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Microbeam analysis — Guidelines for misorientation analysis to assess mechanical damage of austenitic stainless steel by electron backscatter diffraction (EBSD)

Analyse par microfaisceaux — Lignes directrices relatives à l'analyse des défauts d'orientation pour l'évaluation des dommages mécaniques de l'acier inoxydable austénitique par diffraction d'électrons rétrodiffusés (EBSD)

Reference number
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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Mechanical damage such as creep or fatigue, in engineering materials can be assessed by misorientation analysis using electron backscatter diffraction (EBSD) technique. The EBSD technique measures crystal orientation of sample surface by indexing EBSD patterns which are acquired by scanning its surface with electron beam in a scanning electron microscope (SEM). It can give orientation maps and misorientation maps. To determine the degree of damage induced in the materials, the misorientations calculated from the mapping data are qualified by various parameters such as the local misorientation, which is an averaged misorientation between neighbouring measurement points, and intra-grain misorientations, which is an averaged misorientation between the reference orientation assigned to each crystal grain and measurement points inside the grain. These misorientation parameters correlate well with the degree of mechanical damage caused by deformation, fatigue and/or creep. Therefore, the magnitude of the material damage can be estimated using the correlation curve which represents the relationship between the misorientation parameters and the degree of the damage (hereafter called correlation curve).

In the EBSD measurement, the crystal orientation is identified through electron beam illumination to the material surface, acquisition of the EBSD pattern by an image detector, and then crystal orientation identification by indexing of the EBSD patterns. It was shown that the point to point accuracy of the crystal orientation measurement is about 0,05° to 0,5°. The misorientation parameters vary depending on SEM conditions, observation conditions, EBSD pattern acquisition conditions and crystal orientation identification conditions. Several measurement parameters are determined for calculating the misorientation parameters. In particular, the local misorientation greatly depends on the distance between the measurement points (step size). Furthermore, the accuracy of the crystal orientation measurement and the definition of the misorientation parameters may depend on the hardware and software used for the measurement and analysis. There are several venders of commercial EBSD measurement and analysis systems. The correlation curve obtained for a certain condition using a certain measurement system is not always comparable with other master curve obtained with different conditions or systems. Therefore, it is necessary to have a standard to measure comparable master curves to show the degree of mechanical damage by using any EBSD systems.

This document describes measurement procedures and conditions and definitions of misorientation parameters independent on the measurement system in order to assess damage of austenitic stainless steel precisely.

Microbeam analysis — Guidelines for misorientation analysis to assess mechanical damage of austenitic stainless steel by electron backscatter diffraction (EBSD)

1 Scope

This document describes the guidelines for misorientation analysis to assess mechanical damage such as fatigue and creep induced by plastic and/or creep deformation for metallic materials by using electron backscatter diffraction (EBSD) technique. This international standard defines misorientation parameters and specifies measurement conditions for such mechanical damage assessment. This document is recommended to evaluate mechanical damage of austenitic stainless steel, which is widely used for various components of power plants and other facilities.

In this document, the mechanical damage refers to the damage which causes the fracture of structural materials due to external overload, fatigue and creep; excepting the chemical and thermal damages themselves.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 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.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
area averaged intra-grain misorientation
average of intra grain misorientation of all pixels in the measurement area

3.2
area averaged local misorientation
average of local misorientation of all pixels in the measurement area

3.3
blank point
non-indexed point (pixel) due to insufficient quality of the EBSD pattern

Note 1 to entry: This can occur for a variety of reasons, such as insufficient specimen surface condition, dust or contamination on the specimen surface, 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.

Note 2 to entry: See ISO 13067:2020, 3.4.2 for definition of non-indexing.

3.4
electron backscatter diffraction
EBSD
diffraction process that arises between the backscattered electrons and the crystal planes in a highly tilted crystalline specimen when illuminated by a stationary incident electron beam

[SOURCE: ISO 24173:2009, 3.7]

3.5
electron backscatter diffraction pattern
EBSD pattern
Kikuchi pattern like electron diffraction pattern which is generated on a phosphor screen or photographic film by backscatter diffracted electrons in a SEM

Note 1 to entry: A specimen is generally tilted to 70 degrees to get better quality of the diffraction pattern.

[SOURCE: ISO 24173:2009, 3.8, modified — The definition has been modified.]

3.6
grain averaged intra-grain misorientation
one value for each grain by averaging intra grain misorientations

3.7
grain averaged local misorientation
average of local misorientation of all pixels in a grain

3.8
grain boundary
lines between grains in an EBSD orientation map

Note 1 to entry: Grains are defined by grouping connected neighbour pixels which misorientation is less than the specified tolerance angle.

[SOURCE: ISO 13067:2020, 3.2.1, modified — The definition has been modified.]

3.9
Hough transform
mathematical transformation of image processing techniques, which converts a line in an image to a point

Note 1 to entry: This allows automated detection of bands in an EBSD pattern.

Note 2 to entry: In EBSD, a linear Hough transform is used to identify the position and orientation of the Kikuchi bands in each EBSD pattern, which enables the EBSD pattern to be indexed. Each Kikuchi band is identified as a bright spot in Hough space. The Hough transform is essentially a special case of the Radon transform. Generally, the Hough transform is for binary images, and the Radon transform is for grey-level images.

[SOURCE: ISO 24173:2009, 3.12, modified — The definition has been modified.]

3.10
indexing reliability
numerical value that indicates the confidence/reliability of indexing result which indexing software place in automatic analysis procedure

Note 1 to entry: 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.

