INTERNATIONAL STANDARD

ISO 13067

First edition
2011-11-01

Microbeam analysis — Electron backscatter diffraction — Measurement of average grain size

Analyse par microfaisceaux — Diffraction d'électrons rétrodiffusés — Mesurage de la taille moyenne des grains

Reference number
ISO 13067:2011(E)

COPYRIGHT PROTECTED DOCUMENT

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 13067 was prepared by Technical Committee ISO/TC 202, *Microbeam analysis*.

Introduction

The mechanical and electromagnetic properties of engineering materials are strongly influenced by their crystal grain size and distribution. For example, strength, toughness and hardness are all important engineering properties that are strongly influenced by these parameters. Both bulk materials and thin films, even as narrow two-dimensional structures, are influenced by grain size. For this reason, it is important to have standard methods for its measurement with commonly used and agreed terminology. This International Standard describes procedures for measuring average grain size from maps of local orientation measurements using electron backscatter diffraction.

Microbeam analysis — Electron backscatter diffraction — Measurement of average grain size

IMPORTANT — The electronic file of this document contains colours which are considered to be useful for the correct understanding of the document. Users should therefore consider printing this document using a colour printer.

1 Scope

This International Standard describes procedures for measuring average grain size derived from a two-dimensional polished cross-section using electron backscatter diffraction (EBSD). This requires the measurement of orientation, misorientation and pattern quality factor as a function of position in the crystalline specimen[1].

NOTE 1 While conventional methods for grain size determination using optical microscopy are well-established, EBSD methods offer a number of advantages over these techniques, including increased spatial resolution and quantitative description of the orientation of the grains.

NOTE 2 The method also lends itself to the measurement of the grain size of complex materials, for example those with a significant duplex content.

NOTE 3 The reader is warned to interpret the results with care when attempting to investigate specimens with high levels of deformation.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 16700, *Microbeam analysis — Scanning electron microscopy — Guidelines for calibrating image magnification*

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

ISO 21748, *Guidance for the use of repeatability, reproducibility and trueness estimates in measurement uncertainty estimation*

ISO 23833, *Microbeam analysis — Electron probe microanalysis (EPMA) — Vocabulary*

ISO 24173:2009, *Microbeam analysis — Guidelines for orientation measurement using electron backscatter diffraction*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply. The reader is also referred to ISO 24173 and ISO 23833 for additional terms and definitions.

3.1 Terminology associated with EBSD measurement of grain size

3.1.1
step size
distance between adjacent points from which individual EBSD patterns are acquired during collection of data for an EBSD map

3.1.2
pixel
picture element
smallest area of an EBSD map, with the dimensions of the step size, to which is assigned the result of a single orientation measurement made by stopping the beam at a point at the centre of that area

3.1.3
orientation
mathematical description of the angular relationship between the crystal axes of the analysis point and a reference frame, usually the specimen axes

3.1.4
indexed
a pixel is said to be indexed if the orientation calculated from the EBSD pattern acquired for that pixel meets a predetermined threshold for reliability

3.1.5
indexing reliability
numerical value that indicates the confidence/reliability that the indexing software places in an automatic analysis

NOTE This parameter varies between EBSD manufacturers, but can include:

a) the average difference between the experimentally determined angles between diffracting planes and those angles calculated for the orientation determined by EBSD software;

b) the difference between the number of triplets (intersections of three Kikuchi bands) in the EBSD pattern matched by the chosen orientation and the next best possible solution, divided by the total number of triplets.

3.1.6
orientation map
crystal orientation map
map-like display of pixels derived from the sequential measurement of crystal orientation at each point in a grid [see Figures 1 b) to 1 f)] showing the crystallographic relationship between the pixels and the reference frame

3.1.7
pattern quality
measure of the sharpness of the diffraction bands or the range of contrast within a diffraction pattern

NOTE Different terms are used in different commercial software packages, including, for example, band contrast, band slope and image quality.

3.1.8
pattern quality map
map-like display of pixels derived from the sequential collection of EBSD patterns at each point in a grid [see Figure 1 a)] showing the pattern quality of the individual pixels

NOTE 1 Since measures of pattern quality can change at features such as grain boundaries and with orientation, the pattern quality map can give an indication of grain shape and size.

NOTE 2 Pattern quality maps can also indicate areas of heavy deformation and inadequate preparation, such as residual scratches.

NOTE 3 Small particles and features also contribute to the pattern quality map.

3.1.9
pseudosymmetry
potential for an EBSD pattern to be indexed in several different ways due to internal similarities within the EBSD pattern

NOTE 1 Pseudosymmetry is a problem with some crystal orientations, usually when a main zone axis is in the centre of the pattern. Typical cases are a {0001} pole for a hexagonal structure and a <111> pole for a cubic structure.

NOTE 2 Structures such as high-symmetry tetragonal crystals with an axial ratio, c/a, approximately equal to 1 are also likely to exhibit pseudosymmetry in EBSD patterns.

3.1.10
misorientation
given two crystal orientations, the misorientation is the rotation, often defined by an angle/axis pair, required to rotate one set of crystal axes into coincidence with the other set of crystal axes

3.1.11
disorientation
due to crystal symmetry, there can be several axis/angle pairs which represent the same misorientation, in which case the one having the smallest angle is called the disorientation

NOTE 1 For most crystal symmetries, there are multiple symmetrically equivalent axes for the disorientation with the smallest misorientation angle.

NOTE 2 Misorientation and disorientation are terms which are often used interchangeably. Disorientation is the more rigorous term here, but misorientation is the more frequently used.

3.1.12
forescatter imaging
orientation contrast produced from electrons which channel out of the specimen

3.1.13
electron-channelling contrast imaging
ECCI
orientation contrast produced from electrons which channel into the specimen

3.1.14
barrel distortion
difference in lateral magnification between the central and peripheral areas of an image such that the lateral magnification is less at the periphery

NOTE A square object in the centre of the field appears barrel-shaped (i.e. with convex sides).

3.1.15
pincushion distortion
difference in lateral magnification between the central and peripheral areas of an image such that the lateral magnification is greater at the periphery

NOTE A square object in the centre of the field appears cushion-shaped (i.e. with concave edges).

