New techniques for imaging and identifying defects in electron microscopy

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Defects in crystalline materials control the properties of engineered and natural materials, and their characterization focuses our strategies to optimize performance. Electron microscopy has served as the backbone of our understanding of defect structure and their interactions owing to beneficial spatial resolution and contrast mechanisms that enable direct imaging of defects. These defects reside in complex microstructures and chemical environments, demanding a combination of experimental approaches for full defect characterization. In this article, we describe recent progress and trends in methods for examining defects using scanning electron microscopy platforms, where several emerging approaches offer attractive benefits, for instance in correlative microscopy across length scales and in situ studies of defect dynamics.

Keywords: defects, scanning electron microscopy (SEM), scanning transmission electron microscopy (STEM), crystal
Introduction

The paradigm underpinning materials science and engineering that structure controls properties and performance is determined by the governing role of defects in mediating properties ranging from mechanical strength and damage tolerance (1–3) to optoelectronic response (4) to phase transformation phenomena (5). Direct experimental characterization of crystalline defects dates back to the invention of electron microscopy (6), yet the past decade has witnessed tremendous advances in new electron imaging and diffraction-based modalities for quantifying defects and their corresponding ensembles, interactions with other microstructural features, and dynamics. In the case of extended defects such as dislocations and planar faults, pioneering developments have taken an orthogonal tack from the race for spatial resolution in electron microscopy, and instead have targeted correlative characterization techniques, temporal resolution to capture dynamics in situ, and statistical quantification of defect evolution and organization. This is required to deploy modern materials for emerging technologies such as additive manufacturing and optoelectronics, and those used in extreme environments, where a toolbox of materials characterization probes is necessary to advance our understanding of the links between defects and materials properties.

This article focuses on methods for identifying and quantifying defects that are amenable to scanning electron microscopy platforms, which offer versatility for multi-modal and in situ characterization, as well as differences in scattering physics owing to different primary electron beam energies. We highlight recent advances and trends in defect imaging and characterization using electron backscatter diffraction, electron channeling contrast imaging, and diffraction-contrast scanning transmission electron microscopy approaches. We conclude by assessing the role of the emerging interplay between multimodal microscopy and data science on our understanding of defect-property relationships in advanced materials.
Defect characterization using electron backscatter diffraction (EBSD)

Progressive development of EBSD has increased in sophistication and presently provides fast automated indexing of electron diffraction patterns in the SEM (for more information, see (7) (8) (9) and (10)). For defect analysis, this has been augmented by recent developments of the HR-EBSD method initially advanced by Wilkinson and colleagues (11, 12) which uses direct cross correlation of EBSD patterns which enables a resolution of better than $10^{-4}$ in (relative, deviatoric) elastic strain and $10^{-4}$ rads in (relative) lattice rotation. For metallic structures, recent advances have included pattern remapping (13, 14) which has improved the robustness of elastic strain measurements in metals. For all these EBSD techniques, the scanning nature of the electron beam in the SEM and automated analysis of very large maps (often $>>10k$ mapped points) at a range of length scales enables rich microstructure maps to be generated (for reviews on the EBSD technique see (15, 16)). The information within each measured EBSD map is rich, and the information (e.g. grain shape, orientation) can be correlated together providing insight into crystal orientation, grain boundary network. These data can be linked with other imaging modes such as backscatter and secondary electron imaging, as well as AFM (17) and Raman microscopy (18) to provide correlative approaches to understand the lattice state in materials. Through applying Nye’s analysis (19–22), and afforded by the increased precision of the HR-EBSD approach (23), it is now routine to assess the storage of so called “geometrically necessary dislocations” (GNDs) which give rise to lattice curvature; furthermore, a statistical treatment of the variation in lattice shear stress distributions can be used to assess the “statistically stored dislocations” (SSDs) (24, 25), which are related to closely bound dislocations that do not give rise to lattice curvature e.g. dislocation dipoles and multipoles. Mapping of GND content is a popular use of the EBSD method and these quantitative maps can span a range of dislocation densities ($1x10^{12}$ to $~5x10^{15}$ dislocations per m$^2$) and length scales (from $~2$ nm to $10$ μm step size) (26) which are often difficult to assess with other methods. Understanding of defects with
HR-EBSD has proven popular in metals (27–30), semiconductors (31, 32), ceramics (33), and geological materials (34–36).