3.11
intra-grain misorientation
misorientation of each pixel with the average orientation of the grain

Note 1 to entry: See Figure 3.

Note 2 to entry: A map that displays the deviation of a pixel to a reference orientation

3.12
local misorientation
average misorientation between the measured pixel (P_1) and neighbouring pixels

Note 1 to entry: See Figure 2.

Note 2 to entry: When the misorientation between the measured pixel (P_2) and neighbour pixel exceeds the threshold angle like the measured pixels at the grain boundary as shown in Figure 2, these pixels are excluded from the misorientation calculation.

3.13
master curve
correlation curve obtained experimentally between misorientation parameter and mechanical damage degree

Note 1 to entry: It is used to estimate damage degree quantitatively.

3.14
minimum grain size
number of pixels required to constitute a grain

Note 1 to entry: If the sum number of measurements constituting a grain are less than this value then the grain is excluded.

3.15
misorientation
angle/axis pair, required to rotate one set of crystal axes into coincidence with the other set of crystal axes for the given two crystal orientations

Note 1 to entry: The smallest angle used here.

3.16
misorientation parameter
parameter calculated from misorientation such as “local misorientation”, “intra-grain misorientation”

Note 1 to entry: It is classified as 3 groups; parameter for each pixel, grain or area.

3.17
pattern quality
measure of the sharpness of the diffraction bands or the range of contrast within a diffraction pattern

Note 1 to entry: Different terms are used in different commercial software packages, including, for example, band contrast, band slope and image quality.

3.18
pixel
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

[SOURCE: ISO 13067:2020, 3.1.2]

Note 1 to entry: This is different from "camera pixels".

3.19
scanning grid
pattern of spacing of measurement points

Note 1 to entry: A regular hexagonal grid or a regular square grid is adopted generally. A hexagonal (square) grid means that the individual points making up the scan area are situated on a hexagonal (or square) array.

3.20
step size
distance between adjacent points from which individual EBSD patterns are acquired during collection of data for an EBSD map

[SOURCE: ISO 13067:2020, 3.1.1]

4 Abbreviated terms

CCD	charge coupled device
CMOS	complementary metal-oxide semiconductor
EBSD	electron backscatter diffraction
EBSP	electron backscatter diffraction pattern
SEM	scanning electron microscope/microscopy
WD	working distance

5 Equipment for EBSD measurement

See ISO 24173:2009, Clause 4.

5.1 SEM, EPMA or FIB instrument, fitted with an electron column and including controls for beam position, stage, focus and magnification.

5.2 Accessories, for detecting and indexing electron backscatter diffraction patterns, including:

5.2.1 Phosphorescent ("phosphor") screen, which is fluoresced by electrons from the specimen to form the diffraction pattern.

5.2.3 Image acquisition device, with low light sensitivity, for viewing the diffraction pattern produced on the screen.

5.2.4 Computer, with image processing, computer-aided pattern indexing, data storage and data processing, and SEM beam (or stage) control to allow mapping.

NOTE Modern systems generally use charge-coupled devices (CCDs) or complementary metal-oxide semiconductor (CMOS).

6 Preparation

6.1 Calibration

The procedures described in ISO 24173 shall be followed.

6.2 Specimen preparation

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

The procedures set out in ISO 24173:2009, Annex B shall be followed.

For specimen preparation for EBSD analysis, the following equipment can be required (depending on the types of specimen to be prepared, ISO 24173:2009, Annex B):

— cutting and mounting equipment;

— mechanical grinding and polishing equipment;

— electrolytic polisher;

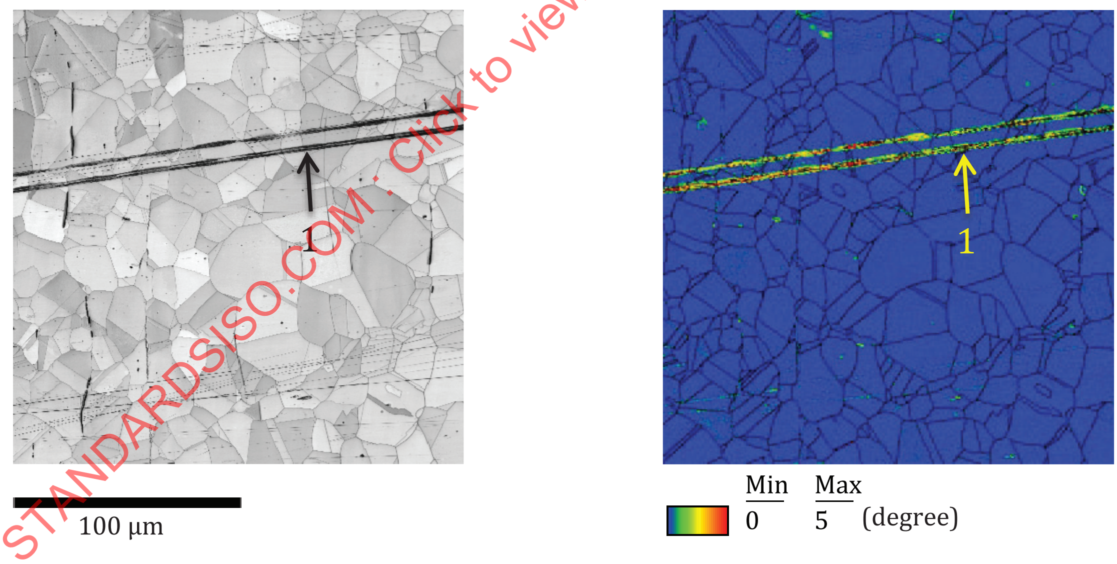
— ultrasonic cleaner;

— ion-sputtering equipment; and

— coating equipment.

