Recent Developments of Crystallographic Analysis Methods in the Scanning Electron Microscope for Applications in Metallurgy

Rafael Borrajo-Pelaez and Peter Hedström

Department of Materials Science and Engineering, KTH Royal Institute of Technology, Stockholm, Sweden

ABSTRACT

The field of metallurgy has greatly benefited from the development of electron microscopy over the last two decades. Scanning electron microscopy (SEM) has become a powerful tool for the investigation of nano- and microstructures. This article reviews the complete set of tools for crystallographic analysis in the SEM, i.e., electron backscatter diffraction (EBSD), transmission Kikuchi diffraction (TKD), and electron channeling contrast imaging (ECCI). We describe recent relevant developments in electron microscopy, and discuss the state-of-the-art of the techniques and their use for analyses in metallurgy. EBSD orientation measurements provide better angular resolution than spot diffraction in TEM but slightly lower than Kikuchi diffraction in TEM, however, its statistical significance is superior to TEM techniques. Although spatial resolution is slightly lower than in TEM/STEM techniques, EBSD is often a preferred tool for quantitative phase characterization in bulk metals. Moreover, EBSD enables the measurement of lattice strain/rotation at the sub-micron scale, and dislocation density. TKD enables the transmitted electron diffraction analysis of thin-foil specimens. The small interaction volume between the sample and the electron beam enhances considerably the spatial resolution as compared to EBSD, allowing the characterization of ultra-fine-grained metals in the SEM. ECCI is a useful technique to image near-surface lattice defects without the necessity to expose two free surfaces as in TEM. Its relevant contributions to metallography include deformation characterization of metals, including defect visualization, and dislocation density measurements. EBSD and ECCI are mature techniques, still undergoing a continuous expansion in research and industry. Upcoming technical developments in electron sources and optics, as well as detector instrumentation and software, will likely push the border of performance in terms of spatial resolution and acquisition speed. The potential of TKD, combined with EDS, to provide crystallographic, chemical, and morphologic characterizations of nano-structured metals will surely be a valuable asset in metallurgy.

KEYWORDS

Electron microscopy; steels; metals; EBSD; TKD; ECCI

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CONTACT  Rafael Borrajo-Pelaez  rafaelbp@kth.se

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1. Introduction

The understanding of the relationship between microstructure and macroscopic properties plays a key role in the development and manufacture of materials. The ultimate aim of metallurgy is to control the microstructure of metals by processing in order to optimize their properties and performance, thus microstructural investigations are essential. The field of metallography has undergone dramatic advancement over the past few decades thanks to the continuous developments of electron microscopy, where scanning electron microscopy (SEM) has become a tool of common use, serving as a platform for the development of materials characterization techniques. Quantitative metallography has made significant progress by the discovery of electron backscatter diffraction (EBSD) and the introduction of automated EBSD in the 1980s and early 1990s, and its concurrent commercial availability.\(^4\)\(^6\) This technique has allowed fast and robust analysis of bulk polycrystalline materials and quantitative characterization of micro- and macro-texture, orientation correlations between phases, grains, and domains, as well as phase identification and size distributions of constituents. The application of EBSD has, for instance, contributed to the understanding of phase transformations in high-performance steels,\(^7\)\(^8\) deformation of light metals,\(^9\)\(^10\) grain boundary engineering in superalloys,\(^11\) and grain coarsening in hard metals.\(^12\)\(^13\) A comprehensive review of the physical fundamentals of EBSD can be found in Wilkinson and Hirsch.\(^14\) Furthermore, overviews of the application of EBSD in characterization of grain and subgrain structures,\(^15\) applications in steels research,\(^16\) and EBSD in conjunction with other characterization techniques are available.\(^17\)\(^18\) In 2011, Zaefferer critically assessed EBSD and diffraction-based techniques in the transmission electron microscope (TEM), comparing them with respect to, e.g., their spatial and angular resolution.\(^19\) He concluded that EBSD is in many cases preferred over the TEM techniques for orientation mapping, but for truly nano-crystalline materials, TEM was suggested to be superior. Since then, however, in 2012 a complementary approach to EBSD in the SEM has been proposed by Keller and Geiss, named transmission-EBSD (t-EBSD), also known as transmission Kikuchi diffraction (TKD), or electron forward scatter diffraction (EFSD).\(^20\)\(^–\)\(^22\) The main difference between standard EBSD and this new technique lies in the sample geometry. While the former is performed over bulk materials, the latter is based on the use of electron transparent samples, so that the electrons are transmitted through the sample, reducing the volume of interaction between the electron beam and the specimen. This translates into significant improvements of the spatial resolution of one order of magnitude with respect to EBSD.

A technique which attracts increasing attention in the recent years is electron channeling contrast imaging (ECCI). ECCI has been available for 50 years, but is currently being applied in larger extent in metallurgy due to recent technical developments that have simplified the analysis.\(^23\)\(^–\)\(^25\) Moreover, the improved SEM performance together with the operation of ECCI in controlled diffraction condition, i.e., in combination with EBSD, has enabled high-resolution studies of nano-scale structures.\(^26\)\(^\)\(^27\)

The application of EBSD, TKD, and ECCI for the characterization of metallic microstructures is likely to continue expanding. These techniques are obviously important alone but their complementarity should also be considered. By combining them, new possibilities in metallography arise and, hence, it is worthwhile surveying the current state of the complete set of tools for crystallographic analysis in the SEM. The purpose of this report is to review their latest technical advancements and their use in research in metallurgy. Application examples are specifically taken from research on steels. It should be noted that crystallographic analysis using dedicated electron transmission detectors in SEM is covered in this report only for the case of on-axis TKD.

The structure of this document is the following. The remaining part of this Introduction, Section 1.1, briefly presents recent developments of relevance in SEM technologies. Sections 2–4 provide the current status of EBSD, TKD, and ECCI, respectively, including examples of application of these techniques in steel research. Finally, Section 5 contains the concluding remarks and provides a brief outlook about the opportunities of these techniques in the field of metallurgy.