The HR-EBSD and EBSD techniques provide quantitative assessment of the state of the crystal lattice and therefore they lend themselves towards direct quantitative linking with materials models, such as crystal plasticity finite element modelling. For HR-EBSD, the technique can only measure relative variations in lattice strain within each grain, which can be related to the relative stress through Hooke’s law. Nevertheless, even these types of residual stresses, caused by the presence of lattice defects, are important to understand the nature of the lattice strain state during deformation (37, 38) or that of thin film crystal growth (12).

EBSD continues to evolve as a technique, most notably with ever faster detectors (modern CMOS based detectors can capture patterns at >2000 Hz) and the emergence of direct detectors (39–41), which provides high angular information within the patterns themselves, thus offering the potential for direct quantitative comparison with high quality dynamical simulations (42). This has the potential to unlock further information about the structure and nature of defects, as it may be possible to correlate the selective blurring of different diffraction bands with the nature of the dislocation structures within the interaction volume (43). This approach is analogous to line broadening approaches used commonly within the X-ray community (44) using advances in pattern analysis (45, 46), but note that dynamical scattering and diffraction effects and the detector physics would have to be considered.

At a smaller length scale, transmission Kikuchi diffraction (TKD) (47) has opened up STEM based diffraction within the SEM, where wide angle (i.e. short camera length as compared to the TEM) Kikuchi patterns are now captured routinely. These wide-angle patterns can be indexed using conventional EBSD analysis methods, and thus the microstructure of nano-crystalline materials can be now unlocked (48, 49). Advances in this area are likely to involve dynamic testing using in situ methods, including heating to observe nano-grain growth (50), indentation-based testing similar to the TEM (51), but with fast STEM based quantification of the associated diffraction patterns, and thus unlocking the
identification of stress induced phase transformations and crystallographic reorientation via mechanisms such as twinning. This will take in situ EBSD and mechanical down a length scale further beyond micro-cantilever (52) and micropillar testing (53–55).

**Electron channeling contrast imaging (ECCI) of defects**

Electron channeling contrast imaging (ECCI) is an SEM-based imaging technique by which extended defects, i.e. dislocations, stacking faults, grain boundaries, nano-twins and precipitates and elastic strain fields in crystalline matter can be observed directly on bulk samples through backscattered electrons (BSE). ECCI is not a new technique; EC contrast was observed and correctly interpreted in the 1960s by Coates (56). Its features and potential applications were treated in detail by Joy et al. (57). Nevertheless, the technique remained exotic because of shortcomings of the available microscopes. Only with the advent of SEMs with high-brightness thermal field emission guns, parallel illumination, sensitive backscatter detectors and versatile stages, the technique developed into a useful and competitive technique. Many research groups have since contributed to the better understanding and application of this technique, e.g. (58) on dislocation imaging, (59) on Burgers vector analysis, (60–63) on simulation of dislocation images and understanding of contrast formation and ourselves on the combination of EBSD with ECCI for accurate diffraction condition determination (64).

The contrast formation principles for ECCI are very much the same as in bright field scanning transmission electron microscopy (STEM) with the important difference that one does not directly observe the diffracted electron intensity but rather the backscattered intensity which is modulated by the scattering and diffraction as the beam enters the bulk crystalline sample and subsequently escapes. As a consequence, the contrast is weaker and inverted compared to STEM. Furthermore, as the signal stems from a bulk sample (and not from a thin foil as in STEM) a blurring background signal arises from those electrons which, due to inelastic scattering processes, have left the original
coherent electron wave field. The spatial resolution of ECCI is limited mainly by scan control, the width of the primary electron beam, and by its broadening inside of the sample. At optimum microscope conditions the lateral resolution is currently on the order of 8 nm (64). Depending on the electron imaging and sample conditions (mass and defect density) the signal originates approximately from the first 100 nm below the surface (64).