3.2 Terminology associated with grains and grain boundaries determined via EBSD

3.2.1
grain boundary
line separating adjacent regions of points in an EBSD orientation map with disorientation across the line greater than a minimum angle chosen to define the grain boundaries

3.2.2
grain
region of points with similar orientation (within a tolerance), completely enclosed by grain boundaries and greater than the minimum size defined to exclude isolated (often badly indexed) points as small grains

3.2.3
sub-grain boundary
line separating adjacent regions of points in a grain with a difference in orientation across the line smaller than that defining a grain but greater than that defining a sub-grain

NOTE Effectively, sub-grain boundaries are grain boundaries with a smaller misorientation limit than that defining a grain boundary. These boundaries can have a characteristic linear appearance and exhibit a characteristic misorientation.

3.2.4
sub-grain
region of points with similar orientation completely enclosed by boundaries greater than the minimum sub-grain boundary angle

3.2.5
special boundary
boundary between two grains having a special orientation relationship within a tolerance associated with identifying them in orientation maps

3.2.6
twin boundary
particular case of a special boundary between crystals oriented with respect to one another according to some symmetry rule, in which the boundary itself is planar and is a characteristic crystallographic plane (for both crystals) and, frequently, one crystal is the mirror image of the other

NOTE For example, in face-centred-cubic structures, the characteristic misorientation defining a common twin can be described as a 60° rotation about the <111> axis with the boundary plane normal to the rotation axis.

3.2.7
recrystallized grains
new set of undeformed grains formed by consuming deformed grains through nucleation and growth processes

NOTE Measurements of misorientation within grains by EBSD can be used to distinguish between deformed and undeformed grains.

3.2.8
phase
physically homogeneous volume in a material having the same crystal structure and chemical composition

3.3 Terminology associated within grain size measurement

There are a variety of ways of representing average grain size. This subclause outlines some of the more common terms used, and the reader is referred to Annex A for more details about other terms, about the standards available and about the applicability of methods for particular grain shapes and distributions.

3.3.1
line intercept
distance between the points at which a straight line crossing a grain intersects the grain boundary on each side

NOTE See ASTM E112 for more details.

3.3.2
equivalent circle diameter
D_{circle}
diameter of the circle with an area equivalent to the grain section area, given by:

$$D_{\text{circle}} = (4A/\pi)^{1/2}$$

where A is the area of the grain.

NOTE The ASTM grain size number, G, is given by:

$$G = -6{,}64 \log_{10} D_{\text{circle}} - 2{,}95$$

where D_{circle} is measured in millimetres.

3.3.3
Feret diameter
perpendicular distance between two parallel lines drawn in a given direction tangential to the perimeter of an object on opposite sides of the object

NOTE 1 It is also known as the calliper diameter.

NOTE 2 Different variants of the Feret diameter are used. For example, the Feret diameter can be measured in the vertical and horizontal directions or in any two directions at right angles to each other.

3.3.4
grain shape
property whose value is determined by fitting an ellipse round the grain and measuring the aspect ratio, i.e. the ratio of the length of the minor axis to the length of the major axis

NOTE 1 It is sometimes referred to as grain elongation.

NOTE 2 The value lies in the range 0 to 1.

NOTE 3 There are several ways of fitting the ellipse round the grain, and different methods can result in small differences in the measured aspect ratio.

3.3.5
grain shape orientation
angle between the major axis of an ellipse fitted round the grain and the horizontal direction, usually measured counterclockwise

3.4 Terminology associated with data correction and uncertainty of EBSD maps

3.4.1
misindexing
assigning an incorrect orientation or phase to the measured EBSD pattern

NOTE This can occur for a number of reasons, e.g. pseudosymmetry effects, attempting to index a poor pattern or attempting to index a pattern from an unanticipated phase for which the indexing software is not configured.

3.4.2
non-indexing
non-assignment of an orientation due to insufficient quality of the EBSD pattern

NOTE This can occur for a variety of reasons, such as roughness of the specimen, dust on the specimen, overlapping patterns at the grain boundary, a poor-quality pattern due to the effects of strain, or if the pattern is from an unanticipated phase.

3.4.3
data cleaning
process chosen to accommodate non-indexed and misindexed data within the map, using a given set of parameters, typically based on the characteristics (orientation, phase) of a certain number of nearest neighbours [see Figures 1 b) to 1 f)]

NOTE A wide range of terms (not necessarily mathematically precise) is used in the various commercially available software packages for different data-cleaning operations, including noise reduction, extrapolation, dilation and erosion.

4 Acquiring a map by EBSD for grain size measurement

4.1 Hardware requirements

The reader is referred to ISO 24173 for equipment needed to acquire electron backscatter patterns, index the patterns (determine the orientation) and either step the beam across the specimen surface or, less commonly, step the stage, keeping the beam stationary to acquire a map.

4.2 Software requirements

4.2.1 The software shall allow the orientation data (or other parameters, such as pattern quality derived from each diffraction pattern) to be displayed as a map.

4.2.2 The software shall correct misindexed pixels or fill in non-indexed pixels (see 6.2 and 6.3).

4.2.3 The software shall use orientation data to define the positions of boundaries in accordance with the criteria selected.

4.2.4 The software shall identify grains as regions of connected pixels from the set of boundary points and measure grain size parameters. Special treatment may be applied to grains that intercept the map edges, e.g. removal or weighting.

5 Acquiring the map for grain sizing by EBSD

5.1 Specimen preparation

In order to achieve a high degree of indexing of individual pixels, it is necessary to produce a surface finish which produces EBSD patterns of sufficient quality to be indexed reliably. The criteria used for indexing reliability shall be defined and reported by the user.

The surface preparation method adopted will be dependent on the material and also on its condition, e.g. metallurgical heat treatment. The reader should refer to standard texts on polishing and etching and Annex B of ISO 24173:2009. Over-etching of grain boundaries should be avoided since it leads to increased numbers of non- and mis-indexed points and to low index reliability at the grain boundaries.

If necessary, the specimen may be coated with a thin conductive coating (such as carbon) to prevent charging and electron beam drift and thus avoid distortion of the image.

5.2 Defining specimen axes

If the specimen is known to be strongly textured, e.g. from thermomechanical processing, the axes of the specimen shall be identified prior to preparation for EBSD such that EBSD measurements can be related to these axes. These axes are usually related to the rolling direction, to a growth direction or to a principal applied stress.

