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REVIEW

# Applications of electron backscatter diffraction to materials science: status in 2009

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**Abstract** Over the last two decades electron backscatter diffraction (EBSD) in the scanning electron microscope has become a powerful tool for the characterisation of crystalline materials. Via an in-depth analysis of published work in 2008 compared with 2003, this article captures the present contribution that EBSD is making to materials science. From the analysis it is shown that some aspects of EBSD application have increased greatly in recent years, particularly the range of materials analysed, microtexture determination, general microstructure characterisation, application to interfaces and combinations of EBSD with other applications such as modelling or materials testing. On the other hand some other applications of EBSD are still emerging, such as macrotexture determination, true phase identification and three-dimensional EBSD.

## Introduction

Electron backscatter diffraction (EBSD) in the scanning electron microscope (SEM) is a powerful experimental tool for materials scientists, physicists, geologists, and others, which can be applied in principle to any crystalline material. Over the last two decades EBSD has revolutionised the characterisation of such materials. Following commercialisation of the product at an early stage, EBSD has steadily attracted interest in SEM user communities.

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The principles of EBSD are expounded in detail in several texts, and so will not be described in any detail here [1–4] (http://www.ebsd.com). Briefly, the technique is based on acquisition and analysis of diffraction patterns from the flat surface of a specimen. To obtain an EBSD diffraction pattern a stationary beam of electrons is sited on the specimen in an SEM. Backscattered electrons diffract at crystal lattice planes within the probe volume, according to Bragg's law. The fraction of diffracted backscattered electrons which are able to escape from the specimen surface is maximised by tilting the specimen so that it makes a small angle with the incoming electron beam. The diffracted signal is collected on a phosphor screen and viewed with a low-light video camera. These diffraction patterns provide crystallographic information which can be related back to the position of origin on the specimen. Evaluation and indexing of the diffraction patterns is output in a variety of both statistical and pictorial formats. The most comprehensive of these outputs is the 'orientation map', which is a quantitative depiction of a region of microstructure in terms of its crystallographic constituents.

The first real-time EBSD systems were dedicated to measurement of local orientations, i.e. 'microtexture'. Automation of diffraction pattern indexing enabled orientation mapping, and hence marked the beginning of the shift towards EBSD becoming a truly comprehensive characterisation tool. A few years ago the advent of EBSD in the field emission gun SEM (FEGSEM) heralded an era of improved spatial resolution, now down to the nanometre range [5]. More recently fast mapping has greatly improved the efficiency of EBSD mapping investigations, and has allowed certain in situ studies. At the time of writing manufacturers quote indexing rates of 400-750 patterns per second (http://www.oxford-instruments.com/products/microanalysis/ebsd/Pages/ebsd.aspx, http://www.edax.com/products/TSL.cfm).

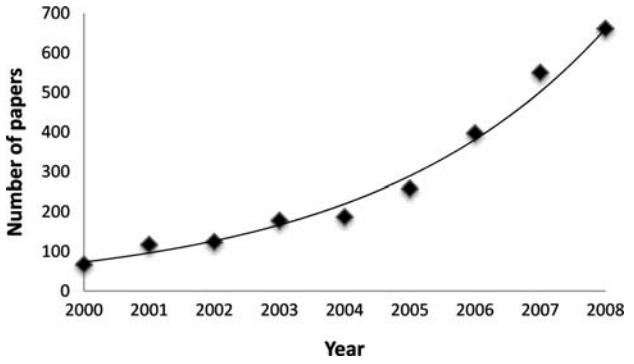


**Fig. 1** Graph showing exponential growth in number of EBSD-related publications in the period 2000–2008. (Source: ScienceDirect)

There is also, currently developments in 'three-dimensional EBSD', mainly via Dual-Beam focussed ion beam (FIB) microscopy [6].

Nowadays EBSD is used for determination of orientations and orientation distributions (texture), general microstructure characterisation and quantification, classification of interface crystallography, phase discrimination and identification and strain assessment [4]. Figure 1 is a graph showing the exponential growth of EBSD-related papers in the scientific literature since 2000, based on output from the search engine ScienceDirect (ScienceDirect® is a registered trademark of Elsevier B.V.). The absolute number of EBSD-related publications will be more per year than that quoted on Fig. 1. However, the majority of important journal publications are captured and the growth trend in EBSD-related output is depicted accurately.