Figure 2(a) shows an example of an ECC image, taken from a lightly deformed high entropy alloy showing a high density of dislocations mainly arranged in planar slip bands. Some dislocations appear in conjunction with stacking faults, demonstrating a relatively low stacking fault energy; others appear in pairs likely indicating short-range ordering. Using the same principles as conventional diffraction-based TEM the crystallographic character of the defects, e.g. Burgers vector and line direction of a dislocation, can be quantified in many cases. In order to do so, however, a tool is required to accurately determine the crystal orientation and the active diffraction conditions. This can be done via EBSD (64, 65), electron channeling patterns (66) or interpretation of contrast variation for multiple channeling contrast images (67). The EBSD-based approach is depicted in Figure 2(b): a sample is placed in EBSD position (1) to determine the crystal orientation of a desired grain (2). The sample is then moved close to the BSE detector and is tilted to the approximate channeling conditions using the knowledge on crystal orientation; the sample tilt and rotation is then slightly adjusted to maximize contrast (3). By scanning over the sample an ECC image is obtained (4).

ECCI is advantageous as it works with bulk samples, rather than thin foils thus reducing bending and strain relaxation. Therefore, defect evolution can be observed with in situ or quasi in situ experiments with improved boundary conditions, for example during deformation, annealing or chemical modification. Furthermore it can be used on very large (up to cm²) improving statistical treatments. This often overcomes issues with contrast and resolution, as compared to complementary TEM and STEM based defect analysis.
The powerful features of ECCI are illustrated in Figure 2(c-e) with an example from a study on hydrogen-dislocation interaction in a high-Mn austenitic steel sample: Figure 2(d) displays the ECC image of the dislocation field formed around a nano-indent (indenter sphere diameter 1 µm, indentation depth 100 nm). The current diffraction conditions are displayed by the active diffraction vector, \( g \). Individual dislocations are visible immediately outside of the indentation area, approximately 200 nm from the indenter center. Different slip systems are visible, reaching the surface at different distances from below the indent. The traces of the \{111\} slip planes, obtained through EBSD orientation measurements, are displayed in the figure as well. The fact that the indent is observed on a bulk sample makes it possible to observe many more of these indents, obtained from different grains and at different locations, as it is shown in Figure 2(c). From the dislocation fields obtained for each grain it was possible to extract, with high statistical significance, which features were systematic and which ones stochastic. In a subsequent step the sample was lightly polished to remove the indents but conserving the grains; it was then electrolytically charged with hydrogen and the same grains indented and observed again. One resulting indent is shown in Figure 2(e). Here, dislocations with extended stacking faults reach much further out than in the uncharged sample, indicating a reduction in stacking fault energy by hydrogen and an increase of dislocation density as proposed by the hydrogen enhanced local plasticity (HELP) mechanism (68).

**Diffraction-contrast scanning transmission electron microscopy**

Some of the earliest work on TEM included the imaging of crystalline defects (69), providing experimental corroboration of the theory of defects in materials. Today, TEM is often the quintessential mode of direct imaging and characterization of individual crystalline defects. Routine diffraction-contrast TEM is based on local deviations from the Bragg condition owing to defect-induced displacement fields. Conventional TEM (CTEM) approaches using parallel beam illumination allow for not only imaging of defects, but characterization of both Burgers and line vectors from dislocations as inferred
from their shape in the image and from invisibility conditions in diffraction; furthermore the displacement vectors from planar faults can be characterized. Imaging of defects using higher order diffraction vectors, known as weak-beam TEM (70), facilitates studies of the fine structure of dislocations owing to contrast that is localized near the defect, but requires long exposure times that demand stable and drift-free conditions.

Recently, diffraction-contrast STEM has been rekindled as a promising approach to characterize crystalline defects (71–74), in part thanks to the advent of high-quality electron sources. The primary advantages of STEM-based defect studies in which a convergent focused probe is used, in comparison to CTEM approaches, include the suppression of auxiliary contrast features such as bending contours and thickness fringes (Figure 3(a,b)), reductions in extinction contrast from inclined dislocations, and the ability to image defects in relatively thick specimens owing to the mitigation of chromatic aberrations arising from post-specimen lenses needed in CTEM. These collective benefits offer exciting opportunities for semi-automated quantification of defect densities because of more uniform contrast (75). Recent studies have shown that dislocation invisibility is still applicable in STEM for Burgers vector determination, and moreover that weak-beam conditions are achievable albeit with high signal-to-noise ratios enabled by annular STEM detectors where the signal is integrated over a given acceptance angle (72). Taken as a whole, the advantages of STEM diffraction-contrast defect imaging additionally lend themselves very well to in situ experiments and tomography, making it the emerging tool of choice in modern defect-property studies.