It should also be considered to avoid any phase transformation during specimen preparation.

Undesired damage on the specimen surface shall be removed carefully. In order to obtain the desired damage-free surface for misorientation analysis, final polishing using colloidal silica or electro-polishing is effective. If scratches remain on the surface, they cause larger misorientation as shown in Figure 1



a) Pattern quality map

b) Local misorientation map

Key

1 scratches

Figure 1 — Example of remaining scratches

7 Measurement procedures

7.1 Setting SEM operating conditions

7.1.1 Accelerating voltage

Accelerating voltage ranging between 10 kV and 20 kV is recommended to get reasonable EBSD patterns. Increasing the accelerating voltage may result in increasing beam spread in the specimen, and hence make the spatial resolution worse. It depends on the specimen Z number, but in most cases, there is no merit to use higher accelerating voltage more than 20 kV with recent high sensitivity image detectors.

See ISO 24173:2009, 5.3.1.

7.1.2 Probe current

Increasing the probe current increases the number of electrons contributing to the diffraction pattern and it will give brighter EBSD patterns. This improves the signal/noise ration of the EBSD patterns resulting in better band detection and better orientation determination. Therefore, it allows shorter camera exposure time, namely faster mapping.

See ISO 24173:2009, 5.3.1.

7.1.3 Magnification observation

Depending on the observation's purpose, it is recommended that the measurement area is nearly equal to the observation area because of that the effective probe diameter depends on SEM's magnification.

The measurement area should be set to include more than about 100 grains to avoid effects of individual grains becoming too large. Therefore, the magnification should be set between 300 and 500 times in case of that the grain size (diameter) is about couple of 10 µm.

7.1.4 Working distance

The ideal working distance for EBSD is the working distance at which the brightest region of the raw EBSD pattern (i.e. without background correction) becomes nearly at the centre of the phosphor screen. It is set at around 15 mm in general case, but it can be changed depending on each SEM/EBSD system configuration. Short working distances generally improve the spatial resolution of EBSD measurements, although additional care shall be paid to avoid collisions between the specimen and the pole-piece or the backscatter detector (if present).

See ISO 24173:2009, 5.3.2.

7.1.5 Focus

EBSD measurement will be done with a highly tilted specimen in a SEM. Therefore, the focus can vary depending on the beam position on the specimen (see ISO 24173). The dynamic focus is recommended to be used to avoid the out of focus condition at upper and lower of measurement area.

See also ISO 24173:2009, 5.3.

7.2 Setting the EBSD measurement conditions

7.2.1 Background correction

EBSD patterns generally have a bright centre and become much darker near the corners. The brightness of raw EBSD pattern images decrease seriously in the surrounding area. Background correction should be used to make the “raw” EBSD pattern image into ones with more uniform brightness across its whole area with better local contrast.

See ISO 24173:2009, 5.3.6.

7.2.2 Binning

The number of camera pixels, which form EBSD pattern image acquired through an image detector can be adjusted by binning setting. If the image size becomes smaller, it means binning is set larger. Then the time required for measurement becomes shorter, though the accuracy of orientation measurement can become lower in general. Large binning does not always get the faster measurement speed either, because of that the measurement speed is sometimes limited by the image processing speed or data transfer speed. The accuracy of orientation measurement for distinguishing about 0,5° orientation difference, can be acquired by setting the binning to the image size between 100 × 100 and 200 × 200 camera pixels.

See ISO 24173:2009, 5.3.4.

NOTE The binning is applicable to CCDs cameras and not applicable to CMOS cameras.

7.2.3 Pattern averaging

Quality of EBSD pattern image can be improved by averaging patterns collected more than one frame at the same measurement point. However, the pattern averaging makes the measurement speed slower a lot. For this reason, it is recommended to increase the probe current to acquire the same quality of EBSD patterns, instead of using pattern averaging. The quality of EBSD pattern is also controlled by adjusting the binning size and the gain of the image detector.

See ISO 24173:2009, 5.3.5.

7.2.4 Hough transform

Band detection during EBSD refers to the automatic detection of Kikuchi bands in an EBSP via use of a Hough transform. Hough transformation technique is used to extract bands from an EBSD pattern acquired by an image detector. A suitable set of Hough transformation parameters should be set depending on the features of EBSD patterns. These parameters may affect to the speed and the accuracy of Hough transformation calculation.

See ISO 24173:2009, 5.3.7.

7.2.5 Measurement area

It is recommended to include reasonable number of grains, it is expected more than 100 grains as noted in 7.1.3, in the measurement area to evaluate the average status of polycrystalline materials. If the measurement area becomes larger, then the time for measurement becomes longer. In this case, it might be needed to consider about the stability of SEM’s such as the beam drift or specimen drift.

7.2.6 Step size

This depends on the type of specimen and the purpose of measurements. Reasonable number of measurement points needs to be included in each grain. As the step size becomes smaller, more detail distribution of misorientation can be obtained, though the time for measurement becomes longer. Also, the resolution limit of EBSD should be taken into account to decide the step size. It should not be smaller the resolution limit. Though it depends on specification of SEMs and measurement conditions, EBSD’s resolution limit is estimated about couple of 10 nm.

To compare the local misorientation, it is necessary to set the step size same. Because the local misorientation depends on the step size.

7.2.7 Scanning grid

Either the square grid or the hexagonal grid can be chosen in some software. Both types of grid are usable for misorientation calculation, however use of the same grid type is recommended to compare the results.