5.3 Stage positioning and calibration

The procedures set out in ISO 24173 shall be followed. The specimen shall be fixed to the scanning electron microscope (SEM) stage in the desired orientation with the specimen axes relative to the stage axes and imaged at a working distance at which the SEM and EBSD image magnification has been calibrated and at which the EBSD system itself has been calibrated to index diffraction patterns.

The purpose of this calibration is to check that there is no influence of distortion on the recorded patterns and to ensure that the tilt angle relative to the specimen is correct. Reference [13] discusses distortion round the edges.

The specimen tilt has a significant effect on the image magnification in the direction on the specimen surface normal to the tilt axis. Great care shall be taken to measure the tilt angle of the specimen surface accurately.

NOTE A 1° change in tilt angle at a tilt angle of 70° will cause a change of ~5 % in the size of the step used in the direction on the specimen surface normal to the tilt axis when collecting data for the map.

5.4 Linear calibration

Follow the recommendations of ISO 16700.

5.5 Preliminary examination

An initial examination of the specimen shall be made to identify an initial set of operating parameters needed to map the orientation of the specimen with an acceptable level of accuracy and within an acceptable period of time over an area sufficient to give data on a statistically significant number of grains.

The reader is referred to ISO 24173 for information needed to measure the orientation.

5.6 Choice of step size

5.6.1 If the grain size and shape are not known already, an approximate grain size and shape estimation shall be performed by a quick imaging technique. An optical microscope might work on a region with only slight polishing relief or on an etched region adjacent to that to be examined by EBSD. Forescatter[10] or electron-channelling contrast imaging using diodes mounted on the EBSD detector, or imaging with the specimen current, can also produce images relatively quickly.

As an alternative to mapping, some EBSD software offers a line intercept method as a mapping mode. This can be used to quickly give an approximate grain size measurement.

5.6.2 The step size should be chosen in relation to the average grain size, unless information on a particular minimum size is required. In either case, it has to be recognized that a judgement is being made on the minimum number of pixels that are used to define a grain either by a lineal or areal method. See also 6.3 and Figures 1 d), e) and f) for the effects of step size choice.

A simple rule that can be applied to a preliminary scan is that the step size should be less than 10 % of the approximate mean grain size[2]. To confirm the validity of the chosen step size, repeat the mapping of a single area at several step sizes and determine the maximum size below which no significant difference in average grain size is determined. This choice has a direct influence on the accuracy of the grain size measurement.

5.6.3 In choosing the step size, the spatial resolution of the system needs to be considered. The step size is preferably larger than the interaction volume, which will be determined both by the material examined and the operating parameters of the SEM, such as the filament type, accelerating voltage and aperture size.

5.7 Determination of the level of angular accuracy needed[7][8]

The speed with which EBSD patterns are acquired (including any averaging of patterns) affects the precision with which band edges can be detected and thus the angular accuracy of the calculated orientation. Other factors, such as the Hough resolution and the number of bands chosen to match the calculated orientation, also affect the calculation time as well as the angular accuracy.

If too long a time is taken for acquisition and calculation, problems of specimen drift can be increased significantly and fewer points will be acquired in a given time, reducing the statistical significance of the data acquired. To minimize drift, it is recommended that the specimen have a good earth (ground) path and be securely fastened to the stage. Avoid carbon tabs. A thin carbon coating might also be necessary for insulating specimens.

If the time taken is too short, then levels of indexing reliability will be reduced. The settings chosen as a compromise between the two opposing factors above shall be recorded.

To save time, EBSD patterns may be saved without indexing during mapping and subsequently indexed off-line to investigate the effect of some of the above parameters on indexing accuracy.