Parallel developments in modern solid-state STEM have enabled integration with commercial SEM platforms (Figure 3(c)), augmenting their conventional functionality. In such transmission modalities, the lower primary beam energies from SEM sources (<30 kV) are appealing in applications that suffer from low contrast in weakly scattering objects and knock-on beam damage (76). These conditions favor studies of organic and low atomic number materials, which indeed have been the primary focus of so-called STEM-in-SEM (77), or
transmission SEM (TSEM) (78, 79), techniques. Pioneering studies showed the promise of TSEM at 30 kV and below in a large-chamber SEM environment, which date several decades ago (80–82). Approaches reported since have emphasized the need to control the acceptance angles used for image formation since camera lengths must be set physically (such as through the use of pre-machined aperture masks (77, 83) and a digital micromirror device (84)). Modern segmented detectors available for TSEM offer a range of annuli with controlled acceptance angles (and even azimuthal segmentation) and even high-angle annular regions for mass-thickness dark field imaging. The reduction in price and improvement in quality of direct electron detectors is likely to assist further in improving TSEM.

The utility of TSEM extends well to the imaging and characterization of crystalline defects as was demonstrated in earlier work (85–87), with recent studies advancing this capability and understanding. Diffraction-contrast images of dislocations and stacking faults in semiconductors (88) and metallic materials (78) reproduce the distinct advantages of a convergent scanning probe and also offer practical benefits of providing statistical studies of large areas enabling high-throughput studies since many specimens can be incorporated in the large vacuum chambers. The incorporation of more sophisticated instrumentation for in situ studies of defect dynamics and evolution than cannot fit in the small volume of a TEM holder is a notable advantage. The multimodal detection schemes offered in a well-equipped modern SEM (such as EBSD, EDS, CL, ECCI, back-scattered detectors) pair nicely with TSEM and facilitate exciting simultaneous correlative studies. One unique aspect of TSEM for defect imaging arising from the low primary beam energies is that defect contrast is strongly localized compared with the electron energies used for TEM and STEM (~200-300 kV). As studied by Callahan et al., TSEM conditions show image qualities for dislocations and stacking faults that are reminiscent of weak-beam TEM conditions, although they are formed from strong beams providing very good signal-to-noise ratios (78) (Figure 3(d-f)). This can be rationalized by considering that the image width
of a defect is proportional to the effective extinction distance for a given diffraction vector, defined as

$$\xi_{g}^{eff} = \frac{\xi_{g}}{(1 + s_{g}^{2} \xi_{g}^{2})^{1/2}},$$

where $s_{g}$ is the deviation parameter. Weak beam TEM narrows the defect contrast by increasing $s_{g}$, whereas TSEM at 30 kV provides an approximately 3x decrease of the extinction distance $\xi_{g}$ compared with 200 kV TEM, which results in narrow defect widths even in the case of strong beams (i.e. $s_{g} = 0$). In addition, a smaller Ewald sphere at 30 kV (and below) implies that the deviation parameter increases more rapidly as one moves away from the Bragg condition, suggesting that the strain field near a dislocation will provide more localized contrast (Figure 3(g,h)). These collective features make defect observations using TSEM amenable to materials that possess high dislocation densities or where fine structure needs to be resolved (e.g. dissociated dislocations), as well as where defect-obstacle interactions are of interest (89). The field of TSEM as applied to characterization of defects is nascent and offers exciting practical and fundamental benefits for materials research. Advancements in this vein are necessary, such as in navigating reciprocal space to specific diffraction conditions by making use of on-axis cameras providing diffraction patterns (88), as well as in advanced positioning systems analogous to those found in advanced X-ray synchrotron beamlines.