8 Calculation of misorientation

8.1 Defining grains

8.1.1 General

Misorientation is calculated between two crystal orientations of measured pixels. Misorientation between two crystal orientations of measured pixels in different grains is not considered to be related to the damage discussed here. Therefore, the misorientation should be calculated among the crystal orientations of pixels within a same grain. For this reason, the grains must be defined before calculating misorientation data.

8.1.2 Setting the misorientation to define grains

Grain is a group of connected pixels under the condition that the misorientation between neighbour pixels is less than the tolerance angle (angle for defining grains). The tolerance angle of 5° is recommended to define the grains. The tolerance angle of 15° was checked and it was considered too large to define the grains, because of that the cases where obviously independent two grains were connected under this condition were often observed.

A 5° a grain definition angle is recommended.

8.1.3 Setting of minimum grain size

It is recommended to eliminate small grains which are consisted of 9 pixels or less. Such small grains can produce some biased misorientation data.

8.1.4 Caution

Intra-grain misorientation, local misorientation, averaged intra-grain misorientation and averaged local misorientation are calculated based on defined grains, so it is affected by the parameters to define grains, such as the grain definition angle, minimum grain size, or grain diameter itself. When the grain-based misorientation are evaluated, it is recommended to check how the parameters affect these misorientation values by changing the grain definition angle or minimum grain size. When the specimen has some sub-grain structure, special attention should be paid to calculate grain based misorientation. Some grain based misorientation becomes larger as the grain size becomes larger, so some reasonable caution should be paid when materials with different grain size are compared.

8.2 Data screening

8.2.1 Evaluation of reliability of measured data

When EBSD (map) data is acquired, some pixels were not indexed correctly due to poor quality of EBSD pattern images, which results in wrong or unreliable orientation data. Sometimes, these continuous pixels with wrong or unreliable orientation data can form small grains. If the minimum grain size is defined by 10 pixels, the pixels with lower reliability are eliminated automatically from misorientation calculation.

The pixels can also be eliminated with lower reliability by setting some tolerance using pattern quality or indexing reliability. However, these settings are not necessary in general when the minimum grain size is defined.

8.2.2 Treatment of blank pixels

The eliminated pixels are not used in misorientation calculation and result in blank points. The amount of blank points can be increased when damage level becomes higher or sometimes specimens were not prepared correctly. It is possible to replace these blank points by data cleaning procedure and fill the blank points in the orientation map by indexed points. But data cleaning procedure can introduce artefacts. Data cleaning procedure should not be used to get more accurate results.

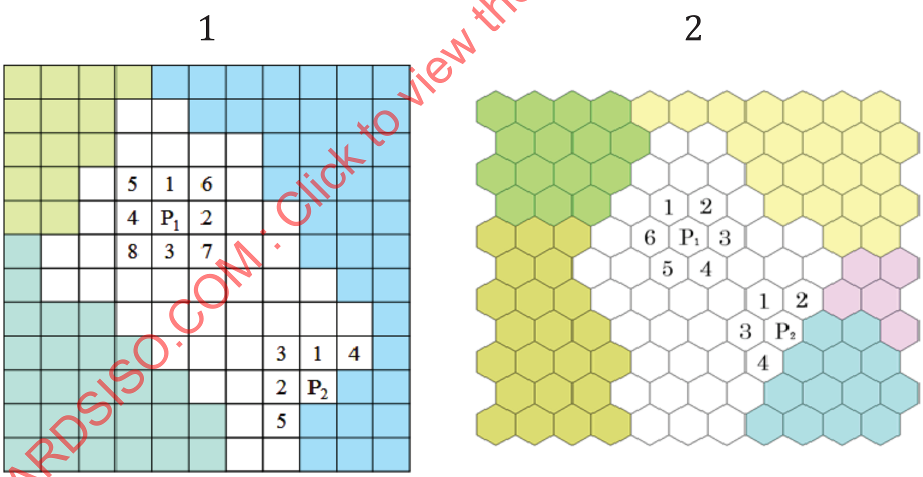
8.3 Calculation of misorientation parameters

Terms of misorientation parameters are defined in Clause 3. Misorientation parameters in discussion are calculated based on pixels, grains and area such as scan area.

a) Local misorientation

Local misorientation is calculated by averaging the misorientation between the measured pixel(P_1) and neighbour pixels with the same distance from the measured pixels. Figure 2 shows the schematic illustration for local misorientation in both cases of square grid and hexagonal grid. Formulae (1), (2) and (3) show how to calculate the local misorientation.

There are other options for local misorientation calculation in case of square grid. All 8 pixels surrounding P_1 can be used in the calculation. When the misorientation between the measured pixel and neighbour pixel exceeds the threshold angle like the measured pixels at the grain boundary as the pixel P_2 shown in Figure 2, these pixels are excluded from the misorientation calculation.



