the eyes of fossilized trilobites, the shells of living and extinct marine invertebrates, and mineral granules secreted by earthworms.*

*Lee is using EBSD to study a silicate mineral known as olivine in Martian meteorites, which are rocks blasted off the surface of Mars by impacts from comets and meteorites that eventually find their way to Earth. Studying the microstructure of olivine with EBSD can help to reveal whether these rocks were ever exposed to water on the Martian surface or whether carbon dioxide from the Martian atmosphere was ever sequestered in them.<sup>1</sup>*

*Trilobites are some of the earliest multi-celled organisms, thriving in the Earth's seas from around 500 million years ago to 250 million years ago. As such, their visual system was very primitive, with lenses made from the mineral calcite. Lee is using EBSD to study the microstructure of these lenses and gain an understanding of how they worked.<sup>2</sup> 'We've been looking at how the lens grew and how it operated, and that's dependent on the microstructure of the calcite,' explains Lee. 'So that's quite a direct link between the microstructure you get from EBSD and an important scientific problem.'*

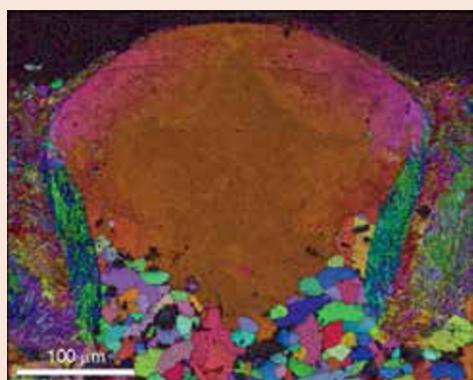


Fig 7. Electron backscatter diffraction map of a single calcite lens from a specimen of the trilobite *Geesops schlotheimi* from Germany, courtesy Martin Lee, University of Glasgow. The oval-shaped lens is in the center of the image. To its left and right hand sides is a finely crystalline calcite that supports the lenses, called sclera, and below the lens is the more coarse grained calcite of limestone rock that contains the fossil. Above the lens and along the top of the image is epoxy resin in which the specimen is mounted. The colours of this EBSD map show the crystallographic orientation of calcite crystals. The sclera and limestone contain crystals with many different orientations, whereas lens calcite has more or less the same orientation. The c-axis of the lens calcite is aligned more or less parallel to incident light, thus minimising the deleterious effects of birefringence.



Fig 8. Photograph of an enrolled specimen of the trilobite *Austeropssmoothops* from Morocco, courtesy Martin Lee, University of Glasgow. This lateral view of the specimen shows one of the animal's two eyes, which is composed of many small rounded lenses.

Although EBSD is often one of several analytical techniques that Lee uses when studying these materials, it was the only technique he could use to study the trilobite lens. 'The lenses are quite big [hundreds of microns] and so EBSD is probably the only way you can really get this information,' he says.

According to Lee, EBSD does have disadvantages, but these are intimately bound up with one of its main advantages: 'EBSD produces lots of data very quickly in a nice format and so one has to be very careful not to take the data at face value and to think about what it shows.'

1. *Nature Communications*, 2013, **4**, 2662 (DOI: 10.1038/ncomms3662).

2. *Palaeontology*, 2014 (DOI: 10.1111/pala.12088).

## PROBLEMS AND SOLUTIONS

Today's EBSD systems may be highly automated, but getting the best out of EBSD requires understanding both its intricacies and how it interacts with the material being studied. So, for example, although EBSD can analyse a whole range of different crystalline samples, certain fragile materials such as zeolites and biological shells can be damaged by the SEM beam, degrading the quality of the resultant Kikuchi patterns. Preventing this damage means either restricting the scan time to just a few microseconds, thus reducing the amount of data that can be gathered about the material, or turning down the power of the SEM beam, with the associated loss of resolution.

This propensity to cause damage prevents EBSD from being used to study soft biological material such as proteins or DNA, as does the fact their structure changes on scales that are too small for EBSD to capture. This means that EBSD cannot determine a specific crystal orientation at a specific point on the protein because a single scan will cover several different orientations. EBSD has a similar resolution problem with synthetic polymers.

Indeed, the resolution that can be achieved with an EBSD system very much depends on the microstructure of the material being studied. For a start, the resolution is higher for materials with a higher atomic number, as they reflect more electrons and so generate a larger signal, allowing a tighter beam and a smaller interaction volume. The resolution can also vary for the same material, depending on the state of that material; so the resolution may be higher for a pristine version of a material such as aluminium than for a version that has been deformed in some way.