We offer the following comparison of the methods described above for defect assessment, as summarized in Table 1. Taken as a whole, both ECCI and EBSD offer the distinct advantage of being applicable to the surface of bulk materials and over large imaging areas, in comparison to CTEM analysis. EBSD provides quantitative insight owing to the collection of spatially-mapped diffraction patterns (encoding crystallographic phase, lattice strains, orientations, and rotation gradients), although drawing links to dislocation configurations and arrangements requires thoughtful inference and often recourse to models. By comparison, ECCI provides a direct means of imaging and characterizing defects,
although the contrast and spatial resolution is generally poorer than the CTEM counterparts because of differences in interaction volumes. If higher spatial resolutions are needed, then transmission imaging and diffraction modalities applied to thin specimens, such as diffraction-contrast STEM (and the SEM-based version TSEM) and TKD fulfill these needs, provided the specimen preparation challenges can be overcome and any thin film effects on defect structure can be reconciled. The use of lower incident electron energies (e.g. 30 kV) used in TSEM compared with CTEM results in larger electron probe sizes, yet offers very narrow dislocation image widths with high signal-to-noise ratio, which would be beneficial in instances where dislocation densities are high or dislocation-obstacle interactions are difficult to discern. In all cases, the practical benefits of SEM environments such as ease-of-use and large chamber sizes conducive to sample throughput or *in situ* instrumentation often outweigh the fundamental limitations of the ‘simpler’ electron microscope.

Table 1. Order of magnitude comparison of defect characterization approaches.

|                    | ECCI  | EBSD | TKD  | CTEM | STEM |
|--------------------|-------|------|------|------|------|
| Lateral resolution | 10 nm | 20…500 nm 1) | 10 nm 1) | 1 nm | 0.1 nm |
| Depth of observation | 50…100 nm 1) | 10…30 nm 1) | 10 nm 1) | 100…200 nm | 20…50 nm |
| Observable area    | $10^8 \mu m^2$ | $10^8 \mu m^2$ | $10^4 \mu m^2$ | $10^4 \mu m^2$ | $10^3 \mu m^2$ |
| Sample type        | bulk | bulk | thin foil | thin foil | thin foil |

1) Depending on atomic number of sample and acceleration voltage

**Advances in algorithms and data science: toward predictive defect-property relationships**  
The quality of the information now obtained with state-of-the-art microscopy is high contrast and information rich. Each microscope image contains information which is a convolution of microscope conditions (e.g. beam convergence angle, scanning directions, detector contrast) and the beam-material
interactions (i.e. signal modulation due how the electron interacts with the sample and the signal escapes). When diffraction patterns (and spectroscopic information) are obtained at each mapped point, the richness of the information increases further. In Figure 4, we highlight examples of further insights from correlative microscopy (multi modal, including chemistry and structure) and *in situ* microscopy (providing time) approaches. The volume and complexity of these approaches automatically lend themselves to applications of “big data” and “machine learning” approaches. In gathering data, the SEM community has been ahead of many in this regard, as automation has been key in handling and reducing the data obtained (e.g. with automated indexing of diffraction patterns) to provide easy ways to interpret micrographs and provide immediate and direct interpretation.

Deterministic analysis of SEM data is routine, *i.e. where the data reduction strategy is known a priori*, as most SEM experiments are established with a good idea of how to optimize contrast to see particular features. For instance, EBSD data reduction and analysis is made easier as new Open Source toolboxes are released that simplify and improve the common handing of typically operations, ranging from the ‘trivial’ tasks of plotting of mapped data and the rendering of grain boundary structures networks in 2D and 3D (as performed in MTEX (90) for 2D and 3D EBSD, and 3D within Dream3D (91)). Quantitative handling and data processing (e.g. indexing with the Hough/Radon transform) of EBSD diffraction patterns is now afforded in AstroEBSD (92) and this is enhanced with the establishment of a translatable description of the frames of reference used (93).

Forward modelling is increasingly *used as greater computation power and numerical approximations makes solving of complicated electron-matter physics interactions reasonable, which makes fitting of these models tractable*. For electron modalities including electron channeling and diffraction can be performed using EMSSoft (94), which has widened the opportunity to perform high quality pattern matching based indexing of electron channeling patterns (ECPs) and EBSD. Advances in this area will likely include the use of forward models to
provide, for instance, the generation of physics based templates for matching different dislocation types that thread the surface of bulk semiconductors (95) and improve the robustness to image noise thus realizing automated defect analysis (96) in industrial processes.