Key

1 square grid

2 hexagonal grid

Figure 2 — Schematic illustration for local misorientation calculation in both square grid and hexagonal grid

for square grid:

$$LM = \frac{\sum_{i}^{4} \beta(P_1, i)}{4} \tag{1}$$

$$LM = \frac{\sum_{i}^{8} \beta(P_1, i)}{8} \tag{2}$$

for hexagonal grid:

$$LM = \frac{\sum_{i}^{6} \beta(P_1, i)}{6} \tag{3}$$

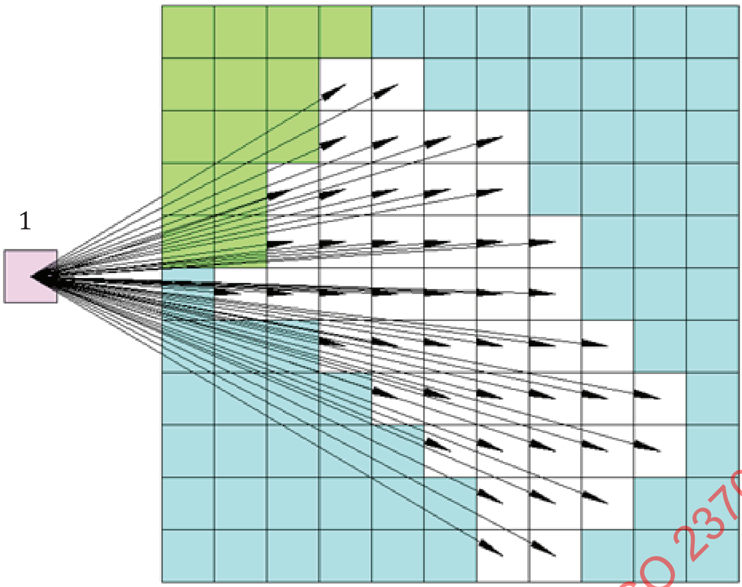
where

LM	is the local misorientation;
β (A, B)	is the misorientation between A and B;
P_1	is the point of interest.

b) Intra-grain misorientation

Intra-grain misorientation is the relative misorientation of each pixel with the average orientation of the grain. Figure 3 shows the schematic illustration for intra-grain misorientation. The idea for the calculation of intra-grain misorientation is same in both square grid and hexagonal grid.

There are other options to set the reference orientation. The orientation of the pixel with the minimum local misorientation can be set as the reference orientation.



Key

1 reference orientation (grain average orientation)

Figure 3 — Schematic illustration for intra-grain misorientation calculation

c) Grain averaged local misorientation

Grain averaged local misorientation is calculated by averaging local misorientation value of all pixels in the grain. It then becomes one value for one grain. This means all misorientations among pixels in the grain are averaged.

d) Grain averaged intra-grain misorientation

Grain averaged intra-grain misorientation is calculated by averaging intra-grain misorientation value of all pixels in the grain. It then becomes one value for one grain. This means all misorientations among pixels in the grain are averaged.

e) Area averaged local misorientation

Area averaged local misorientation is calculated by averaging local misorientation value of all pixels in the measurement area.

f) Area averaged intra-grain misorientation

Area averaged intra-grain misorientation is calculated by averaging intra-grain misorientation value of all pixels in the measurement area.

9 Material damage assessment

9.1 General

Material damage is assessed using the misorientation parameters by qualitative or quantitative manner. In the qualitative assessment, mapping data of the misorientation parameters is used to characterize the material damage and its distribution. On the other hand, in the quantitative assessment, the misorientation parameter is calculated for each mapping data to represent the degree of the material damage. The following misorientation parameters are recommended. It should be noted that small damage can be difficult to detect due to resolution of misorientation analysis. On the other hand, relatively large damage can deteriorate the EBSD pattern and makes it difficult to identify the misorientation.

9.2 Misorientation parameter for qualitative assessments

The local misorientation is recommended for assessing the material damage. Since the local misorientation reflects local gradient of the crystal orientation, it correlates with density of the geometrically necessary dislocations. It should be noted that the local misorientation depends on the step size for crystal orientation measurement.

The intra-grain misorientation is the misorientation from the reference orientation and its magnitude depends on choice of the reference orientation. Therefore, the intra-grain misorientation does not correlate with the dislocation density directly.

9.3 Misorientation parameter for quantitative assessments

The average of the local misorientations in measured area (area averaged local misorientation) and that of the intra-grain misorientation (area averaged intra-grain misorientation) represent the degree of the material damage for the measured area. If the size of the measured area is large enough for averaging the microstructural inhomogeneity of material damage, the area averaged local and intra-grain misorientations correlate with the degree of the material damage. The area averaged intra-grain misorientation is recommended because the intra-grain misorientation is hardly affected by the step size and not much affected by sample preparation, SEM observation and crystal measurement conditions.

By preparing the relationship between the degree of material damage and the magnitude of the misorientation parameters, the material damage can be quantified from misorientation parameters identified by the EBSD measurement. Once the master curve is obtained for the area averaged intra-grain misorientation, the curve is applicable for various measurement conditions because the intra-grain misorientation is hardly affected by the measurement conditions.

In Annex A, round robin measurements were made for creep damaged stainless steels. It shows that, when the measurement was made according to the prescribed procedure, the area averaged intra-grain misorientations were almost identical regardless of SEM and EBSD systems used for the measurements.

10 Report

Record the following measurement conditions as a measurement record.

It is not easy to write down all measurement conditions; for formatting, refer to Table 1 as an example.

a) general:

   1) a reference to this document, i.e. ISO 23703;

b) sample preparation:

   1) sample type;

   2) how to cut off, dimension;

   3) how to polish;

   4) how to fine polish;

c) observation condition:

   1) SEM specification;

   2) accelerating voltage;

3) beam current;

4) working distance;

5) dynamic focus;

6) observation magnification;

d) EBSD measurement:

1) date (year/month/day);

2) laboratory;

3) measurement system;

4) measurement camera;

5) software;

6) camera settings;

7) measurement camera pixel size (applied camera pixels actually);

8) measurement area;

9) step size;

10) grid (square grid or hexagonal grid);

11) Hough transform;

e) misorientation calculation:

1) software;

2) data screening condition;

3) tolerance angle for grain boundary definition;

4) minimum grain size.