1.1. Developments in scanning electron microscopy

In this subsection, we aim to provide a brief account on recent developments in SEM which have bearing on crystallographic analysis. Decades of continuous advances in SEM have contributed to obtain high resolution and image quality.\(^28\)\(^29\) The general efforts to improve SEM performance have mainly been directed towards the maximization of the brightness of the electron sources and the mitigation of fundamental aberrations in the electron-optical systems. Great improvements have been achieved by substituting conventional thermionic electron emission guns for field emission guns (FEGs). The large size of thermionic filament tips, with diameters up to several tens of micrometers, is associated with relatively low brightness, and the occurrence of evaporation of cathode material and thermal drift during operation.\(^29\) On the other hand, field emission from the sharper FEG emitters,
with tip radii down to several tens of nanometers, produces electron beams which are two orders of magnitude smaller in diameter, more coherent and with up to three orders of magnitude greater current density or brightness than what can be achieved with thermionic emitters. This results in significantly improved signal-to-noise ratio, spatial resolution, emitter life, and reliability compared with thermionic devices.30 There are two different types of FEG: cold-cathode emitters and Schottky emitters. In cold cathodes, electron emission takes place from the tip of a sharpened tungsten single crystal. In the case of the thermally assisted Schottky type, a reservoir of zirconium oxide is attached to a wire of single crystalline tungsten in the form of a coating, in order to lower its work function and thus enhance the electron emission. Table 1 provides a comparison between the typical brightness, energy spread, and source size of thermionic, Schottky and cold field emission guns.31

Crystallographic techniques in the SEM, relying on the detection of backscattered or forescattered electrons (BSE/FSE) of energies close to the energy of the primary electron beam, have benefited from the availability of these advanced electron sources, associated with higher electron back-/forescatter signal-to-noise ratios and smaller probe sizes. A lower acceleration voltage is beneficial to reduce the probe volume for studies on bulk samples. This translates into enhanced spatial resolution, but on the other hand, low acceleration voltage is associated with increasing electron-optical aberrations, and higher sensitivity towards contamination effects.32,33 Furthermore, the brightness of the electron probe scales with acceleration voltage and it can be difficult to achieve sufficient signal at high-resolution with very low primary electron energies. This effect is lower using FEG guns, compared to thermionic filaments. Current developments in FEGs aim towards the substitution of tungsten tips for other materials to improve the source performance. Promising options currently being developed are carbon cone nanotips, with better current stability, and multi-walled graphene nano-cones.34,35

Another factor limiting SEM performance in crystallographic analyses is electron-optical aberrations, mainly spherical and chromatic aberrations. Spherical aberration is caused by the difference in focal length between rays travelling far from the lens’ optical axis and rays close to the axis.36 Chromatic aberration, in turn, occurs because the focal length of the objective lens in the SEM varies with the energy of the electrons passing through it. It can be described as \( C_d \frac{dE}{E} \), where \( C_d \) is the chromatic aberration coefficient, \( dE \) is the energy spread of the electron beam, and \( E \) is the accelerating voltage of the beam. The use of FEGs contributes to mitigate chromatic aberration, since they produce beams with narrow energy distributions, i.e., low \( dE \). For instance, the value of \( dE \) for cold-cathode and Schottky field emitters is in the order of 0.2–0.3 eV and 0.3–1 eV, respectively, as opposed to the larger 1–3 eV of thermionic sources (see Table 1).37 SEM resolution diminishes strongly when low accelerations are required (low \( E \)), a problem which has motivated investigations on two additional methods to counteract chromatic aberration: the use of monochromators and aberration correctors.29,32 Monochromators narrow the energy spread \( dE \) of the electron beam, resulting in improved SEM image resolution at low energies. Aberration correctors consist of multistage quadrupole–octopole optics which reduce the value of \( C_d \).38 Aberration coefficients scale up with the microscope working distance (WD). Hence, high-performance SEM, e.g., ECCI imaging, requires the minimization of WD, which entails restrictions of the tilt and manipulation range of the sample and access limitations to the detectors. Aberration corrected SEMs can remove these WD constraints. Unfortunately, the use of aberration-corrected SEM is associated with significant extra cost, difficulties related to alignment procedures, and the reduction of imaging depth of field produced by the much larger numerical aperture required by this type of lenses.36 Another innovation towards high-resolution SEM has been the shortening of the specimen-lens distance by the introduction of magnetic immersion lenses where the specimen is placed inside the lens magnetic field, as well as high signal collection efficiency in-lens detectors. The schematic on Figure 1a shows an example of in-lens objective lens and Figure 1b presents a semi-in-lens objective lens with a through-the-lens secondary electron detector. However, immersion lenses are subject to limitations. One of them is the constraint imposed by the magnetic nature of certain specimens. Another undesired effect is the impact of their magnetic field on the diffracted electrons in the direction to the EBSD camera.39 This causes bending of the diffraction patterns, which need to be corrected to flatten the Kikuchi bands. One of the latest designs of objective lens is the so-called hybrid lens, a compound lens consisting of both electromagnet and electrostatic lenses. Unlike semi-in-lens objective lenses (Figure 1b), hybrid lenses do not leak an

| Electron source               | Brightness (A/cm² sr) | Energy Spread (eV) | Source size (nm) |
|------------------------------|-----------------------|--------------------|------------------|
| Thermionic                   | \( 10^5 \)–\( 10^6 \) | \( 1–3 \)            | \( 5,000–100,000 \) |
| Schottky field emission      | \( 10^5 \)            | \( 0.3–1 \)         | \( 15–30 \)       |
| Cold field emission          | \( 10^8 \)            | \( 0.3 \)           | \( <5 \)          |

Table 1. Properties of electron sources as a function of their type. Data extracted from Suga et al. 2014.31 (© Elsevier. Reprinted with permission from Suga et al.31 Permission to reuse must be obtained from the rightsholder.)
Hence, the hybrid objective lenses allow EBSD, TKD, and ECCI analyses at shorter WD. This is especially critical in TKD and ECCI as will be discussed further in Sections 3 and 4. Finally, it is worth mentioning the benefits for EBSD and ECCI of microscopes with depth-of-field mode, or with at least a very low convergence angle of the beam, providing improved imaging of tilted samples, with fewer requirements in terms of dynamic focusing. Another general consideration that is critical when utilizing crystallographic methods in the SEM is the sample preparation. For all three techniques, it is of high importance to have excellent samples, which in the case of EBSD and ECCI means a deformation-free surface in order to enable good diffraction and channeling signal, respectively. Furthermore, in the case of EBSD, it is essential for the specimen’s surface under analysis to have gentle topography so that the signal is not shadowed and can reach the detector. In the case of TKD the sample thickness is also critical. Over the last years, sample preparation methodologies and instrumentation have been improved in parallel with SEM technologies. Examples of this are the sample preparation procedures via focused ion beam (FIB) and broad ion beam (BIB). In addition, traditional methods such as electro-polishing and vibrational polishing have become important in this new field.