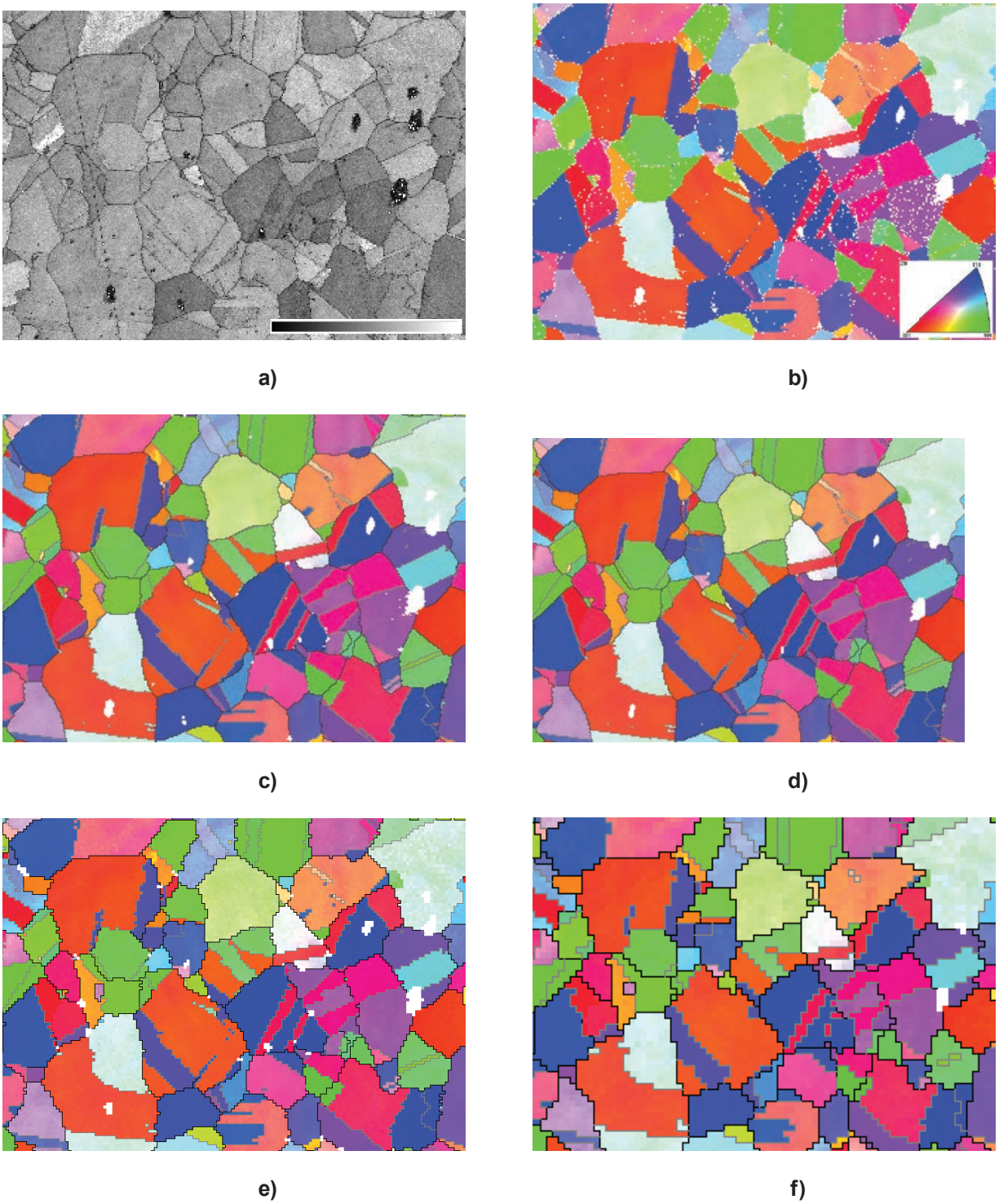


Figure 1 — An area of an Ni superalloy mapped by EBSD under different conditions

Figure 1 includes:

a) a pattern quality map (grey-scale range covering 20 to 160 of 256 grey levels), generated using a 0,5 µm step size;

b) from the same data set, the raw orientation map (96,7 % indexed) with non-indexed points in white and inverse pole figure colouring of orientations (specimen normal direction, with key bottom right);

c) Figure 1 b) after removing clusters of 3 pixels or less and replacing the unindexed pixels by orientations based on their six nearest neighbours (99,3 % indexed);

d) similar to Figure 1 c), but based on two instead of six nearest neighbours (99,8 % indexed);

e) the same area mapped with a 1 µm step size;

f) the same area mapped with a 2 µm step size.

Figures 1 c) to f) all use the orientation key shown in Figure 1 b) and show grain boundaries (>10°) in black and twin boundaries (60° ± 1°, [111] ± 1°) in grey.

5.8 Choice of areas to be mapped and map size

The areas chosen for examination shall be representative of the microstructure as a whole, and, if there is variation with position in the specimen, the positions examined shall be recorded in relation to the specimen geometry.

For conventional linear-intercept measurements, standards such as ASTM E112 recommend measurement of a minimum of 50 grains from a minimum of 3 fields. Local precision can be increased significantly by measurement of 500 to 1 000 grains, and the overall uncertainty, quoted as a confidence level, is determined by the variation from field to field and is reduced by increasing the number of fields.

Because EBSD maps enable the size of all grains in a given field to be measured, the minimum number of 50 grains can generally easily be exceeded, and large areas examined relatively quickly. The use of running-average plots can be useful in showing that a stable, repeatable value has been obtained. At low magnifications, errors in orientation measurement can increase round the periphery of the image. Some acquisition software will allow these effects to be corrected by calibration. It is sometimes better to measure a larger number of fields, smaller in area, at high magnifications to obtain an average with lower uncertainty.

A method of quantifying grains at the edge of the image is required[3], and frequently grains that are cut by the edge of the image are not taken into account. If a small number of grains is obtained from a single map, a poor average grain size might result because grains cutting the edge of the image are not available for evaluation. This is more important if the grain size distribution is large. The “bias” incurred can be compensated for by the Miles-Lantuéjoul correction[4] by assigning each particle a weight that is proportional to the chance it has of being contained within the measurement field.

With some equipment and software, it is possible to join together EBSD maps of adjacent areas. This should be avoided since joining maps in this way can lead to errors of alignment and the creation of false boundaries. Since grain size is a statistical quantity, it is better practice to take measurements on several separate areas.

NOTE 1 If statistical tools can be used to reduce errors in joining maps together, this process of grouping maps could be of interest.

NOTE 2 Difficulties in aligning images might be caused by using too low a magnification, giving rise to aberrations in the images, such as radial distortions (e.g. pincushion and barrel distortion) and scan rotation or an incorrectly set up SEM that shows poor orthogonality in the scan. Orthogonality errors can be observed and corrected for with the aid of a rectangular grid.

5.9 Considerations when examining plastically deformed materials

Where there is a high degree of damage, e.g. from plastic deformation, it might be impossible to obtain good diffraction patterns. This makes indexing impossible or leads to inaccurate measurement of orientation or phase. Subclauses 6.2 and 6.3 consider the treatment of maps where this occurs, but it should be noted that, in cases where a substantial number (>10 %) of pixels are not indexed reliably, this treatment can distort the results and introduce significant inaccuracies.

Furthermore, deformation often leads to the formation of new grains and sub-grain boundaries. However there is no universally agreed definition of the misorientation angles that define these boundaries since the significance of the boundary angle will vary depending on material type and the property under consideration.

Thus, even if good indexing is achieved, it is essential that any measurement of size in a deformed material specify the misorientation angle used to define a grain boundary.

Heavily deformed microstructures can also show significant anisotropy, and several definitions might be needed to give representative descriptions of the grain size.

A further possible consequence of deformation, particularly at elevated temperatures, is the formation of strain-free recrystallized grains. In such cases, these grains might have a significantly larger grain size than the initial grains, resulting in a bimodal grain size distribution and the need to map at different step sizes to resolve the distribution.

6 Analytical procedure

6.1 Definition of boundaries

6.1.1 Grain boundary angles

After following the steps above and acquiring data to plot, for example, maps of orientation, grain boundaries can be drawn on the maps. This requires the angles defining the various possible boundaries to be chosen. Guidelines for this are given below but, whether these or other methods are used, the definitions and procedures used for grain size values shall be stated with all results.

For relatively simple equiaxed grain structures, such as fully recrystallized metals or undeformed cast metals, the misorientation that is used to define the grain boundary may be taken to be as little as 5°. For these types of material, there is evidence that misorientation angles between 5° and 15° make little difference to the average grain size[5].