Table 1 — EBSD measurement conditions[a]

		Standard conditions		Examples		
		Condition A	Condition B	Example for OIM	Example for HKL	Example for CrystAlign
Observation conditions	SEM	–	–	Carl Zeiss ULTRA55	JSM-7001F	Carl Zeiss Merlin
	Accelerating voltage (kV)	15	15	15	20	15
	Beam current	10	10	Medium	Current 10 Aperture 4	High
	WD (mm)	15	15	15	17~31	20
	Dynamic focus	Yes	Yes	Yes	Yes	Yes
	Magnification	–	-	× 400	× 400	× 400

Table 1 *(continued)*

		Standard conditions		Examples		
		Condition A	Condition B	Example for OIM	Example for HKL	Example for CrystAlign
Orientation measurement conditions	Measurement system	–	–	TSL OIM	HKL Channel5	CrystAlign
	Measurement camera	–	–	HIKARI	NordlysNano	e-FlashHR
	Software	–	–	OIM Version 5.2	AZtecHKL V2.2	ESPRIT2.0
	Gain (dB)	10	5	5	–	100
	Black level	50	50	2,61	–	–
	Exposure	–	–	0.1 sec	10 msec	7 msec
	Binning	8 × 8	1 × 1	8 × 8	8 × 8	20 × 20
	Pixel	-	-	80 × 60	168 × 128	80 × 60
	Background subtract	Yes	Yes	Yes	Yes	Yes
	Auto contrast	Yes	Yes	No	Yes	No
	No. of frames	–	–	-	2	–
	Target material	–	–	FCC and BCC	FCC and BCC	FCC and BCC
	Range (μm x μm)	–	–	200 × 200	200 × 200	200 × 200
	Step size (μm)	–	–	0,5	0,5	0,5
	Grid	–	-	Hexagon	Square	Square
Hough transform	Binned pattern size	90	120	120	-	AUTO
	Rho fraction (deg)	85	85	85	–	AUTO
	Max/Min peak count	8/3	8/3	8/3	8	AUTO
	Min peak magnitude	5	5	5	–	AUTO
	Min peak distance	23	23	23	–	AUTO
	Peak symmetry	0.5	0.5	0.5	–	AUTO
	Theta step size	1	0.5	0.5	–	AUTO
Data analysis conditions	Software	–	–	OIM Version 5.2	AZtecHKL V2.2	ESPRIT2.0
	Threshold for data screening	–	–	CI ≥ 0.1	–	Max. BMM 2
	Tolerance angle for grain boundary	5°	5°	5°	5°	5°
	Minimum number of pixels for single grain	9	9	9	9	9
	Data clean up	No	No	No	No	No

[a] Examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

Annex A
(informative)

Round robin crystal orientation measurement for damage assessment

A.1 General

In Reference [2], crystal orientations of creep damaged Type 316 stainless steel were measured by 10 organizations using the same specimens, passed in a round robin, in order to investigate the scatter in material damage assessment using the EBSD technique. The measurements were performed according to this guideline. Two misorientation parameters, the local and intra-grain misorientations, were calculated using mapping data of measured crystal orientations. It was concluded that the area averaged intra-grain misorientation can be used for measurement of the creep damage (plastic and creep strain). The empirical relationship between the area averaged intra-grain misorientation and the degree of the creep damage, which are denoted as the master curve in this guideline, can be shared regardless of the SEM and EBSD system used. The summary of the round robin measurement is presented in this annex.

A.2 Observed specimens and measurement conditions

The material used for the round robin measurement was Type 316 austenitic stainless steel. The chemical composition is shown in Table A.1. Solution heat-treatment was conducted by water quenching after holding 1 h at 1 343 K. Test specimens, whose geometry is shown in Figure A.1, were machined from the material and subjected to creep tests at 973 K with a constant stress of 100 MPa. The creep life for this test conditions was 1 108,1 h. Then, the tests were interrupted at scheduled times between 267,7 h to 694,4 h as shown in Table A.2. In this way, seven kinds of interrupted specimens and one non-tested specimen were prepared for the EBSD measurement. The magnitude of plastic and creep strain (hereafter called "strain") at the end of the tests is shown in Table A.2.

After the creep tests, gage sections of the specimens were prepared for the round robin measurement. The surface was polished using up to #2 000 emery paper followed by 9, 3 and 1 µm diamond paste polishing and finished by colloidal silica (0,04 µm) polishing in order to obtain flat surfaces free from damage induced by machining and the paper and paste polishing.

The crystal orientation measurements were conducted by 10 organizations: IHI Corporation (A), Institute of Nuclear Safety System (B), TSL Solutions K. K. (C), National Institute for Occupational Safety and Health, Japan (D), Chubu Electric Power Co. (E), Nippon Steel & Sumikin Technology Co. (F), Niigata Institute of Technology (G), Iwate University (H), Kobelco Research Institute (I) and Bruker AXS K.K. (J). The EBSD systems used for the measurements were the OIM® (A, B, C, D, E and F), HKL® (G, H and I) and CrystAlign® (J) systems. The measurement conditions are summarized in Table A.3. The common step size of 0,5 µm was applied for all organizations. The size of the measured area was 200 × 200 µm^2 except for one organization (J).

A.3 Measurement results

A.3.1 Crystal orientations

Figure A.2 shows the mapping of the EBSD pattern quality parameter, which is referred to as the image quality (IQ) for the OIM, band contrast (BC) for the HKL and pattern quality (PQ) for the CrystAlign systems, obtained using the non-tested specimen. It is possible to know the quality of the SEM and diffraction observation conditions from the pattern quality parameter. For example, if the SEM observation is a bit out of focus, a blurred grain boundary is seen in the pattern quality mapping. The mapping results shown in Figure A.2 indicate that the SEM observation and EBSD pattern acquisition were fine for all measurements.