2. Electron backscatter diffraction (EBSD)

2.1. EBSD measurements and interpretation

The schematic of a typical EBSD setup configuration is shown in Figure 2a, where a bulk sample in 70° tilt position is irradiated with an electron beam. The Kikuchi patterns produced by the diffraction of backscattered electrons on the lattice planes yielding diffraction cones are captured by the side-mounted phosphor screen of an EBSD detector. Recent developments in EBSD have increased the speed, reliability, and versatility of this technique. Substantial improvements in camera technology include higher

Figure 1. Schematics of two types of high-resolution SEM magnetic immersion objective lenses for low specimen-lens distance: (a) in-lens objective lens and (b) semi in-lens objective lens with a through-the-lens secondary electron detector.

Figure 2. Schematics of the typical experimental setups used in (a) EBSD, (b) TKD, and (c) ECCI, for both high-tilt and low-tilt configurations.
sensitivity and pattern acquisition rates which have increased by 10-fold in the last decade, leading to the current operation conditions with acquisition frequencies above 1,000 frames per second, and higher pixel resolutions over 5 Mpix, as well as better accuracy in the definition of sample-pattern geometry. It should be mentioned nonetheless that the highest speed is not obtained using the highest pixel resolution, but binning of several pixels is necessary in such measurements. The availability of high-speed digital cameras not only has benefitted EBSD, but it has also enabled the emergence of EBSD-based dark-field imaging, a promising technique consisting in the use of the EBSD detector as an imaging device.\textsuperscript{45,46} Extreme binning of the EBSD CCD camera makes it possible to acquire images at a rate comparable to slow scan imaging in the SEM, providing information on topography, atomic density, and orientation contrast. Additional developments have recently provided an alternative to CCD-based detectors, aiming to suppress phosphor screens in favor of direct electron detection to eliminate inefficiencies. A first commercially available CMOS-based detector has been released, enabling a dramatic increase of the maximum frame rate of EBSD (exceeding 3,000 frames per second) while minimizing the penalty of extreme pixel binning and associated loss of diffraction pattern detail.\textsuperscript{47–49}

Other sources of enhancement reside in image processing, phase identification methods, and data analysis.\textsuperscript{18,50} Improvements in SEM discussed in the previous section, such as the introduction of FEGs, have contributed to a significant enhancement of EBSD’s spatial resolution. The interaction volume during EBSD measurements depends on the material density and beam acceleration voltage, and the increasing sensitivity of detectors has made it possible to reach lower acceleration voltages.\textsuperscript{51} It is worth mentioning that Kikuchi diffraction patterns are produced by quasi-elastically scattered electrons, i.e., electrons with spatial resolution when considering all the BSE electrons, which contribute to the background but not to the diffraction signal of interest.

The physical resolution of EBSD is defined as the smallest distance between the current electron beam position and a large angle grain boundary where still no signal, i.e., no Kikuchi band, from the neighboring grain is obtained on the detector. The state-of-the-art physical resolution of EBSD reaches limits of about 20 nm for dense materials such as Pt, 50 nm for lighter materials e.g. transition metal alloys, and 200 nm for aluminum alloys. The referred values apply in the direction parallel to the tilt axis, but physical resolution is significantly lower in the direction perpendicular to the tilt axis because of the interaction of the electron beam and the surface tilted to 70°.\textsuperscript{44,52,53} The effective resolution, a function of the physical resolution, denotes the accuracy of the EBSD software algorithm to resolve a large-angle grain boundary. It can be defined as the smallest distance between two points of different orientations where these orientations can still be uniquely distinguished by the software algorithm.\textsuperscript{54,55} The effective resolution is usually better than the physical resolution, although there are known exceptions such as the case of heavily deformed materials or low-angle grain boundaries. Values of effective resolution three times better than the physical resolution have been reported.\textsuperscript{17,50,56} The angular resolution, i.e., the smallest difference in orientation that can be detected between two consecutive points, is approximately 1° for high speed EBSD mapping. The value of angular resolution depends on parameters related to pattern binning and the Hough transform used during pattern band indexation. These parameters can be optimized, and angular resolutions as low as 0.1° can be obtained at the expense of mapping speed.\textsuperscript{19} Even lower angular resolution down to 0.003° is achieved through Wilkinson’s pattern cross-correlation technique.\textsuperscript{57} These values of angular resolution are comparable to those obtained by X-ray diffraction, and by diffraction techniques in the TEM (e.g. convergent beam electron diffraction, CBED) where angular resolutions range between 1–3° and 0.01°, depending on the technique and the resolution selected for the Hough transform.\textsuperscript{19,58}

Improvements in the interpretation of diffraction patterns have also contributed to further development of the EBSD technique. The majority of commercial EBSD systems use Bragg’s equation to predict the positions of the Kikuchi band edges. These band edges correspond to lattice planes, while their intersections correspond to zone axes, and both relate to the crystallography and crystal orientation. The band width, given by Bragg’s law, depends on the lattice parameters and the acceleration voltage. For further understanding of Bragg’s law and its connection to Kikuchi patterns the reader is referred to the works by Schwartz et al.\textsuperscript{44} and Baba-Kishi.\textsuperscript{59} Kinematic diffraction models are adopted in order to estimate the relative intensities of the Kikuchi bands. The pattern bands are detected by means of Hough transform, and the angles between the corresponding crystal planes are used as a key to match against the theoretical interplanar angles.\textsuperscript{56,60} Figure 3 shows the comparison of several EBSD patterns obtained from a duplex stainless-steel sample where two phases, ferrite and austenite, are present. Figures 3a and 3b show the experimentally obtained Kikuchi patterns of ferrite and austenite respectively, while Figures 3c and 3d display the kinematical simulation of the EBSD patterns of ferrite and austenite. As the complexity of the sample’s
structure increases, the pattern indexation becomes more challenging. For instance, when the sample is formed by multiple phases, especially if unknown phases are present, or when phases to be distinguished have similar crystal structure, indexing routines often fail unpredictably, as the result will match more than one phase. An alternative approach in such cases is to use the lattice parameter, i.e., the band width, in addition to the angle between bands as a criterion for sorting the solutions. For this method to be effective, it is required that the lattice parameters of the phases involved are sufficiently different. This feature is now available in commercial EBSD software packages. Another way to make phase discrimination between phases of similar symmetry is to run EBSD in tandem with EDS, with a configuration as shown in Figure 2a. However, this approach only works when the chemistry of the phases is distinct and, for instance, it may not be applicable to discriminate vital phases in steels such as ferrite, bainite, and martensite. In addition, large size differences of the beam interaction volume exist between EBSD and EDS due to the use of different signals, i.e. backscattered electrons versus characteristic X-rays. In order to provide a quantitative example, it can be noted that Monte Carlo electron trajectory simulations performed with the software Casino, considering an electron beam in EBSD configuration with a primary electron energy of 20 keV interacting with a Fe substrate, indicate that the backscattering diffraction interaction depth is about 50 nm (assuming low-loss electrons with more than 90% of the primary electron beam energy contribute to the diffraction), whilst the interaction depth for X-rays is about 1 μm. When the aforementioned methodologies cannot be used to discriminate constituents such as martensite, bainite, and ferrite, the pattern quality or band contrast, a metric related to the quality of the Kikuchi pattern, can be used. Phases or constituents that have different defect density will yield patterns of different quality, enabling a methodology which has been used successfully in a number of cases.