The objective of this article is to document and to discuss the current status of EBSD applications. The discourse is based mainly on the survey of the scientific literature relating to EBSD in 2003 and 2008. The database from 2008 is analysed in some detail, with pertinent examples quoted.

## Material types

Figure 2a shows the output of EBSD-related publications in 2003 and 2008 categorised according to material type, namely metals and alloys where one phase predominates, ceramics, composites, electronic materials, geological materials, superconductors and other materials such as intermetallics. There is also a category where the main focus of the publication is development of EBSD technology or underlying theory rather than application to specific materials. The database for 2003 contains 167 papers and that for 2008 contains 601 papers, i.e. there has been a four-fold increase in output over 5 years. Figure 2b and c show the data as pie charts for 2003 and 2008, respectively, in order to highlight shifts in EBSD application to material types over the time period, rather than absolute numbers of publications.

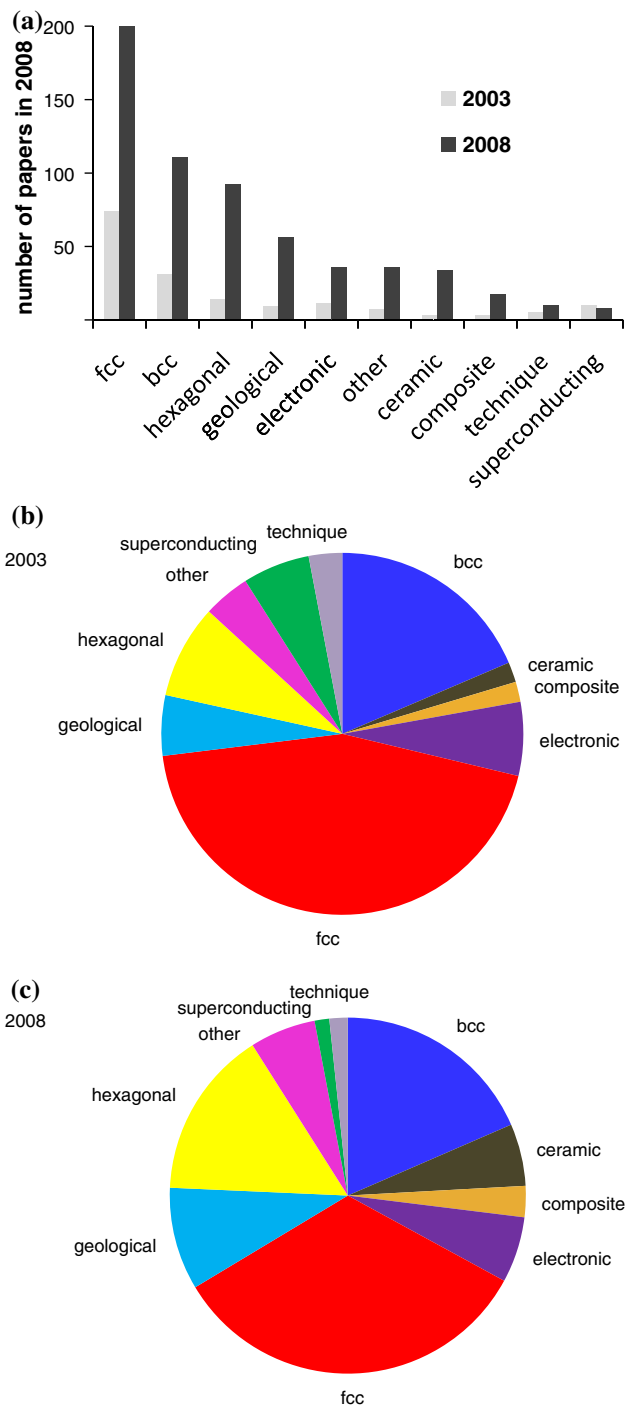


**Fig. 2** **a** Histogram showing output of EBSD-related publications in 2003 and 2008 categorised according to material type. **b** Pie chart showing output of EBSD-related publications in 2003 categorised according to material type. **c** Pie chart showing output of EBSD-related publications in 2008 categorised according to material type