Statistical (often information blind) approaches are being applied as numerical tools and scripts are increasingly common. These approaches tend to provide inferred correlation but rarely causation. For defect analysis can be built using tools such as multivariant statistical analysis (MSA) and principal component analysis (PCA) to improve signal to noise and enable common microstructural features to be identified with EBSD and TKD (97), and future applications in the SEM community will likely build in concert with developments of HyperSpy (98). Taken as a whole, the most promising avenues for advanced quantitative and predictive defect-property relationships view experimental innovations and data science not only as parallel tracks, but inextricably intertwined.

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**Stefan Zaefferer** studied physical metallurgy and metal physics at the TU Clausthal in Germany where he also obtained his PhD degree. During PhD and post doc times in Paris and in Kyoto he developed the computer program TOCA for on-line indexing of TEM and SEM diffraction patterns and microscope control. Since 2000 he is head of the research group „Microscopy and Diffraction“ at the Max-Planck-Institut für Eisenforschung (MPIE) and university lecturer at the RWTH Aachen as well as guest professor at various universities. His research interests span the development of tools for electron microscopy and investigation of microstructure mechanisms in various structural and functional materials.
Figures

Figure 1. EBSD measurements of defects and defect properties, including: High Angular-EBSD stress near a dislocation pile up and a demonstration of the Eshelby Frank and Nabarro prediction that the stress decays as $distance^{-1/2}$. Reprinted from (99) with permission from Elsevier; a demonstration that HR-EBSD provides increased resolution to map the accumulation of dislocations with respect to microstructural features (shown here in copper) (23); and High Angular Resolution TKD showing mapping of the stress and rotation fields around a single dislocation (100).
Figure 2. ECC imaging of crystalline defects. (a) ECC image of a lightly deformed high entropy alloy, showing planar bands of dislocations. (b) EBSD-assisted ECCI to determine crystallographic orientation and corresponding diffraction conditions. (c-e) Hydrogen-dislocation interactions in a high-Mn austenitic steel sample, with ECCI imaging of dislocations in the vicinity of an indentation (d) prior to and (e) following H-charging.
Figure 3. Imaging of crystalline defects using a variety of diffraction-contrast electron microscopy. (a,b) A comparison of faulted Ni-based superalloy in a severely bent foil imaged using (a) CTEM and (b) STEM at 200 kV, demonstrating the muting of bending contours via STEM imaging. Reprinted by permission of the publisher (Taylor & Francis Ltd, http://www.tandfonline.com) (101). (c) Schematic of TSEM (or STEM-in-SEM) imaging conditions at 30 kV using a transmission detector in an SEM. (d-f) Comparison of a Co-based superalloy following high temperature creep deformation and possessing stacking faults and dislocations, as imaged using (d) 30 kV TSEM, (e) 200 kV CTEM, and (f) 200 kV weak-beam (WB) CTEM. Note the similarity in fringe density within the stacking faults for TSEM and WB images. Reprinted from (78) with permission from Elsevier. (g,h) Dynamical diffraction simulations showing integrated intensities across edge dislocations in Co for (g) 30 kV and (h) 200 kV STEM probes, demonstrating the localization of dislocation contrast at low primary beam energies owing to the smaller extinction distances. Simulations courtesy of P. Callahan.
Figure 4. Applications of modern defect imaging and diffraction modes to in situ observations and correlative microscopy. Examples of in situ results (left panel) are shown for EBSD (reprinted from (52) with permission from Elsevier), ECCI (reprinted figure with permission from (102). Copyright (2019) by the American Physical Society), and TSEM (reprinted from (103) with permission from Elsevier). A highlight of correlative microscopy (right panel) demonstrates the use of ECCI to identify defects for site-specific investigations, including S/STEM and atom probe tomography (APT) methods to characterize structural and chemical details, respectively, at a superlattice intrinsic stacking fault in a CoNi-based superalloy (104).