**Fig 9. Orientation map of a deformed pearlitic steel alloy**

The conductivity of the material can influence the resolution, because ideally you want the electric charge that naturally builds up around the scan point to dissipate as quickly as possible. Otherwise the electric charge at one scan point may interfere with the scan at a neighbouring point if they are too close together, thus placing a limit on the step size between successive scan points.

In addition, keeping the sample as still as possible aids resolution. This can be a particular problem with EBSD because of the need to tilt the sample, which ensures the system is always fighting against gravity.

Obviously, this drift becomes more of a problem as the sample gets larger and/or heavier, because of the increased difficulty of holding it in place. Furthermore, although EBSD doesn't place as many restrictions on the sample as other analytical techniques, such as XRD and TEM, the sample obviously can't be too large otherwise it won't physically fit into the scanning chamber or on the stage. The only other real restriction is the need for the sample to have a smooth, well-polished surface for the SEM beam to scan across, and this can be produced by techniques such as mechanical polishing, electropolishing or ion beam etching.

The user also needs to ensure the system is physically set up in such a way that the SEM beam has an uninterrupted passage to the sample and the diffracted electrons have an uninterrupted passage to the phosphor screen, preventing any blocking of the beam or electrons. This can be particularly tricky when the EBSD system is combined with other analytical techniques, such as EDS, that also need a clear line of sight to the sample. Adding to the complexity is the fact that the sample needs to be tilted with respect to the detector for EBSD but not for techniques such as EDS.

Thus, fitting all the components of a combined EBSD and EDS system around a sample while ensuring that both systems have an uninterrupted view can be a challenge. It's made simpler if both the EBSD and EDS systems are made by the same company, as the two systems have then been designed to go together without interfering with each other in any way. The other advantage of obtaining EBSD

and EDS systems from the same company is that it ensures the software platforms for each system are compatible with each other. Indeed, such combined systems from a single company can often be controlled by a single, integrated software platform, allowing the user to switch between operating each system independently or together at the click of a mouse.

One of the great advantages of combining EDS with EBSD is that it helps to overcome one of the main limitations of EBSD, which is its difficulty in distinguishing similar crystal structures, such as phases where the lattice parameters of each phase differ by just 2%. If these phases differ in their chemical composition, however, then they can easily be distinguished by EDS.

The final challenge when using EBSD is actually interpreting the information that it generates. The latest systems can produce huge amounts of data and present that data in a wide variety of different ways, revealing information about the phases, crystal orientations and grain characteristics of the material. The real trick, though, is relating this information back to the questions being asked about the material; determining crystal orientation is one thing, working out what that orientation reveals about the history or physical properties of the material is something different. But this is really a challenge for the user rather than for EBSD.

## WHAT'S NEXT?

Faster computers and the introduction of digital cameras mean that EBSD has already come a long way since the first commercial systems were introduced over 20 years ago. Whereas the first systems could only scan the surface of a material at the rate of a single scan point per second, today's EBSD systems can scan at a rate of up to 1400 points per second. This increase in speed allows users either to collect the same data faster or to collect more data over the same scan period. What this means in practice is that today's system can produce a high-resolution image of the crystal orientation over a  $500\mu\text{m}^2$  region of the surface of a material in around a minute.

The adoption of digital cameras has also made EBSD systems more sensitive. High-performance optics that can focus light with minimal loss of intensity and charge-coupled devices with high quantum efficiencies ensure the cameras can rapidly capture the maximum amount of visual information from the phosphor screen.

The latest phosphor screens, meanwhile, display good, clear images of the Kikuchi patterns that nevertheless decay quickly to allow high-speed analysis. They can also respond to a wide range of SEM beam operating voltages, producing a high level of signal even at low voltages. Further advances in these components should continue to enhance the speed and sensitivity of EBSD, improving the resolution and allowing clear images of the Kikuchi bands to be produced from even poor quality or fragile samples.