For other materials, with more complicated grain structures, larger angles, typically 10° or 15°, depending on material, are used. Measurement of grain size as a function of misorientation angle might be useful in gathering information on the structure. Care shall be taken to ensure that the prescribed angle is not so large that it results in two or more distinct, preferred orientations being encompassed within the angular range[11][12].

6.1.2 Handling incomplete boundaries

In some materials, particularly after deformation, selected boundaries might not extend completely between two regions to terminate at a triple point with another boundary because the measured misorientation changes along the length of the boundary fall below the defined grain boundary angle. In such cases, it might be possible to extrapolate the boundary by reducing the minimum angle generally used elsewhere in the map (see 5.6) to a new, lower, value. If this is done, it shall be recorded with the final result (preferably reporting the effect on mean size with and without extrapolation). It is, however, preferable to measure size with reduced misorientation angles defining grain boundaries and to note the effect of this reduction on the measured grain or sub-grain size.

6.1.3 Dealing with special boundaries

With conventional techniques, special boundaries such as twins in cubic materials, which can be identified by their morphology, are frequently ignored for the purposes of grain size measurement. Since EBSD measures angle/axis quantitatively, these boundaries can be easily determined by software and excluded from EBSD measurements of grain size. However, since there will be some variation in measured angle and axis about the idealized value, the tolerances used to define the boundaries shall be recorded (e.g. ±2° from 60° about <111>). The following should also be noted:

a) EBSD will define some boundaries as twins because they meet the defined misorientation tolerances, whereas conventional optical microscopy would not identify them because the typical morphology of twinning is not obvious. This effect can be reduced by only including those boundary segments with a trace, which also satisfies the requirements for a twin plane[9].

b) Removal of grain boundaries in a) will lead to larger grain sizes than if twins are included.

6.2 Post-acquisition treatment of raw data

Rarely will every pixel in an EBSD orientation map be correctly indexed. In addition to errors in orientation measurement for each pixel indexed, some pixels will not be indexed. The relative proportions of these pixels will depend on specimen preparation, the nature of the specimen, the SEM operating conditions and the EBSD indexing parameters.

In simple recrystallized specimens, it is normally possible to achieve a level of 95 % of pixels with acceptably high index reliability. This level of 95 % should be the target for all maps, but in many cases this level is not reached and, if the raw data is used to determine grain size without the data-cleaning steps described in 6.3, serious errors in size might result. Equally, incorrect or excessive manipulation of the raw data can alter the final measured sizes significantly.

NOTE In multiphase materials, it might be necessary to treat each phase differently, using a separate dataset for each phase.

6.3 Data-cleaning steps

6.3.1 Remove all grains with a number of pixels lower than a user-defined value (typically 3 to 5)[5]. See also 6.3.4 for removal of the smallest grains after image processing. The threshold value and the number removed shall be recorded.

6.3.2 Index any singly unindexed pixel where it is surrounded by x or more pixels of the same orientation, where the value of x is dependent on the grid used for mapping (square or hexagonal). Care needs to be taken if this process is repeated several times (a single pass is generally sufficient for non-indexed points at grain boundaries). The percentage indexed should not be increased by more than, typically, 5 %. The percentage indexed in this manner shall be reported.

The effect of data cleaning (which has the potential to introduce artefacts) shall be investigated according to the information required on the grain size. (For example, the mean value might be less sensitive to data cleaning than the whole histogram, in particular for the smaller grain sizes.) The histogram of grain size given by analysis of the raw data shall be recorded to assess any excessive artefacts introduced by data cleaning.

6.3.3 Optionally, an orientation filter, such as Kuwahara filter[6], may be used to reduce errors in orientation measurement. This is especially important for heavily deformed specimens. Use of such a filter shall be recorded with the results.

It should be borne in mind that the Kuwahara filter can introduce diagonal features into maps, and care should be taken to check that the features produced are actually present in the microstructure, e.g. by looking at the forescatter image or EBSP quality map.

6.3.4 Choose the minimum grain size to be included in the calculations of the grain size. For conventional linear-intercept measurements, the minimum recommended length to measure is 10 pixels (so that, in the worst case, a +1 pixel error at one end and a –1 pixel error at the other end would give a maximum error of 20 %); this would suggest a minimum grain size area of 100 pixels. However, it has been shown[5] that measurements on all grains $>$10 pixels in area gives valid results since EBSD validates each pixel in a grain by measuring its orientation. Pixellation errors, which alter the true grain size at small sizes, are approximately 5 % for an area of 10 pixels.

It is therefore recommended that all grains $>$10 pixels in area be included in the calculation of grain size. This agrees approximately with conventional image analysis, where the minimum size of an object is 9 (3×3) pixels for a square grid or 7 for a hexagonal grid to avoid erosion by a single layer of pixels deleting an object completely.

6.3.5 Maps showing (a) grains of more than 10 pixels and (b) grains of less than 10 pixels shall be investigated alongside maps of diffraction pattern quality which might highlight the significance of any data omitted from the calculation.

6.3.6 All grains touching the edge of a map shall be excluded from calculations of grain size (see also 5.8).

6.3.7 Figures 2 to 4 show examples of the effects of some of the above methods on the measurement of grain sizes in the nickel material used to make the maps shown in Figure 1.

Figure 2 shows cumulative grain section distributions from a larger region of the 0,5 µm step size maps shown in Figures 1 b) to d), resulting from various data-cleaning methods:

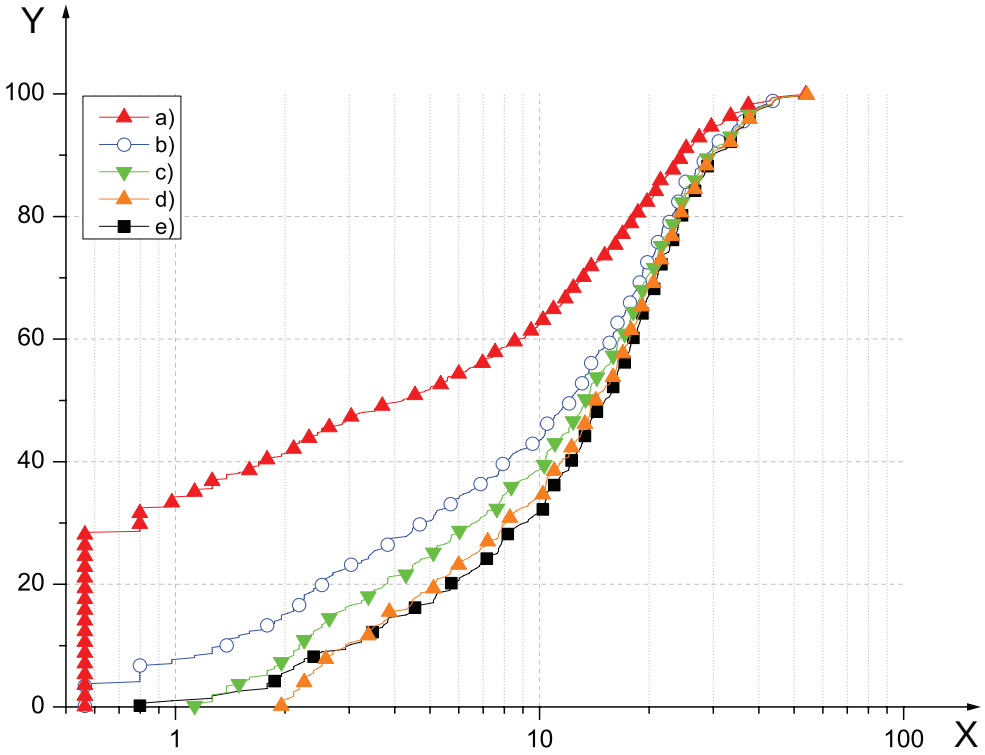
a) raw data;

b) removed single isolated pixels, dilated into unindexed pixels where five neighbours indexed;

c) as b), but removed 3-pixel clusters;

d) as b), but removed 10-pixel clusters;

e) as b), followed by removal of all grains with two or fewer neighbours.



Key

X grain size (circle equivalent diameter), expressed in µm

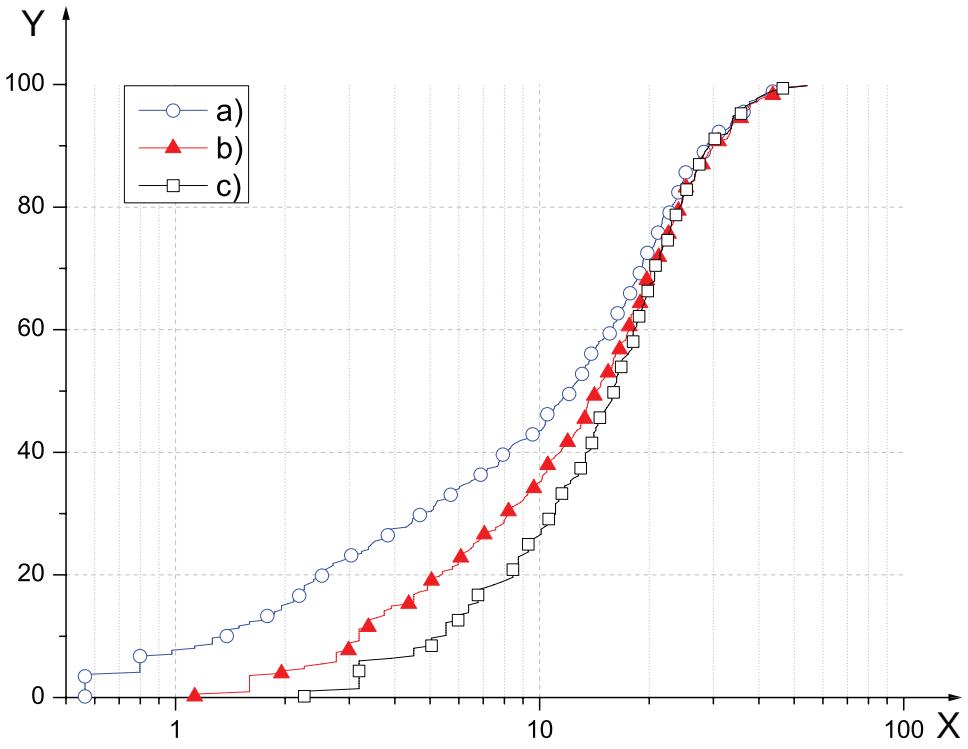
Y cumulative probability, expressed in %

Figure 2 — Cumulative grain size distributions resulting from different data-cleaning methods

Figure 3 shows cumulative grain size distributions resulting from maps of the same area produced with step sizes of a) 0,5 µm, b) 1 µm and c) 2 µm, [the 0,5 µm data is the same as that shown in Figure 2)] after data-cleaning steps involving the removal of single isolated pixels and dilation into unindexed pixels where five neighbours were indexed. See also Table 1.

Table 1 — Mean values for grain size, i.e. circle equivalent diameter, from the graphs shown in Figure 2 to illustrate the differences in measured average grain size depending on the data-cleaning method chosen

Data-cleaning method		0,5 µm		1,0 µm		2,0 µm	
		Size µm	Number	Size µm	Number	Size µm	Number
a)	Raw data	9,3	571				
b)	Single pixels removed	13,7	304	15,7	265	16,9	242
c)	3-pixel clusters removed	14,8	280				
d)	10-pixel clusters removed	15,7	261				
e)	Grains with <2 neighbouring grains removed	16,1	250	16,6	248	17,5	231