A substantial improvement in pattern interpretation can be obtained by simulating a quantitative description...
of the EBSD patterns (EBSPs) and hence extracting the information provided by intensity variations in the EBSPs. The aforementioned standard simulation method that establishes a reflector ranking based on kinematic theory has a limited capability. The local intensity distribution within an EBSP is difficult and time-consuming to calculate since the main assumptions of the kinematic theory are not fulfilled. A complete simulation of the observed intensities is only possible by applying the dynamical theory of electron diffraction which properly includes the effects of multiple scattering in the crystal. Furthermore, it was known that the effective TEM and the hierarchic structure of martensite was not consistently known. Some authors had suggested that twin-related martensite-martensite boundaries were preferred in lath martensite, whereas other reports showed other types of orientation correlations. Some of these inconsistencies might have been related to the poor statistics attainable by careful TEM operation. Moreover, many of these studies had been performed by selected area diffraction analysis, which offers a limited angular resolution and often accuracies as low as a few degrees are obtained, unless special care is taken. In such cases, the accuracy is in the same order as the different ORs proposed. It should be noted that Kikuchi diffraction analysis in the TEM has a higher angular resolution, as described in Section 2.1. In addition to the angular uncertainty in many studies, all TEM methods applied to that date had relied on the presence of sufficiently thick regions of retained austenite as a reference. Therefore, it had been difficult to study the details of ORs of martensite and bainite in most low-alloyed steels with a high martensite start temperature (Ms) and thus low fraction of retained austenite.

The major advantages of EBSD compared to TEM, such as the automatized measurements of large areas of bulk samples with routine angular resolutions of about 0.5°, have stimulated the reinvestigation of the orientation correlations of vital phases in steels. Morito et al. performed systematic EBSD investigations, in combination with TEM, on the microstructure of lath martensite in low-carbon steels and in alloy steels. The more significant statistics of the EBSD data with high angular resolution led them to extract conclusions on the most frequent martensite-martensite boundaries in these steels and also to find out that some of the previous observations, e.g., twin boundaries in low-carbon lath martensite, were either insignificant from a statistical point of view or were subject to inaccuracies in the TEM analyses. Their observations have led to better understanding of shape strain accommodation and the nucleation and growth mechanisms of the laths.

A comprehensive example, illustrating the effect of alloying on the martensitic microstructure is extracted from Stormvinter et al.; see Figure 4. This example shows the change of martensite microstructure, and the quantification of the martensite boundaries (Figure 4a), as a function of carbon content, from an interstitial-free (IF) steel to a steel with 1.8 wt.% C. It is shown that the low-angle misorientation martensite boundaries, preferred in low-carbon lath martensite (Figure 4b) are replaced by twin-related boundaries when the carbon content is 0.75 wt.% C (Figure 4c). For high-carbon plate martensite, the units will instead arrange in plate groups (Figure 4d). These EBSD results were enabled by the development of methods to determine the OR without the presence of retained austenite. It should be mentioned that the spatial resolution of EBSD does, in general, not allow for investigations of the planar defects in plate martensite, which are often in the order of 5–10 nm, except in favorable cases when both resolution and orientation are optimized and defects are larger. Instead, studies of the defects are possible using TKD,
explained in the next section, or by means of automated orientation mapping in the TEM.85–87

Similar EBSD investigations as for martensite in carbon steels have been conducted on bainite and metastable austenitic stainless steels and it has been shown, for instance, that the preferred boundaries depend on the temperature of formation and relate to the available driving force and defect mobility.88–91

2.3. EBSD in the measurement of strain, crystal rotation, and dislocations

A relevant contribution of electron diffraction in the SEM to the field of metallurgy is the possibility to measure lattice strain and rotation at the submicron scale. The presence of strain in crystalline materials produces effects in their lattice, different for elastic and plastic strains, which can be noticed in the EBSPs. Elastic strain causes changes in the lattice parameter associated to the direction of the applied or residual strain. This translates into shift of zone axes and change in the width of some of the EBSP bands. If the lattice is bent, lattice planes are not exactly parallel, which results in slight deviations in the Bragg angle along the length of the planes, and it manifests itself in a slight blurring of the edges of the diffraction bands, in addition to axis shift and variations in the band width.92 Plastic strain is generally associated with the formation of dislocations, which causes pattern quality degradation. If the diffraction volume is contained within a subgrain, i.e., a region of high dislocation density but with a net Burgers vector of zero, then pattern degradation is due to local perturbations of the diffracting lattice planes producing incoherent scattering. If the diffraction volume extends to more than one subgrain, then the pattern degradation is mostly due to the superposition of the patterns from each individual subgrain. It must also be mentioned that pattern quality may still be high, e.g., inside dislocation cell structures or in the micro-scale due to recovery or recrystallization even though the material has suffered from macroscopic deformation. Regardless of the type of strain, the referred effects on the patterns can be as subtle as a fraction of a pixel in band shift or slight pattern quality degradation. Therefore, high precision is required in order to detect these phenomena.93 Measurements of relative strain and rotation via high-resolution EBSD are based on the direct comparison of EBSPs. One of the most successful approaches is to measure changes in interplanar angles.
and then determine the deviatoric components of the elastic strain tensor. Cross-correlation functions are used to detect small shifts over regions of interest (ROI) in the patterns, compared to a reference pattern obtained from the crystal in a known strain state.\textsuperscript{94-96} The resolution on the pattern shifts is in the sub-pixel range, typically $\pm 0.05$, being the precision of the method $\sim 10^{-4}$ in strain and $10^{-4}$ rad ($0.006^\circ$) in rotation.\textsuperscript{97,98} Even higher precision is achievable if signal to noise ratio in the patterns is improved by simple integration.\textsuperscript{99} Additional experimental factors must be taken into account for high accuracy in the results. Very careful sample preparation is necessary to obtain good surface finish, minimizing any generation of plastic deformations on the sample that may lead to pattern distortion and to mask the true strain.\textsuperscript{93} Aberrations must be minimized in the lenses used for pattern acquisition, since image shifts can produce strain uncertainty of $\sim 3 \times 10^{-3}$. The experimental setup geometry must be accurately determined, in particular, the pattern center location and the detector distance. Uncertainties of $\sim 0.5\%$ in these measurements can induce errors of $\sim 5 \times 10^{-3}$ in strain measurements.\textsuperscript{98} The need for using a reference EBSP can pose considerable experimental limitations on the measurement, as there is not always a region of the sample for which strain and orientation are known, or a strain-free region. Recent developments of the technique aim toward the use of simulated EBSPs with known strain, which can be obtained from simple Bragg’s law simulations.\textsuperscript{100} It is expected that this approach will extend to the use of reference patterns simulated with dynamical diffraction theory, as mentioned in the previous section. The pattern simulation approach removes the problem of the reference pattern, enabling the measurement of strain and rotation in the absence of an area of known rotation, or a strain-free region. Preliminary work has been published by Kacher et al.\textsuperscript{100} and Villert et al.\textsuperscript{69} However, the consolidation of this approach requires overcoming the current lack of mechanisms to calculate the pattern center with sufficient accuracy.