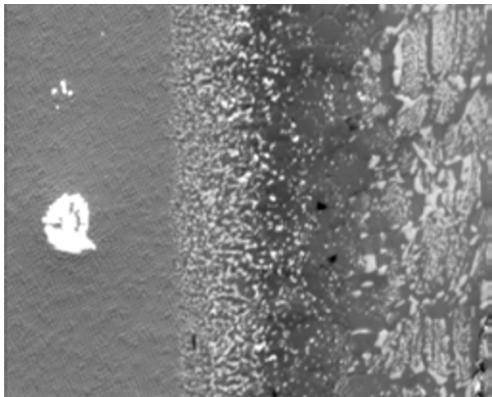
New ways of conducting EBSD, such as transmission EBSD, are also being developed. Transmission EBSD works with samples so thin that the electrons in an SEM beam pass straight through

them rather than rebounding off. The electrons are still diffracted during their passage through the material and so still produce Kikuchi bands on a phosphor screen, although this screen needs to be placed directly opposite the SEM rather than at a 90° angle. The advantage of transmission EBSD is that it can produce images with a higher spatial resolution than conventional EBSD while still using the same system components.

Another recent development is using an EBSD detector as an array of electron imaging detectors. With this approach, multiple regions of interest are defined on the phosphor screen, and the incident electron signal onto each ROI is monitored as the electron beam is rastered over the sample surface. Microstructural images are then created for each ROI from the variation in the incoming signal. These images show different contrast images, including orientation, phase, and topographic contrasts, and can be easily mixed to highlight specific contrasts or features of interest.

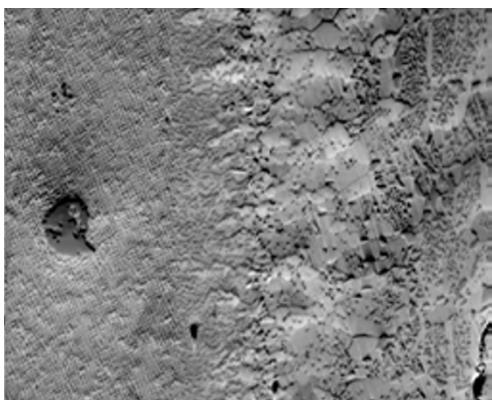
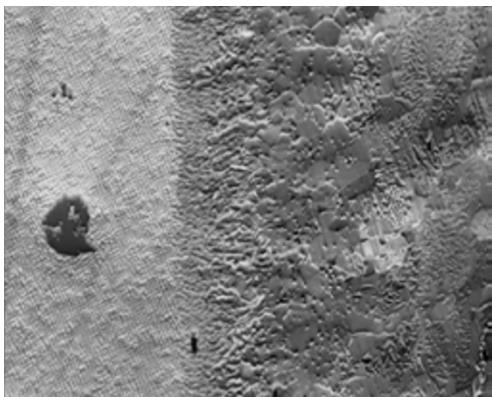
EBSD is also being combined with other analytical techniques. As occurs with EDS, one popular option is to combine EBSD with other characterization techniques, allowing researchers to obtain as much information as possible about the microstructure of a material. Analytical techniques that can be combined with EBSD include TEM, atomic force microscopy and atom-probe tomography.

Increasingly, though, researchers are combining EBSD with techniques for measuring physical properties such as hardness, magnetism and electrical conductivity, allowing them to investigate how a material's microstructure affects these physical properties. For example, a team from the Fraunhofer Institute for Non-destructive Testing in Saarbrücken, Germany, recently combined EBSD with magnetic force microscopy to study the effect of crystal orientation



**PRIAS (Pattern Region of Interest Analysis System)**  
imaging simultaneously detects  
multiple imaging contrast  
mechanisms.

These images were collected from thermal barrier coating of a nickel superalloy, with the top image showing atomic number contrast, the middle image showing orientation contrast, and the bottom image showing topographic contrast. These images can also be correlated with the EBSD orientation information.



on the magnetism of an iron-based material made up of cementite ( $\text{Fe}_3\text{C}$ ) particles embedded in a ferrite matrix.<sup>1</sup> This revealed that the magnetic moments of the cementite particles are highly dependent on their specific crystalline structure.

Some researchers are combining EBSD with a whole host of other techniques for both characterizing a material's microstructure and determining its physical properties. For example, a team of scientists from Tata Steel recently reported combining EBSD with optical metallography, electron probe microanalysis, XRD, TEM, tensile testing and density, and elastic modulus measurements.<sup>2</sup> Their aim was to use all these techniques to study the evolution of the microstructure and mechanical properties of low-density steel as it was processed for automotive applications.

The materials that EBSD is being used to study are also expanding beyond the traditional metallic materials, alloys and ceramics. This is being driven both by researchers in new fields becoming aware of the benefits of EBSD and by EBSD being combined with other analytical techniques.