The measured crystal orientations for the specimen of 28,0 % strain are shown by maps in Figure A.3. The crystal orientations are indicated using the colour code. The grain boundaries can be distinguished by the discontinuous change in the crystal orientation. The grain boundaries were determined using the tolerance angle of 5°. The crystal orientation varied slightly in the same grain. So-called geometrically necessary dislocations were accumulated in order to account for inhomogeneous deformation of each crystal grain. The material had an approximately equiaxial grain structure and the 28,0 % strain caused elongation of the crystal grains in the loading direction. It should be noted that the texture was not significant even if 44,6 % strain was accumulated.

A.3.2 Misorientation parameters

Two kinds of misorientation parameters were calculated using the measured crystal orientations: local misorientation and intra-grain misorientation. The local misorientation is the averaged misorientation between neighbouring measurement points and referred to as the local misorientation (LM) for the HKL and the kernel averaged misorientation (KAM) for the OIM and CrystAlign systems. The intra-grain misorientation is the averaged misorientation between the reference orientation assigned to each crystal grain and the orientations at measurement points inside the grain and referred to as the grain reference orientation deviation (GROD) for OIM, grain misorientation (GMO) for HKL and MO average for CrystAlign. The reference orientation was determined as the average of all crystal orientations measured in the grain. These misorientation parameters were calculated for each measurement point. As shown in Figure A.3, the crystal orientation was not homogeneous even in the same grain when the strain was large. The degree of the inhomogeneity can be quantified by the misorientation parameters. In order to derive the representative misorientation parameter for each mapping data set, the misorientation parameters are averaged for the all points in the map. The averaged values of the local and intra-grain misorientations are then defined as the area averaged local and intra-grain misorientations, respectively. It should be noted that the area averaged intra-grain misorientation is not referred to as averaged GROD but to as grain orientation spread (GOS) in OIM.

Figure A.4 shows the change in the area averaged local misorientation with the magnitude of the stain. Since the KAM obtained by the CrystAlign system is divided by the step size, which was 0,5 µm, the KAM values shown in Figure A.4 were multiplied by 0,5 in order to compare the results obtained using OIM and HKL systems. The area averaged local misorientation increased almost monotonically. The inhomogeneous orientation due to the creep damage (strain) was successfully quantified by the area averaged local misorientation. The degree of the creep damage can be estimated using the correlation curve (master curve). However, the correlation showed eminent scatter. The misorientation obtained by organization I tended to be larger than that obtained by the other organizations. On the other hand, the data obtained by C, D, E and F showed relatively small misorientations. The difference in the area averaged local misorientation was about 0,2°. Therefore, for example, when the area averaged local misorientation of 0,4° is obtained for a damaged material, the accuracy of the damage estimated is expected to be more than 10 % in strain.

It is worth mentioning that the local misorientation did not seem to depend on the EBSD system vender. The measurement position followed the hexagonal grid for the OIM system, whereas the square gird was applied for the HKL and CrystAlign systems. The difference in the grid shape did not bring about any significant influence on the local misorientation. It was also revealed that the parameters KAM and LM can be treated as the same parameter; the local misorientation.

The relationship between the area averaged intra-grain misorientation and the magnitude of the strain is shown in Figure A.5. The area averaged intra-grain misorientation also exhibited monotonic increase against the strain. Furthermore, the scatter band was relatively narrow compared with that for the area averaged local misorientation. The strain estimation using the area averaged intra-grain misorientation is expected to be more accurate.

A.4 Discussion

The area averaged local misorientation showed eminent scatter even for the non-tested specimen. The scattering might be attributed, not to the measurement, but to material local inhomogeneity. The size of the measured area was 200 µm × 200 µm or 300 µm × 225 µm and the position of the measurement was chosen arbitrarily from the specimen surface, which was approximately 18 mm × 18 mm. Therefore, the actual local misorientation might differ area by area. However, the area averaged intra-grain misorientation for the non-tested specimen was almost the same for all data sets. This implies that the scatter in the area averaged local misorientation was mainly brought about by the EBSD measurement itself.

The scatter in the area averaged intra-grain misorientation was relatively small. The magnitude of the intra-grain misorientations is relatively larger than that of the local misorientations. The area averaged intra-grain misorientation of the creep damaged specimens was more than 1° whereas the area averaged local misorientation was less than 0,8°. The misorientation from the reference orientation tends to be larger than the misorientation between the neighbouring points. The influence of the error in the crystal orientation measurement on the intra-grain misorientation becomes relatively small if the misorientation error is the same. Furthermore, the influence of the error is reduced by the area averaging for the intra-grain misorientations. Thus, the intra-grain misorientation was not affected so much by the error in the crystal orientation measurement.

Since the correlation between the area averaged intra-grain misorientation and the magnitude of the strain was clear, it is possible to estimate the degree of strain accurately using the area averaged intra-grain misorientation. The master curve did not depend on the SEM and EBSD systems. From the range of scattering of the area averaged local misorientation, the error in the local misorientation was about 0,2° at the maximum case. Since the magnitude of the area averaged intra-grain misorientation was much larger than 0,2° for the creep damaged specimen, the influence of the error is not significant for estimating the magnitude of strain. It should be noted that the area averaged intra-grain misorientation does not depend on the step size, while the local misorientation greatly depends on the step size.