By far the biggest application of EBSD has always been to metallic materials; in both 2003 and 2008 three-quarters of publications which include data obtained by EBSD related to metals. In part the emphasis on metals reflects the relatively simple crystal structures involved and the ease of specimen preparation, in most cases, compared to the other categories in Fig. 2. The application of EBSD to nominally single-phase metals and alloys is straightforward and data on microtexture and microstructure can be obtained in a routine manner. Furthermore, study of texture development is germane to metals processing. The metals category is further subdivided into face-centred cubic (fcc), body-centred cubic (bcc) and hexagonal (hex). In 2003 the emphasis was on fcc metals (Fig. 2b), as a legacy from early EBSD systems which could only solve diffraction patterns from fcc metals. By 2008 the ratio of metal types is biased more towards hexagonal metals and less towards fcc metals (Fig. 2c).

Figure 3 shows a breakdown of numbers of EBSD-related papers in 2008 according to specific metals and their alloys. By far the most popular metallic system for EBSD investigation is aluminium. Other well populated alloys are steels and those based on copper, nickel, magnesium and titanium. The total range of alloys investigated covers practically every metal; one investigation has been reported on each of manganese, tantalum, vanadium, silver, beryllium, zinc and lead (which is included in the 'Other fcc' category). These statistics demonstrate the feasibility of EBSD application to a wide range of metals. However, the statistics in Fig. 3 (and indeed in Fig. 2) should be viewed in the light of normalisation by the amount of total research or usage related to each of these metals.

After metals, Fig. 2 shows that the next largest category of publications where EBSD features is geological materials. The relative proportion has increased since 2003. This is noteworthy because it reflects that recent advances of EBSD in resolution, phase identification and multiphase mapping [4] are enablers in the analysis of geological materials, because they are often complex in terms both of the phases present and their morphology. In the context of the remit of this review, the publications on geological materials will not be discussed any further.

The electronic materials category includes research on materials such as silicon (including solar cells), gallium arsenide, gallium nitride, germanium, diamond and copper. The proportion of EBSD papers is similar in 2003 and 2008. There were 10 or less papers on superconducting materials in both years. In both of these material categories there is often emphasis on the development of specific property-related textures and their relationship to the microstructure. For example EBSD has been used to investigate melt-textured YBCO with embedded Ag-2411 nanoparticles [7].

By far the largest proportion increase between 2003 and 2008 has been application of EBSD to ceramics materials, for example WC–Co alloys [8] and $Al_2O_3$ [9]. Quantification of microstructure appears frequently as an EBSD analysis output, for example to grain size distribution ("General microstructure characterisation and quantification" section). Because of their non-conducting nature