In Case study 2, we saw how Martin Lee at Glasgow University, UK, is using EBSD to study the crystalline structure of the eyes of fossilized trilobites and the microstructure of Martian meteorites, with Lee's group being almost unique in using EBSD to study these materials. Other groups, though, are beginning to use EBSD to study silicon films, solar cells, biological material such as seashells and coral, and even ice.

They are also applying EBSD to the study of new advanced materials such as graphene, a one-atom thick sheet of carbon with some impressive physical and electronic properties. In 2013, a team of scientists led by Rodney Ruoff at the University of Texas

at Austin, USA, used EBSD to study the growth of graphene on copper.<sup>3</sup> The graphene was grown via a process known as chemical vapor deposition, in which a carbon-containing gas (usually methane) is passed over a substrate (in this case copper) at high temperatures, causing graphene to grow spontaneously on the substrate. Using EBSD, Ruoff and his colleagues discovered that large graphene domains usually grow across several copper grains, with each graphene domain possessing a single crystal structure even though the underlying copper grains possess a range of different orientations.

Indeed, EBSD has now become so easy to use and produces such a wealth of interesting information that it is beginning to be utilized by researchers with little interest in or knowledge of the intricacies of crystal structure. Rather, they are using it as another imaging tool or simply to tell whether a material's microstructure has changed by comparing images taken at different times.

From being a fairly niche technique for experts in crystal structure, EBSD is increasingly becoming a mainstream analytical technique for use by all scientists.

1. *Ultramicroscopy*, 2014, **146**, 17–26  
(DOI: 10.1016/j.ultramic.2014.05.003).
2. *Acta Materialia*, 2014, **75**, 227–245  
(DOI: 10.1016/j.actamat.2014.04.031).
3. *Science*, 2013, **342**, 720–723  
(DOI: 10.1126/science.1243879).

## FURTHER READING

EDAX website

(<http://www.edax.com/Literature/EDS-EBSD.aspx>).

Nowell, M. M. and Wright, S. I. (2004). Phase differentiation via combined EBSD and XEDS. *Journal of Microscopy* 213: 296–305 (<http://onlinelibrary.wiley.com/doi/10.1111/j.0022-2720.2004.01299.x/abstract>).

Zaefferer, S. (2007). On the formation mechanisms, spatial resolution and intensity of backscatter Kikuchi patterns. *Ultramicroscopy* 107(2-3): 254-266. (<http://dx.doi.org/10.1016/j.ultramicro.2006.08.007>).

Gourgues-Lorenzon, A.-F. (2009). Application of electron backscatter diffraction to the study of phase transformations: present and possible future. *Journal of Microscopy* 233(3): 460-473. (<http://onlinelibrary.wiley.com/doi/10.1111/j.1365-2818.2009.03130.x/abstract>).

Wright, S. I. and Nowell, M. M. (2009). A review of in situ EBSD studies. In: *Electron Backscatter Diffraction in Materials Science* (eds A. J. Schwartz, M. Kumar, D. P. Field and B. L. Adams), Springer, US ([http://link.springer.com/chapter/10.1007/978-0-387-88136-2\\_24](http://link.springer.com/chapter/10.1007/978-0-387-88136-2_24)).

Wright, S. I., Nowell, M. M. and Field, D. P. (2011). A review of strain analysis using electron backscatter diffraction. *Microscopy and Microanalysis* 17(3): 316-329. (<http://dx.doi.org/10.1017/S1431927611000055>)

Chen, Y. Hjelen, J., Gireesh, S. S. and Roven, H. J. (2012). Optimization of EBSD parameters for ultra-fast characterization. *Journal of Microscopy* 245: 111-118 (<http://onlinelibrary.wiley.com/doi/10.1111/j.1365-2818.2011.03551.x/abstract>).

Trimby, P. W. (2012). Orientation mapping of nanostructured materials using transmission Kikuchi diffraction in the scanning electron microscope. *Ultramicroscopy* 120: 16-24. (<http://dx.doi.org/10.1016/j.ultramic.2012.06.004>).

Maurice, C., Quey, R., Fortunier, R. and Driver, J. H. (2013). High angular resolution EBSD and its materials applications. In: *Microstructural Design of Advanced Engineering Materials* (ed D. A. Molodov), Wiley-VCH Verlag GmbH & Co. KGaA, Weinheim, Germany (<http://onlinelibrary.wiley.com/doi/10.1002/9783527652815.ch14/summary>).