An example of strain mapping in steels can be extracted from the work of Miyamoto et al., who performed EBSD measurements in austenitic-martensitic alloys in order to characterize the strain and rotation distributions produced in the austenitic matrix due to accommodation of the shape strain associated with the formation of martensite. The effect of different morphologies of martensite, e.g., lath, lenticular, and thin-plate, is considered. In the case of thin-plate martensite, the components of strain and rotation tensors in the austenitic matrix have nearly the same magnitude, i.e., the shape strain of thin plate martensite is accommodated by elastic deformation of austenite. The austenite matrices surrounding lath and lenticular martensite, however, present strain tensors whose components are much smaller than the components of the rotation tensors, i.e., most of the shape strain associated with the formation of lenticular and lath martensite is accommodated by plastic deformation in the austenite matrix.\textsuperscript{101} Figure 5a shows an austenitic region surrounding a lenticular martensitic grain in a Fe-33Ni alloy. Line scans of the elastic strain tensor component ($e_{11}$) in the austenitic region reveal an increase of the strain nearby the martensite. Figure 5b shows a pattern quality map with enhanced contrast of the same lenticular martensite grain and the surrounding austenite. As the pattern quality is affected by residual strain in the diffracting volume, strained areas appear darker than unstrained regions of the microstructure in this map, providing an indication of the distribution of strain in the material.\textsuperscript{93}

When a crystal undergoes plastic strain, the distortions originated in its lattice are relieved by the formation of dislocations.\textsuperscript{93} Once generated, dislocations can move and be stored. Different types of dislocations can be differentiated depending on the way they become stored. Those dislocations accumulating by trapping each other randomly are referred to as statistically stored

![Figure 5](image-url)
dislocations (SSDs), and the regions of the material where they accumulate typically have a net Burgers vector of zero. In turn, dislocations located in areas with net non-zero Burgers vectors, across which there are changes in crystallographic orientation or lattice curvature, are known as geometrically necessary dislocations (GNDs). GNDs provide the lattice continuity in the presence of curvature and arrays of this type of dislocations can form subgrain boundaries. The presence of dislocations is known to play a role in the mechanisms of strain-hardening and size-dependent plasticity. These mechanisms, particularly noticeable in the nanometer scale, are explained by physical models that relate the strengthening of materials with dislocations density. Moreover, models that account for dislocation density have been developed to understand the microstructure evolution of steels through different thermomechanical processes. Therefore, the empirical quantification of dislocation density is very important in metallurgy to better understand the behavior of materials at different scales. Dislocation density has typically been measured either by direct methods, such as TEM, or indirect methods, for instance x-ray diffraction (XRD). TEM’s high resolution allows for the clear distinction of dislocations and accurate calculation of dislocation density, unless dislocation density is very high (e.g., ferrous martensite), but the need for ultra-thin sample preparation makes this technique a time-consuming approach. Furthermore, the generation of two free surfaces in TEM specimens may cause stress relaxation and transform lattice strain into lattice rotation. The measurement provided by XRD is an average of the dislocation density in the bulk material. This method is more rapid than TEM, but it is subject to limited spatial resolution. Moreover, this technique requires a model to relate a total displacement gradient field with a certain dislocation density.

EBSD can be used to characterize the dislocation density, since the misorientation between two neighboring points provides an approximate measure for the presence of GNDs. This 2-D approach is based on the measurement of orientation gradients by EBSDs, and the subsequent calculation of the dislocation density tensor. However, spatial resolution and other experimental limitations in the determination of orientation gradients hinder the capability of this procedure to provide dislocation measurements with the high accuracy achievable with TEM. The resulting dislocation density measured by this approach is a lower bound of the total dislocation density, not solely because only GNDs are considered, but also because the entire dislocation tensors cannot be calculated as these require information about orientation gradients in the direction normal to the sample’s surface, which is not available unless 3-D techniques such as FIB/SEM-based 3D-EBSD are applied. An alternative approach to measure dislocation density via EBSD is to sum the dislocation density around an enclosed area, rather than measuring the strain around individual dislocations, removing the spatial resolution limitation. Finally, the advent of TKD (described in the next section) enables mapping on the scale of individual dislocations. However, decreasing the measurement spacing results in a higher dislocation noise floor, since the inherent errors associated to each orientation measurement yield greater orientation gradients and hence higher dislocation densities. To sum up, EBSD provides a fast method to measure local dislocation densities of bulk samples on a wide field of view, without the need of time-consuming sample preparation.

3. Transmission-EBSD or Transmission Kikuchi diffraction (TKD)

Transmission-EBSD is an electron diffraction technique in the SEM which was introduced by Geiss et al. (2010). Based on the standard EBSD setup, the main difference between this technique and EBSD is the use of thin foils instead of bulk samples. In t-EBSD the sample is electron transparent, so the electrons are transmitted through the sample and the diffraction patterns are projected on the phosphor screen of the EBSD detector. Since no backscattering of electrons is involved, the more appropriate acronyms transmission electron forward scattered diffraction (t-EFSD) and transmission Kikuchi diffraction (TKD) have been proposed. A typical TKD configuration is shown in Figure 2a, where the sample is mounted with a tilting angle of 20° from the EBSD detector, i.e. in the direction perpendicular to the standard EBSD sample position. The tilting angle used in TKD commonly ranges between 0° and 40°. The working distance, usually lower than in standard EBSD, i.e., in the 4–5 mm range, should be optimized for each material so that the EBSD illumination at the phosphor screen is highest, since the number of electrons reaching the screen is a function of the scattering angle. The distance between the sample and the EBSD camera should be minimized, and the latter oriented toward the bottom of the specimen in order to maximize the collection efficiency of transmitted electrons. A new dedicated setup configuration for TKD analysis has been recently developed. As shown in Figure 2b, the geometry of the detector is modified, so that the phosphor screen is located in horizontal direction under the sample. This configuration maximizes the collected transmitted signal and the resolution. On the other hand, certain drawbacks need to be addressed before it can be used with the same throughput as the
normal EBSD set-up. First, spots can appear on the pattern depending on the parameters used; second, the presence of the transmitted beam has a great impact on the illumination of the CCD; and third, this TKD configuration presents higher sensitivity to sample thickness, increasing the difficulty to analyze regions with varying thickness in a single analysis, unlike conventional off-axis TKD. In order to extract useful diffraction information from this on-axis configuration, the first and second issues need to be integrated in the acquisition software.

