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

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Foreword

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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 — Guideline 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 terminological 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 (P1) and neighbouring pixels

Note 1 to entry: See Figure 2.

Note 2 to entry: When the misorientation between the measured pixel (P2) 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.

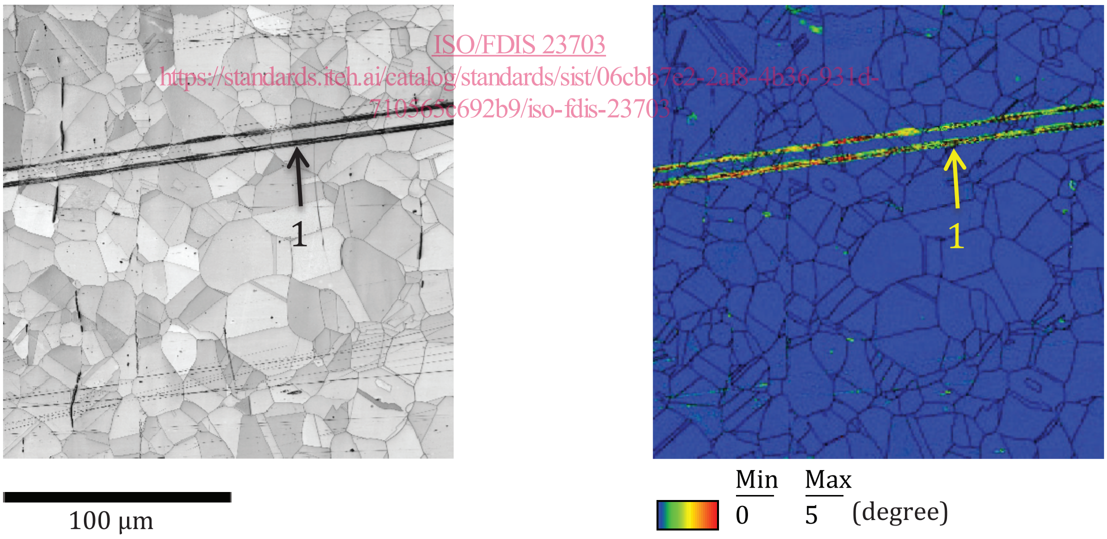
The procedures set out in ISO 24173:2009, Annex B shall be followed.

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