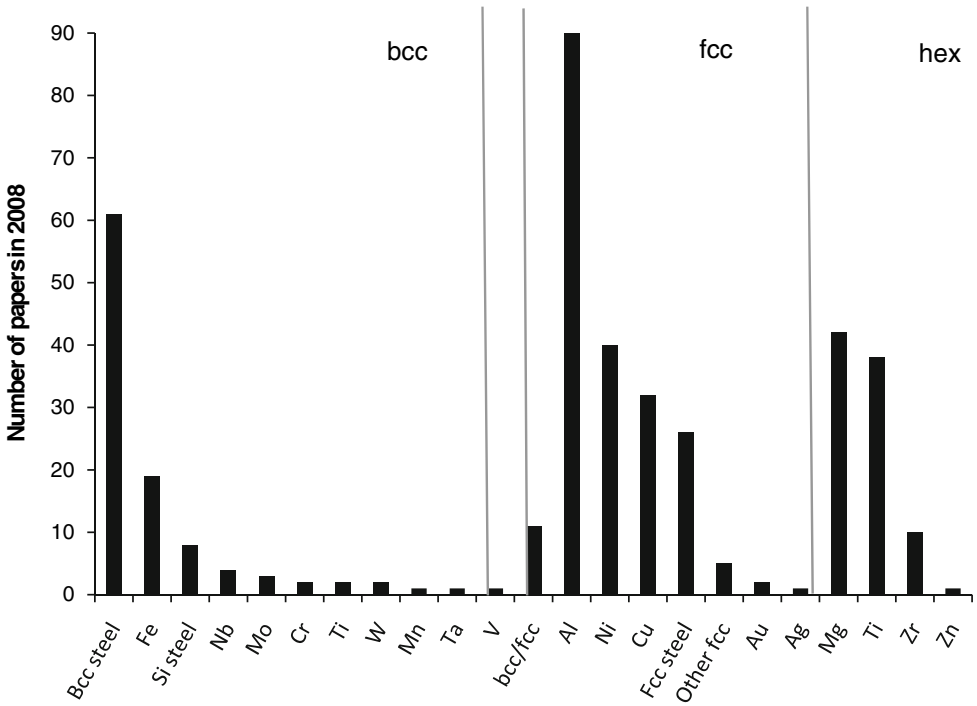


**Fig. 3** Breakdown of number of EBSD-related papers in 2008 according to specific metals and their alloys

there are usually more specimen preparation and data acquisition challenges for ceramics than some other materials classes such as metals. However, it seems from the increase in output that specimen preparation issues are not an insurmountable drawback to EBSD analysis. Composite materials also present a specimen preparation challenge. Despite this, EBSD has been carried out on phases in some composites, for example spark plasma sintered Al-15 at.% $MgB_2$ composites [10].

Materials which have been placed into the 'other' category include intermetallics such as titanium aluminide [e.g. 11], magnetic materials [e.g. 12], multiphase materials [13] and uncommon materials such as uranium [14]. Finally in the list of EBSD papers categorised by material, there are a few articles which are not material-specific but rather address aspects of EBSD technology and theory [e.g. 15].

The vast majority of publications report EBSD on polycrystalline materials having grain size in the micrometre range. A very small number of publications refer to single crystals [e.g. 16]. The increased popularity in recent years of FEGSEMs, which give a three-fold increase in resolution [5], has extended the range of materials which are amenable to EBSD examination to those that are fine-grained or nanocrystalline. In 2008, 11% of papers in the sample population discussed here related to fine-grained or nanocrystalline materials. Similarly, the fine probe size associated with the FEGSEM has facilitated more EBSD examination of deformed materials, namely 9% of papers in 2008.

## Recent applications of EBSD

Figure 4 shows a breakdown of the main applications of EBSD, extracted from articles published in 2008. Sometimes more than one application features in a single report, e.g. often microtexture and microstructure characterisation are carried out concurrently. These applications will now be discussed in more detail.

### General microstructure characterisation and quantification

The biggest growth area in EBSD over the last few years is its application to general microstructure characterisation, even in complex microstructures such as welds [17]. Morphological features that have been traditionally observed by optical microscopy can now be recorded via an EBSD map, with all the entailed benefits of digital imaging and crystallographic data. Figure 5 illustrates a comparison between an optical micrograph (Fig. 5a) and an EBSD pattern quality parameter map (Fig. 5b) from an iron specimen. Clearly the grain structure has been reproduced faithfully in the EBSD map. In a multiphase microstructure, often phases can be distinguished by differences in diffraction pattern quality, for example in an electroplated nickel composite coating co-deposited with titanium oxide (rutile) particles [18].

The growth in application of EBSD as a general microstructure characterisation tool has been expedited by the very fast mapping speeds that can currently be achieved on suitable materials. At the time of writing a speed of 750 points per second has been quoted in the scientific literature [19]. A proviso is that error limits may have to be widened in order to achieve such speeds, which feeds through to lower reliability. Whether or not this can be tolerated depends on the nature of the investigation.

Changes in the microstructure can be observed and quantified readily by EBSD mapping. Hence EBSD has