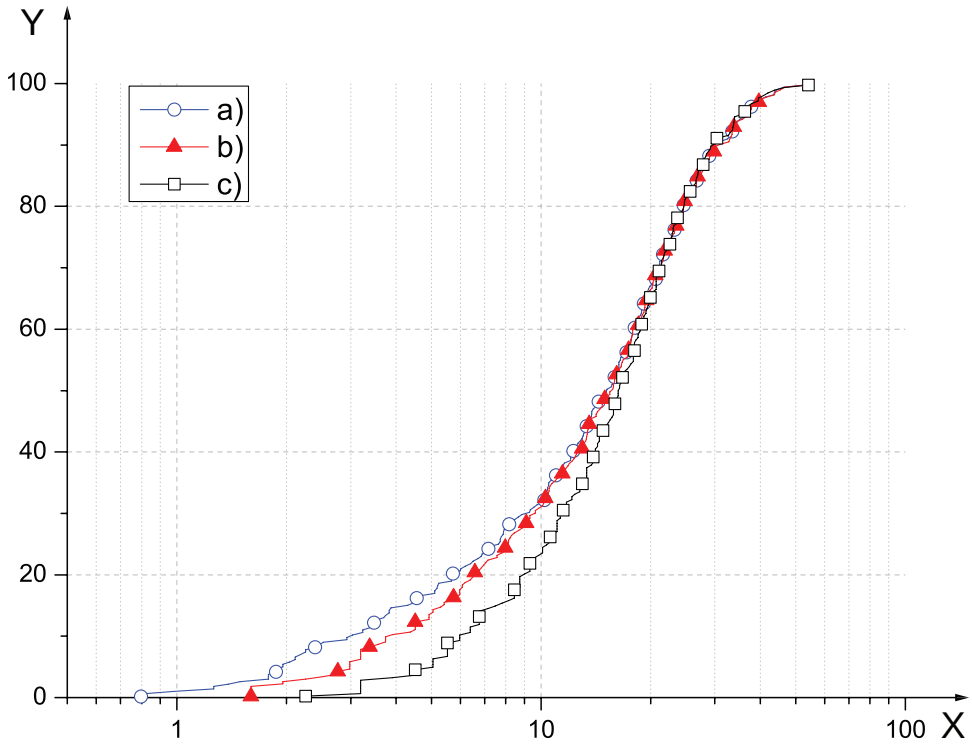
Key

X grain size (circle equivalent diameter), expressed in µm

Y cumulative probability, expressed in %

Figure 3 — Cumulative grain size distributions resulting from different map step sizes and single-pixel removal during data cleaning

Figure 4 shows cumulative grain size distributions resulting from maps of the same area produced with step sizes of a) 0,5 µm, b) 1 µm and c) 2 µm, [the 0,5 µm data is the same as that shown in Figure 2)] after data-cleaning steps involving the removal of grains with two or fewer neighbours and dilation into unindexed pixels where five neighbours were indexed.



Key

X grain size (circle equivalent diameter), expressed in µm

Y cumulative probability, expressed in %

Figure 4 — Cumulative grain size distributions resulting from different map step sizes and removal of grains with two or fewer neighbours during data cleaning

Following the recommendations in this International Standard, only the results for the 0,5 µm and 1 µm step sizes (with a step size <0,1 times the average grain size) should be considered after rejecting features with a size of less than 10 pixels or after removing grains with fewer than two neighbours. The agreement between these methods is better than 6 %.

6.4 Measurement of grain size

Grain size can be measured in many ways, of which the most commonly reported are diameters based on linear intercept, circle equivalent diameter or Feret diameters. Which of these is used depends on the application and whether comparison is being made with other existing measurement techniques.

The reference number of any standard used for metallographic evaluation of the grain size (see Annex A) shall be reported.

6.5 Representation of data

EBSD will give information on the size of all the resolved grains. The average grain size and, if appropriate, the size distribution (maximum, minimum and standard deviation) shall be reported and the uncertainty quoted at the 95 % confidence level.

If plotting the data graphically, consideration should be given to displaying both binned histograms and cumulative distributions, and also whether to plot by size as a function of number or by size as a function of number weighted by area.

7 Measurement uncertainty

The uncertainty shall be determined in accordance with ISO 21748.

Although a comprehensive round-robin exercise has not been performed, some exercises suggest that uncertainties in the average grain size of ±10 % at the 95 % confidence limit are not unreasonable.

Factors which contribute to the uncertainty include:

a) the linear calibration;

b) the resolution;

c) the step size;

d) the post-acquisition treatment of the raw data;

e) the specimen tilt angle;

f) specimen drift.

8 Reporting of analysis results

8.1 The reporting of the analysis results shall be done in accordance with ISO/IEC 17025.

8.2 The specimen preparation method and section analysed shall be clearly indicated, together with the reference number of any standard used for metallographic evaluation of the grain size (see Annex A).

8.3 The SEM and EBSD operating conditions shall be indicated. Indicate gun type, accelerating voltage, working distance, probe current (if available), specimen tilt angle and type of scan (beam/stage), step size, magnification (or image width) and grid shape (square or hexagonal). State the phase and crystallographic structures used for EBSD indexing. The report shall also state the criteria used for reliable indexing and the methods of data cleaning used (see also 8.4).

8.4 If requested by the customer, a map showing the raw data (the data prior to any data-cleaning operations) shall be displayed, to allow the customer to check for artefacts due to the data-cleaning procedures. Report all data-cleaning operations, with the percentage of points cleaned, and the minimum grain size and the misorientation angle used to define the boundaries.

Annex A
(informative)

Grain size measurement

The mechanical properties of engineering materials are strongly influenced by their crystal grain size and distribution. For example, strength, toughness and hardness are all important engineering properties that are strongly influenced by these parameters. It is therefore important to have standard methods, with commonly used and agreed terminology, for their measurement. Standardized methods of measurement of structure ensure that materials with repeatable properties are produced and the link between processing and structure is reinforced. In both single-phase and multiphase materials, the measurement of an average value of the grain size is vital, and there are existing and widely used standards for this purpose, such as ASTM E112[14], ASTM E930[15], ASTM E1181[16], ASTM E1382[17] and EN 623-3[18].

The factors which affect estimates of the grain size include the size and shape of the grains and the size distribution. In particular, determining the effect of the grain size distribution in the material can be important. Also, knowledge of how measurements in two dimensions are related to a three-dimensional shape distribution is crucial to the question of how uniformity (or non-uniformity) of structure relates to strength and toughness. The lack of an agreed method to measure the extent of uniformity is clearly a hindrance to optimized use. At present, subjective assessments are often made from an evaluation by eye of structural images. Improved measurement methods for this purpose will enhance the fundamental understanding of the link between grain size and grain size distribution and mechanical properties.

There are two popular methods in use to estimate the grain size. One method is based on the measurement of an equivalent circle diameter, D_{circle}, (see 3.3.2) and the other method on the use of linear intercepts (LIs). Understanding the comparability of different methods will assist trade and new product development through the presence of a common set of results. The LI method is based on the number-average lengths of intercepts through each crystal/grain along a line drawn across the material surface. The equivalent circle diameter method is based on the number-average area of the grains/crystals. The average area is converted to an equivalent circle diameter as a measure of size. De Hoff and Rhines, in the book "*Quantitative Microscopy*"[19], stated that an empirical relation had been found in measurements in aluminium and ferrous alloys where

$$\text{LI} = \sqrt{\bar{A}} \tag{A.1}$$

where $\bar{A}$ is the average grain area.