Table A.1 — Chemical content of test material (mass fraction %)

Fe	Mn	P	S	Ni	Cr	Mo
Bal.	1,26	0,029	0,011	12,09	17,25	2,05

Table A.2 — Interruption times for creep tests and corresponding plastic and creep strain

Interruption time / h	267,7	487,5	506,5	528,0	561,7	583,8	694,4
Strain / %	7,4	14,2	21,6	24,9	18,4	28,0	44 6

Table A.3 — Summary of SEM observation and EBSD measurement conditions (1 of 2)[a]

System	OIM					
Organization	A	B	C	D	E	F
CCD camera	HIKARI	HIKARI	DigiView IV	DigiView IV	HIKARI	DigiView IV
SEM	Philips XL30	Carl Zeiss ULTRA55	JEOL JSM-7001F	Elionix ERA-8800FE	JEOL JSM-7001F	Hitachi SU6600
Accelerating voltage (kV)	20	20	15	20	20	20
Beam current	unknown	Medium	– 10 nA	1600	15	Medium 5
WD (mm)	15	15	– 16	12 – 17	25	15
Gain dB	10,1	11	unknown	2,1 – 4,7	0,99	3,29
Black level	4	2,61	unknown	1,91	0,96	0,21
Exposure (ms)	60,3	3,35	7,74	0.03	3.0	7,68
Binning	4 × 4	8 × 8	8 × 8	8 × 8	5 × 5	8 × 8

Table A.3 *(continued)*

Pixel	160 × 120	80 × 60	128 × 120	128 × 120	128 × 96	174 × 130
Background subtract	Yes	Yes	Yes	Yes	Yes	Yes
Auto contrast	No	No	Yes	No	No	No
Binned pattern size	120	120	120	120	120	96
Rho fraction	85°	85°	90°	85°	90°	90°
Peak count (max/min)	8/3	8/3	8/3	8/3	7/3	9/3
Min peak magnitude	5	5	5	5	5	5
Min peak distance	23	23	unknown	23	23	20
Peak symmetry	unknown	unknown	~0.8	0.5	0.75	0.72
Theta step size	1°	1°	1°	1°	1°	1°
Range (µm × µm)	200 × 200	200 × 200	200 × 200	200 × 200	200 × 200	200 × 200
Step size (µm)	0,5	0,5	0,5	0,5	0,5	0,5
Grid	Hexagon	Hexagon	Hexagon	Hexagon	Hexagon	Hexagon
Dynamic focus	No	Yes	Yes	No	Yes	Yes
Magnification	× 400	× 400	× 500	× 400	× 500	× 400

[a] Examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

Table A.3 — Summary of SEM observation and EBSD measurement conditions (2 of 2)[a]

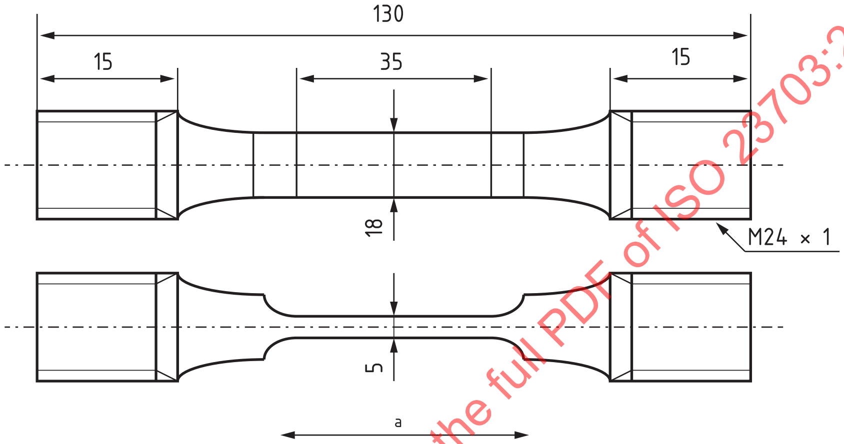
System	HKL Channel5			CrystAlign
Organization	G	H	I	J
CCD camera	NordlysNano	NordlysNano	Nordlys	e-FlashHR
Software	HKL Channel5 SP11	AZtecHKL V2.2	HKL Channel5	ESPRIT2.0
SEM	JEOL JSM-6510LV	JEOL JSM-7001F	Hitachi SU-70	ZEISS Marlin
Accelerating voltage (kV)	20	20	20	15
Beam current	SS70 Aperture 2	Current 10 Aperture 4	Current 5 Aperture 3	High
WD (mm)	19	17 – 31	15 – 16,7	20
Gain (dB)	—	—	—	100
Black level	—	—	—	—
Exposure (ms)	8	10	10	7
Binning	8 × 8	8 × 8	8 × 8	—
Pixel	168 × 128	168 × 128	168 × 128	80 × 60
Background subtract	Yes	Yes	Yes	Yes
Auto contrast	Yes	Yes	Yes	No
Peak count (max/min)	6/4	8	6/4	Auto
Min peak magnitude	—	—	—	Auto
Min peak distance	—	—	—	Auto
Peak symmetry	—	—	—	Auto
Theta step size	—	—	—	Auto
No. of frames	2	2	1	—

Table A.3 *(continued)*

Range (µm × µm)	200 × 200	200 × 200	200 × 200	300 × 225
Step size (µm)	0,5	0,5	0,5	0,5
Grid	Square	Square	Square	Square
Dynamic focus	Yes	Yes	No	Yes
Magnification	× 200	× 300	× 400	× 315

[a] Examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

Dimensions in millimetres



Key

a Rolling direction.

Figure A.1 — Creep test specimen