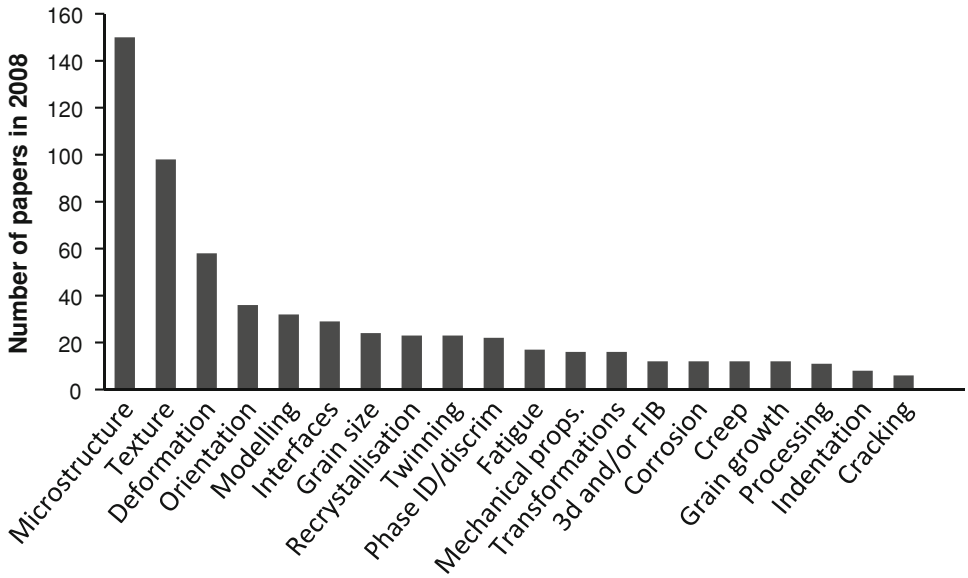


**Fig. 4** Breakdown of the main applications of EBSD, extracted from papers published in 2008

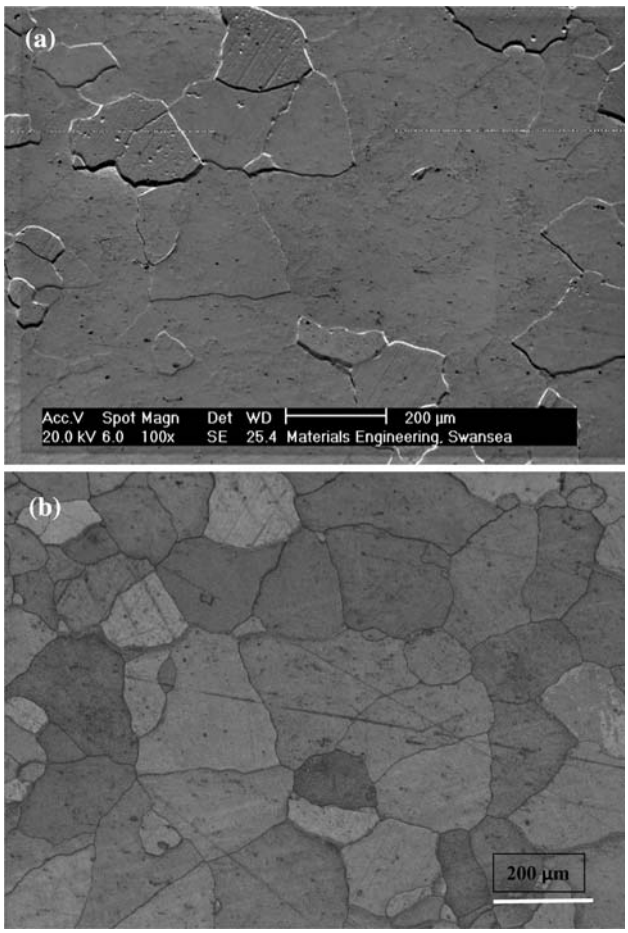


**Fig. 5** Comparison between (**a**) an optical micrograph and (**b**) an EBSD pattern quality parameter map from an iron specimen

come to the fore in the measurement of grain size distribution and furthermore allows accompanying derivative analyses such as grain axis ratio and microtexture according to grain size class. Grain size determination and application of EBSD to phenomena such as grain growth are cited in Fig. 4. It is important for grain size determination that small grains are recognised unambiguously in maps, and that an appropriate choice is made for the lower limit of grain boundary misorientation to define a grain. The practical aspects of grain size determination by EBSD have recently been discussed in detail [20].

## Determination of orientations and orientation distributions (texture)

Microtexture is defined as 'a sample population of orientation measurements which can be linked individually to their location within a specimen' [2]. The determination of microtexture, which historically was the first mainstream application of EBSD, is still a major use of the technique. Pole figures and inverse pole figures are the most popular choice for display and analysis of microtexture data. Orientation distribution function (ODF) display of EBSD is also used, but less commonly.