The electron beam’s accelerating voltage has a strong effect on the quality of TKD patterns and orientation maps. A higher accelerating voltage is generally beneficial in order to get good quality orientation maps, since beam broadening increases inversely to beam voltage. The lower accelerating voltage of TKD compared to TEM results in significant broadening of the scattering cross-section through the sample, causing loss of lateral resolution. At the same time, a certain specimen thickness is needed in both TKD and TEM in order to get sufficient signal from Kikuchi patterns. Therefore, the sample thickness must be optimized as a function of the material, and the use of electron transparent samples introduces additional complications in the specimen preparation procedure in comparison with EBSD. However, TKD presents an advantage versus TEM. In diffraction techniques in the TEM, diffraction patterns are generated from every grain in the sample traversed by the beam. When multiple grains overlap across the beam path, the signal coming from each grain may be impossible to deconvolve. Conversely, in TKD the Kikuchi patterns are generated from only the lowermost surface of the sample and therefore this technique copes better when overlapping grains are traversed by the beam. The accelerating voltage of the beam must be selected as a function of the sample’s mass-thickness as indicated by Rice et al. The minimum thickness that permits to generate a Kikuchi pattern is related to the mean free path, which is dependent on the beam voltage and the atomic number of the material. Sufficient TKD pattern signal for automated indexing has been reported for sample thickness spanning almost three orders of magnitude in different materials, from 5 nm of hafnium dioxide and palladium oxide, to 3 μm of aluminum, corresponding to a mass-thickness range of ~5–810 μg cm\(^{-2}\). Sample preparation via electro-polishing poses additional difficulty in the acquisition of the signal background as compared to foils prepared with FIB. This is due to the thickness variations often present in electro-polished foils, which limits the analysis to only those areas of the sample with homogeneous thickness. In contrast, samples prepared with FIB normally present lower thickness variations.

Keller and Geiss showed that the reduction of the beam-sample interaction volume in TKD versus standard EBSD is associated with a significant enhancement in performance. TKD can produce images with an absolute resolution below 10 nm, i.e., maps of domains where features of diameters down to 5–10 nm can be distinguished from the diffraction pattern quality map, providing significantly better spatial resolution than standard EBSD. TKD enables mapping of materials with grain sizes approximately in the 10–200 nm range. As mentioned in the previous section, the physical resolution can be defined as the distance from a large-angle grain boundary at which diffracted intensities from the two neighboring crystals are obtained. The effective resolution of TKD, as defined in Section 2.1, can reach values as low as 2 nm, depending on the atomic number of the sample. Hence, TKD allows for the routine characterization of nanocrystalline metals and alloys in the SEM, in terms of grain size, boundary, texture, and phase on a scale which had only been achievable by TEM before.

The use of TKD in metallurgy is currently undergoing a rapid expansion and it is predicted that many of the EBSD applications described in this report, where spatial resolution requirements are high, will benefit from the use of TKD. So far, an application area where TKD has been proven successful is for ultra-fine grain (UFG) metal alloys where it is often difficult to distinguish dislocation cell walls from high-angle grain boundaries in the TEM and where the EBSD spatial resolution is inadequate. One application example from this area is drawn from the work by Trimby et al. who, among other investigations, performed TKD measurements on a duplex stainless steel that was deformed via high-pressure torsion (HPT); see Figure 6. TKD provides a means to characterize high-angle and low-angle boundaries in an automated process. Figure 6a shows the TKD pattern quality map (Kikuchi band contrast), where the α-ferrite (bcc) regions are colored in red and the γ-austenite (fcc) regions, in blue. Figure 6b presents the orientation map, in which fine grains with sizes down to a few nanometers and with low angle boundaries are well resolved, confirming the capability of this technique to characterize highly deformed, nanocrystalline metals. TKD is in fact a suitable tool for the study of highly deformed specimens due to the reduction of the sample-beam interaction volume compared to EBSD. This makes it possible to achieve improved pattern quality because the amount of dislocations present in the interaction volume is reduced, increasing the probability of correct pattern indexing. Applications of TKD in steel research have recently included the characterization of phenomena such as phase transformations and cracking.
associated to stress corrosion, and the effect of mechanical attrition surface treatments\textsuperscript{125} in austenitic stainless steels, as well as the formation of nano-grained oxides in Fe-Cr-Al stainless steels.\textsuperscript{126} TKD has been used to study transformation-induced plasticity (TRIP) via stress-assisted martensite, and twinning-induced plasticity (TWIP) in ultrafine-grained multi-phase steels.\textsuperscript{127,128} Furthermore, this technique has been utilized in the characterization of complex microstructures such as those resulting from high plastic deformation in flow formed Cr-Mo-V steel, the nano-sized cementite formation in quenched and tempered steel, and in the examination of carbide-free bainitic steels.\textsuperscript{129–131}

The use of TKD in tandem with EDS provides a tool able to collect simultaneous chemical information with high resolution, effectively limited by the sample thickness as in STEM. Brodusch et al. first published the simultaneous acquisition of TKD and EDS maps in a single run, demonstrating the ability of TKD to identify phases and precipitates in compounds and alloys.\textsuperscript{116} It provides a useful combination of tools for correlating orientation data with chemical segregation. TKD cannot compete with high-resolution TEM or atom probe tomography (APT) in terms of resolution, but it has the dual advantage of statistical significance and automation, and it is a technique suitable for identifying relationships between chemistry and crystallography in submicron-scale structures.\textsuperscript{122} It can be noted that forescatter electron imaging of electron transparent samples has been discussed as a complement to TKD.\textsuperscript{132} Utilizing the forescatter detectors mounted on the EBSD provides efficient means to study crystallographic features in electron transparent samples in a similar way as ECCI can be used for bulk samples.