Thus, since $\bar{A} = \frac{\pi}{4}(D_{\text{circle}})^2$

$$D_{\text{circle}} = \sqrt{\frac{4}{\pi}}\ \sqrt{\bar{A}} = 1{,}13\sqrt{\bar{A}} = 1{,}13 \times \text{LI} \tag{A.2}$$

There is a reasonable probability that a proportion of small grains might not be visible in the nominally planar surface, either as a result of a loss or as a result of resolution limits. This is very difficult to quantify and its effect on the two measurement methods needs to be quantified.

Relevant microstructural standards are:

ASTM E112	Grain size
	Heyn linear-intercept method
	Jeffries planimetric method
ASTM E1181	Duplex grain sizes
ASTM E1382	Grain size using image analysis
ASTM E930	Largest grain size
EN 623-3	Grain sizes in ceramics

ASTM E1181 addresses some of the issues associated with grain size distribution. It discusses various typical examples, such as:

— isolated coarse grains in a finer-grained matrix;

— extremely wide distributions;

— bimodal distributions.

and mentions other difficult issues, such as:

— banded structures, necklace structures and systematic variations across sections of products.

ASTM E1181 also defines several procedures for estimation of the area fractions of coarse and of fine grains, including:

— a comparison procedure using graded area fraction comparison charts;

— a planimetric procedure to measure irregularly shaped regions;

— a point-counting procedure.

However, it advises that the estimation of area fractions of different sizes is subjective and prone to error. It states that the most effective method for measuring intercept distributions is to use a semi-automatic image analysis system with digitizing tablets and electronic pencils/cursors, using a test grid with five evenly spaced horizontal lines. The intercepts are classified by size and the data is presented as a histogram or frequency plot.

The linear-intercept technique is frequently used for measurements. This technique is popular because it also produces size distribution information. It is often found that the measured intercept number distribution is lognormal in form, which gives a straight line when plotted on lognormal probability paper[20]. Another option is measurement of the sectional grain area, as discussed by Vander Voort and Friel[21], but this depends on the production of suitable images for image analysis

The use of EBSD potentially provides a method which is complementary to traditional optical techniques, providing higher spatial resolution and enhanced information about the crystallography. It is, however, relatively slow and capital-intensive compared to optical methods.

Bibliography

[1] RANDLE, V, *Microtexture Determination and Its Applications*, Maney Publishing, 2003, ISBN 1902653831

[2] HUMPHREYS, F.J., Review: Grain and Subgrain Characterisation by Electron Backscatter Diffraction, *J. Mater. Sci.*, **36**, 2001, pp. 3833-3854

[3] MILES, R.E., On the elimination of edge effects in planar sampling, in *Stochastic Geometry*, editors E.F. Kendal and D.G. Kendall, Wiley, New York, 1974

[4] LANTUÉJOUL, C., On the estimation of mean values in individual analysis of particles, *Microscopica Acta 5*, 1980, pp. 266-273

[5] WRIGHT, S.I., A Parametric Study of Electron Backscatter Diffraction based Grain Size Measurements, *Practische Metallographie*, **47**, 2010, pp. 16-33

[6] HUMPHREYS, F.J., BATE, P.S., and HURLEY, P.J., Orientation averaging of electron backscattered diffraction data, *J. Microscopy*, **201**, 2001, pp. 50-58

[7] ENGLER, O., and RANDLE, V., *Introduction to Texture Analysis*, second edition, CRC press, 2009, ISBN 1420063650

[8] WRIGHT, S.I., and ADAMS, B.L., Automatic-analysis of electron backscatter diffraction patterns, *Metall. Trans. A*, 1992, **23**, pp. 756-767

[9] WRIGHT, S.I., and LARSEN, R.J., Extracting twins from orientation imaging microscopy scan data, *J. Microscopy*, **205**, 2002, pp. 245-252

[10] DAY, A.P., and QUESTED, T.E., Comparison of grain imaging and measurement using orientation and colour orientation contrast imaging, electron backscatter pattern and optical methods, *J. Microscopy*, **195**, 1999, pp. 186-196

[11] TRIMBY, P.W., PRIOR, D.J., and WHEELER, J., Grain boundary hierarchy development in a quartz mylonite, *J. Structural Geol.*, **20**, 1998, pp. 917-935

[12] VALCKE, S.L.A., PENNOCK, G.M., DRURY, M.R., and DE BESSER, J.H.P., Electron backscattered diffraction as a tool to quantify subgrains in deformed calcite, *J. Microscopy*, **224**, 2006, pp. 264-276

[13] NOLZE, G., Image distortions in SEM and their influences on EBSD measurements, *Ultramicroscopy*, **107**, 2007, pp. 172-183

[14] ASTM E112, *Standard Test Methods for Determining Average Grain Size*

[15] ASTM E930, *Standard Test Methods for Estimating the Largest Grain Observed in a Metallographic Section (ALA Grain Size)*

[16] ASTM E1181, *Standard Test Methods for Characterizing Duplex Grain Sizes*

[17] ASTM E1382, *Standard Test Methods for Determining Average Grain Size Using Semiautomatic and Automatic Image Analysis*

[18] EN 623-3, *Advanced technical ceramics — Monolithic ceramics — General and textural properties — Part 3: Determination of grain size and size distribution (characterized by the Linear Intercept Method)*

[19] DE HOFF, R.T., and RHINES, F.N., *Quantitative Microscopy*, McGraw-Hill, USA, 1968, pp. 239-241

[20] EXNER, H.E., Methods and Significance of Particle and Grain Size Control in Cemented Carbide Technology, *Powder Metall.*, **13 (26)**, 1970, pp. 429-448

[21] VANDER VOORT, G.F., and FRIEL, J.J., Image Analysis in Measurements of Duplex Grain Structures, *Materials Characterisation*, **29**, 1992, pp. 293-312

ICS 71.040.50
Price based on 18 pages