It is important to distinguish between microtexture data and 'macrotexture' data. Microtexture refers to the orientations in the sampled area only, and does not imply the texture of the entire specimen whereas macrotexture is defined as 'an average texture determined from many grains obtained without necessarily having reference to the location of individual grains within a specimen' [2]. In the past macrotexture could only be measured using X-ray or neutron diffraction, in order to obtain sufficient grain sampling. Nowadays the increased speed of data acquisition means that it is viable to obtain macrotexture by EBSD, but only if due attention is paid to the sampling schedule and quantity of data. The statistical reliability of texture measurements by EBSD have been compared to those obtained by X-rays in steel specimens [21]. The results from the two techniques were comparable. Approximately 10,000 grains were found to produce a very good sampling in these materials. It should be emphasised that this data requirement refers to numbers of grains, and not simply data points. Prior knowledge of the grain size is therefore needed. Texture measurement via EBSD has many advantages over X-ray methods, for example differentiating textures in large and small grains. Despite these advantages, EBSD is little used for macrotexture. Instead, in a number of publications EBSD microtexture data are measured in conjunction with X-ray texture data [e.g. 22, 23].

Some publications refer to measurement of orientations, rather than texture. This implies smaller, more specific datasets and/or more complex materials. For example the influence of grain orientation on oxygen generation in anodic titania has been investigated [24].

## Phase identification and discrimination

The principle of identification of a phase by EBSD is that the full crystal symmetry of the specimen is embodied in the symmetry of the diffraction patterns [25]. If an experimentally acquired, unknown EBSD pattern is indexed using the correct phase match, a consistent and accurate match will be achieved with all parts of the pattern simulation of the phase match candidate. Phase match candidates are selected from compiled or existing external crystallographic databases. Chemical composition analysis of the phase by energy dispersive spectroscopy (EDS) is used as a filter in selection of candidate phases. For example, a trigonal and a hexagonal phase have been identified in the Au–Sn system, despite their similar chemistry [26].

The EDS and EBSD analyses can be performed as two separate steps. A recent advance is the integration of both these steps into a single interface such that chemical and crystallographic data are acquired simultaneously [25]. This requires conjoint data collection from a highly tilted specimen (for EBSD), no shadowing from either the EDS detector or the EBSD camera, and rationalisation of the different dwell times to acquire EDS and EBSD data. A high sensitivity EBSD camera is best used for dedicated phase identification work. The dedicated phase identification package compares automatically-collected diffraction patterns from an unknown phase with simulated patterns from reference phases, using chemical composition from the EDS spectra to filter out impossible solutions. Links to a number of external databases, such as the International Center for Diffraction Data (ICDD), are used to find the candidate reference phases and then to simulate diffraction patterns from the crystallographic parameters, including full structure factor calculations. Often such databases were originally compiled for X-ray diffraction, and conversions are applied for electron diffraction. Phases from all seven crystal systems can be identified by the combined method. For example, complex phase compositions in the Cr–Si–Nb system have been identified, including orthorhombic $(Cr,Nb)_{11}Si_8$ and orthorhombic $(Cr,Nb)_6Si_5$ (http://www.oxinst.com/products/microanalysis/ebsd/ebsd-applications/Pages/applications.aspx).

The EBSD approach to phase identification offers better spatial resolution than does X-ray powder diffraction analysis, plus concurrent microstructure and microtexture information. Despite these advantages it is still relatively little used, as shown in Fig. 4. When it is used, it is more often as 'phase discrimination', that is, when it is known that certain phases are probably present and EBSD is used to distinguish between them. For example EBSD has been used to distinguish between $M_{23}C_6$ (cubic crystal structure) and $M_7C_3$ (orthorhombic crystal structure) in a steel, where identification based on chemistry is not feasible [27].

## Other applications

As mentioned at the end of "Material types" section, the now widespread use of EBSD in the FEGSEM has facilitated application to deformed materials, which in turn has led to more studies of plasticity and deformation processes such as stress-strain behaviour or analysis of deformation by slip or twinning. For example deformation behaviour of Mg alloys during ring hoop tension testing has been investigated [28]. EBSD has also contributed to studies of other materials processing such as extrusion [29], sintering [30], annealing [31] and mechanical properties such as strain hardening [32] or response to nanoindentation [33]. Recovery and recrystallisation studies by EBSD can be enhanced by quantification of the recrystallised fraction. A new method is described in detail elsewhere [34].