4. Electron channeling contrast imaging (ECCI)

When the surface of a crystalline bulk sample is irradiated with an electron beam, the intensity of the backscattered electrons depends on the angle between the beam and the lattice planes of the crystal, since the maxima of the probability density of electrons occur either on the diffracting lattice planes or in between them. Slight local distortions in the crystal lattice, i.e., lattice defects such as dislocations or twins, cause a modulation of the backscattered electron intensity with respect to the lattice, and therefore they produce changes in the contrast, allowing the defect to be imaged in the SEM directly from the collected electron backscattered signal. This is achieved by positioning the incident beam at the Bragg condition for the crystal away from the lattice defect. As the beam is brought close to the defect, the lattice plane tilting around the inhomogeneous area gives a local deviation from the Bragg angle, so the BSE intensity is locally altered producing contrast changes. This technique is known as electron channeling contrast imaging (ECCI). An electron channeling pattern (ECP) records the changes produced in the BSE intensity as the beam

\[ \text{Figure 6. Images from Trimby et al. 2014.}^{122} \text{ (a) TKD pattern quality map overlaid with a phase map, where the } \alpha\text{-ferrite (bcc) regions are colored in red and the } \gamma\text{-austenite (fcc) regions, in blue. (b) Orientation map of a HPT-deformed duplex stainless steel. (© Elsevier. Reprinted with permission from Trimby et al.}^{122}\text{ Permission to reuse must be obtained from the rightsholder.)} \]
incidence angle is varied. Comprehensive accounts on the physical principles and historical development of this technique are available in the literature, and a schematic description of the technique is provided by Wilkinson et al. (1997). Before the advent of high-resolution SEM, the direct characterization of defects in that scale had only been attainable with TEM due to resolution limitations. However, the aforementioned introduction of FEGs and the new generation of electron optics have resulted in electron beams with higher current density, smaller beam spot size and better convergence, enabling direct imaging of lattice defects near the surface by ECCI, which makes this technique a good complement to other crystallographic techniques in the SEM such as EBSD. An important development undergone by this technique in the last decades is the improvement of its setup configuration. Two different setup configurations have coexisted over the years, being common for some time to perform ECCI on a sample tilted at high angle and using a forward-scattered electron detector. At the beginning of the 1990s, for instance, ECCI was conducted with specimen tilt of $\sim 45^\circ$ with respect to the incident electron beam, with a side-mounted BSE detector, as shown in Figure 2c under high-tilt configuration. On the other hand, a low-tilt configuration, schematically described in Figure 2c, was used from the beginning of the 1980s, e.g., for applications in geo-sciences, and later by Simkin et al. (1999). Sample tilting angles below $10^\circ$ enabled the use of general-purpose pole piece-mounted backscatter detectors instead of specialized forward-scattered electron detectors, allowing access to more advantageous ECC imaging conditions. Moreover, the low-angle layout reduced the mechanical interferences between stage and detector, making the maneuvering of the sample easier and removing constraints in the use of in-situ testing apparatus. Since the overall BSE signal is lower in the low-tilt configuration, in order to ensure adequate performance, it is necessary to operate a beam with small convergence angle and sufficient probe current, and to use a BSE detector with high collection efficiency. This new configuration provided dislocation contrast characteristics comparable to those obtained by the high-tilt configuration. It is known that operational parameters can be adjusted to optimize BSE contrast. Works by Cazaux et al. and Aoyama et al. have highlighted the influence of the angle of collection and the acceleration voltage in the channeling contrast. For instance, studies by Aoyama et al. performed on cross-sections of heat-treated steel under various accelerating voltages and collection angles show that

![Figure 7](image_url)

**Figure 7.** Data from Tian et al. ECCI imaging of the deformation structure reduction in model ternary Fe-Cr-Ni alloys after 10% cold rolling. The deformation mechanism changes from stacking faults, hcp-martensite and bcc-martensite to twinning and slip with increasing Ni content. (a) Alloy 18Cr-10Ni (mass%) showing shear bands and bcc-martensite at shear bands, (b) magnification of (a) showing more clearly the stacking faults, hcp-martensite and lath bcc-martensite, (c) alloy 18Cr-12Ni (mass%) showing shear bands but no bcc-martensite, and (d) alloy 18Cr-14Ni (mass%) showing twinning and slip deformation but no clear formation of martensite.
low values of accelerating voltage and low collection angle result in an improvement of channeling contrast. The positive effect of lower acceleration voltage on the ECCI contrast is due to a reduction of both the beam-sample interaction volume, and the cross-section for phonon scattering, which leads to the improvement of diffraction condition and contrast.\textsuperscript{140} An area of research where this capability has become valuable is in the studies of deformation of steels containing the austenite phase. It is known that, depending on the stacking fault energy (SFE) of the steel, different deformation mechanisms will be preferred, and there is an intensive modeling activity in this field at present, where, e.g., first-principles calculations are used to predict the SFE and the operating deformation mechanism, i.e. the mechanical response of the alloy under certain conditions.\textsuperscript{141–144} Talonen and Hänninen presented a seminal work on the deformation of metastable austenitic stainless-steel prone to deformation-induced martensitic transformation and the TRIP effect.\textsuperscript{145} They used ECCI in combination with other characterization methods such as x-ray diffraction (XRD) to further understand the deformation of fcc metals with different SFE. This work also inspired other ECCI studies on metastable austenitic stainless steel model alloys (ternary Fe-Cr-Ni), where the effect of austenite stability on the active deformation mechanism was carefully described.\textsuperscript{146,147} Figure 7 is derived from that work and demonstrates the transition of deformation mechanism with increasing austenite stability, i.e., increasing Ni content and SFE. At low SFE, extended stacking faults, hcp-martensite and bcc-martensite formation are dominating; at intermediate SFE, the deformation occurs mainly via twinning; whereas slip is predominant at even higher SFE. That work shows the powerful combination of ECCI imaging with high-resolution and rapid image acquisition, coupled with phase and twin identification as well as micro-texture determination performed by EBSD.

A recent development of ECCI consists in the execution of this technique under well-controlled diffraction conditions, to obtain quantitative characterization of lattice defects with optimal contrast. ECC imaging of defects is obtained by orienting the crystal matrix exactly into Bragg condition. Until recently, ECCI had been combined with ECPs in order to control the diffraction conditions.\textsuperscript{148} This approach is limited to spatial resolutions above 2 μm, due to the requirement of a large final aperture to allow the beam to cover a large angular regime.\textsuperscript{148} A new setup to conduct ECCI under controlled diffraction conditions was proposed in 2009, in which EBSD is used instead of ECPs to orientate the crystal into optimal diffraction condition.\textsuperscript{140} This method allows the imaging of microstructures with the most favorable electron channeling contrast even in fine-grained and highly deformed materials because of the superior spatial resolution of EBSD, \textasciitilde 30–50 nm,\textsuperscript{56} with respect to ECP.

An example by Weidner et al. is provided to illustrate the visualization of defects in steels via ECCI in a high-resolution FEG-SEM. Figure 8 shows inverted ECC images of (a) a tensile deformed TRIP steel and (b) a cyclically deformed TRIP steel. In both images, individual stacking faults are clearly visible, as well as deformation bands along different activated slip systems.\textsuperscript{149} ECCI-EBSD is a powerful technique to determine dislocation densities in deformed bulk metals at a wide field of view in the SEM. The characterization of dislocations at optimum diffraction conditions enables the estimation of both GNDs and SSDs, as opposed to the most commonly used EBSD method which can only account for GNDs. As a result, this technique provides a much better estimate of dislocation density than EBSD alone, and comparable accuracy to that obtained by conventional bright-field TEM. This is a significant advance in microstructural characterization of deformed materials in the

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure8.png}
\caption{Example from Weidner et al. 2011.\textsuperscript{149} Inverted ECC images of (a) a tensile deformed TRIP steel and (b) a cyclically deformed TRIP steel. (© Carl Hanser Verlag GmbH & Co. Reprinted with permission from Weidner et al.\textsuperscript{149} Permission to reuse must be obtained from the rightsholder.)}
\end{figure}
SEM. Alternative techniques to measure lattice defects had traditionally relied on TEM. The development of ECCI has provided advantages over TEM, such as easier preparation of specimens, since the preparation of bulk samples is less time-consuming than the electron transparent foils used for TEM. ECCI enables the analysis of large areas with good statistics, and facilitates easier implementation of in-situ studies compared to TEM. An additional benefit of ECCI over TEM with regards to sample geometry is that in ECCI only one free surface needs to be exposed, as opposed to the two free surfaces required by TEM, which may cause significant relaxation of stresses.

5. Concluding remarks and outlook

This article reviews the complete set of tools for crystallographic analysis in the SEM, discussing the state-of-the-art of EBSD, TKD and ECCI and the complementary use of these techniques for analyses in the field of metallurgy.

Subsequent enhancements of SEM performance and several decades of developments have positioned EBSD as an indispensable characterization technique in the field of metallurgy. EBSD as a method for orientation measurements provides good angular resolution comparable to TEM-based techniques, but has better statistical significance, and it is more user-friendly, what makes this technique a preferred tool for phase characterization in metals. EBSD allows for the crystallographic mapping of features involving low misorientations, hence making it possible to characterize relevant metallurgic structures and phase changes such as martensitic and bainitic transformations. Recent developments include the use of the lattice parameter in order to distinguish among phases with similar crystallographic structure, and the introduction of novel methods for the characterization of orientation relationships in martensitic alloys, without the presence of the parent phase (austenite) as a reference. The use of EBSD coupled with EDS offers a suitable approach for the combined crystallographic and chemical characterization of metal alloys.

EBSD is an efficient tool to measure lattice strain and rotation at the sub-micron scale. This is achieved from the direct comparison of EBSPs by means of cross-correlation functions over ROIs, using as a reference EBSPs from areas of known state at the sample. The difficulty associated with finding a reference pattern in the sample can eventually be overcome with the introduction of pattern simulation and the use of dynamical diffraction theory.

The characterization of dislocations and other defects via EBSD is limited by the spatial resolution of this technique. Measurements of dislocation density can be obtained via EBSD by quantifying the misorientation between consecutive points, which reveals the lattice curvature and presence of GNDs. The advantages of this method are its high acquisition speed, data statistical significance and the simple specimen preparation associated to bulk analysis. However, the measurement is a lower bound of the actual dislocation density, since EBSD can only account for GNDs, neglecting other types of dislocations such as the SSDs, and because the entire dislocation tensors can only be calculated using 3-D techniques such as FIB/SEM-based 3D-EBSD. A valid alternative to measure dislocation density with EBSD is to sum the dislocation density around an enclosed area instead of measuring the strain at individual dislocations, which avoids spatial resolution limitations.

TKD is a technique for the forescattered electron diffraction analysis of thin-foil specimens. The small interaction volume between the sample and the electron beam enhances considerably the spatial resolution of this technique in comparison to EBSD. It is hence a very suitable substitute of EBSD for the crystallographic characterization of nano-structured materials, such as ultra-fine-grained metals. Furthermore, in combination with EDS, TKD allows the simultaneous structural and chemical analysis in the SEM of domains with grain sizes below 100 nm. Although TKD cannot compete with TEM in resolution, and its thin-foil sample preparation is equally time consuming, this technique presents clear advantages such as high acquisition speed and the widespread availability of SEM.

ECCI is an imaging method which relies on the back-scattered electron contrast produced by the interaction between an electron beam and a crystal lattice. This technique is very useful in metallurgy as a means to image lattice defects near the sample surface, which makes it a good complement to EBSD. ECCI has made significant progress after the recent introduction of a new methodology consisting in the execution of ECCI under controlled diffraction conditions, i.e., in combination with EBSD in order to find the relative position of beam and sample that yields a suitable image contrast. Relevant contributions of ECCI to metallography are the characterization of deformation in austenitic steels and the accurate visualization of defect in steels and other metals. ECCI is a suitable method for the measurement of dislocation density, accounting for both SSDs and GNDs, and presenting a significantly better resolution than EBSD, comparable to TEM, with the additional advantage of its simpler sample preparation requirements, as it is performed over bulk samples.

EBSD and ECCI are mature techniques, yet their use continues to expand both in research and industrial
applications, a trend that is expected to continue. The upcoming introduction of new materials for FEG sources, and advanced aberration correction technologies will likely enable the production of brighter electron sources and facilitate smaller probe volumes and better spatial resolutions, which will equally benefit EBSD, TKD and ECCI. In addition, the possibility of higher analysis speeds, and the increasing availability of dual-beam (FIB/SEM and plasma-FIB/SEM) and tri-beam analysis speeds, and the increasing availability of dual-beam (FIB/SEM and plasma-FIB/SEM) and tri-beam microscopes (FIB/SEM/Laser) with the capability of removing material at high rates, will contribute to expand the potential of 3D-EBSD characterization. During its short life, the advantages of TKD versus EBSD in terms of spatial resolution have been widely demonstrated, although it remains to be proven whether it will become a tool of common use. This technique is undergoing a rapid technical development and its potential, in combination with EDS, to provide simultaneous crystallographic, morphologic, and chemical characterizations will definitely be a valuable asset for metallurgists.

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