The characterisation of interface crystallography, usually via categorisation of the misorientation across grain boundaries, is a long-standing application of EBSD [e.g. 35]. Most of the application has been mainly to characterisation of misorientation distributions in cubic metals, particularly via the coincidence site lattice (CSL) scheme. A more recent trend is the application of EBSD to interface misorientation in more complex materials, including multiphase materials. An example is that the CSL distribution has been measured in alumina and zirconia [36]. Furthermore EBSD is used increasingly to investigate crystallographic and microstructural aspects of both annealing twinning and deformation twinning. For example non-Schmid behaviour during secondary twinning has been investigated in a polycrystalline magnesium alloy [37].

Recently more complex analyses of interfaces are being carried out, such as measurement of the grain boundary plane crystallography in addition to the misorientation. The distribution of boundary planes can either be obtained by a stereological method, which is described in detail elsewhere [38, 39], or by a means of serial sectioning in order to obtain the inclination of boundary planes in three dimensions. Measurement of boundary plane distributions has allowed some hitherto inaccessible observations to be made. For example for many materials the frequency of occurrence of certain planes is inversely correlated to their energy [40], and in 'grain boundary engineered', fcc metals it has been shown that the $\Sigma 3$ boundary category is dominated by incoherent twins, and that $\Sigma 9$ boundaries frequently have low-index planes [41].

Serial sectioning can now be performed in situ using focused ion beam (FIB) tomography. An exciting extension to this technique has been the recent integration of FIB technology with an SEM, to give a 'Dual Beam' instrument capable of both precision sectioning and high resolution imaging. An added benefit of the Dual Beam configuration is that EBSD is possible, which is described elsewhere [6]. The applications of this set-up fall into two main categories: analysis of certain delicate or awkward specimens and acquisition of three-dimensional (3D) data. For example, FIB/EBSD has been used to obtain in situ data from magnetite specimens which would otherwise have lost their magnetic properties [42]. The acquisition of 3D orientation maps [43], obtained by reconstruction of many slices of FIB/EBSD data, provides direct information on all the 3D aspects of microstructure, including grain volume distribution and grain boundary plane crystallography. As yet there are few examples of '3D EBSD' in the literature. One example is the application of FIB/EBSD to deduce the origin of systematic deformation-induced crystallographic orientation patterns around nanoindents in copper [44].

Application of EBSD to phase transformations is a sophisticated application of the technique. It is used to study thermodynamics and kinetics of phase transformations, solidification, solid state phase transformations, environmentally assisted reactions and thin film deposition. A recent comprehensive review has described these applications in detail [45]. Recently, examples of EBSD applied to phase transformations include epitaxy [46], precipitation [47], orientation relationships (e.g. between cementite and ferrite) [48], variant selection [45], and nucleation [49].

EBSD data can be exported to other applications or combined with separate data or test results. In this way EBSD is making a significant contribution to analysis of various degradation and failure phenomena, e.g. corrosion [50], creep [51] and fatigue [52]. These categories are featured on figure 4. EBSD data is also used in conjunction with modelling applications. One such example is that orientation mapping of the microstructure has been used as the basis for finite element analysis of strain localization behaviour in AA5754 aluminum sheet [53].

Lattice strain can be accessed via EBSD by measuring either the amount of diffuseness in diffraction patterns or small shifts in patterns. This is a niche application for EBSD which requires specialised analysis. Recently, strain has been measured in Si–Ge layers on Si [54]. Other aspects of strain-related exploitation of EBSD have concentrated on visualisation of plastic deformation or retrieval of dislocation content, for example by the 'gradient matrix' method [55].

## Concluding remarks

This review has aimed to capture the present contribution that EBSD is making as an investigative tool in materials science. It has been shown that some aspects have increased greatly recently, particularly the range of materials analysed, microtexture determination, general microstructure characterisation, application to interfaces and combinations of EBSD with other applications such as modelling or materials testing. On the other hand some other applications of EBSD are still emerging, such as macrotexture determination, true phase identification and 3D EBSD.

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